High Temperature Thin Film Superconductors and Microstrip Spiral Delay Lines

by

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Abstract

The field of superconductivity has recently begun to grow in an exponential fashion. This thesis has briefly described a history of superconductivity discoveries, provided the brief literature review about the superconductors. The superconductor material realization and characterization are provided. The delay line design method is presented and various printed delay lines are constructed. The experimental results are presented and discussed.

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Chapter I

Introduction

1.1 History of Superconductivity

The field of superconductivity, which has employed the services of a moderate number of engineers and technicians for several decades, has recently begun to grow in an exponential fashion. The reason for the recent explosion of activity is no doubt the remarkable discoveries since 1986 in the regime commonly referred to as ' High-Temperature ' superconductivity. In a very short period of time,the maximum critical transition temperatures increased from 23° K to 35° K and then to 90° K. Indeed, there are at the present time superconductor materials that possess transition temperatures at 110 - 125° K and intriguing possibilities of even higher transition temperatures. As the end of the nineteenth century approaches, enormous strides have been made in understanding the behavior of electromagnetic fields. Even so, there is no widely accepted theory for the behavior of free electrons in metals, particularly at low temperatures. It has been known since the beginning of the nineteenth century that the resistivity of metallic electrical conductors decreases with temperature. In the absence of data at very low temperatures, speculation has flourished.

Heike Kamerlingh Onnes, a Dutch physicist, discovered superconductivity in 1911 at the age of 58. Onnes devoted his career to exploring the limits of coldness. In 1908, he made helium a liquid by cooling it to 452 degrees below zero Fahrenheit (4 Kelvin). Liquid helium enabled him to chill other materials nearly to absolute zero (0 kelvin, the coldest possible temperature).

In 1911, Onnes began to investigate the electrical properties of extremely cold metals. He passed a current through a mercury wire, then measured the electrical resistance of the wire as he chilled it. At 4.2° K, the resistance suddenly vanished ! He indicated that mercury has passed into a new state, which because of its extraordinary electrical properties may be called the 'Superconductive State'. He soon found superconductivity in several other metals.

Onnes knew his discovery would have vast commercial potential as well as scientific importance. An electrical conductor with no resistance could carry current any distance with no losses, or carry current in a loop for the age of the universe ! In one key experiment, Onnes started a current flowing in a loop of lead wire cooled to 4 kelvin. A year later, the current was still flowing undiminished !

German scientists, Walter Meissner and R. Ochsenfeld, discovered that a superconductor won't allow a magnetic field to penetrate its interior. If a superconductor is approached by a magnetic field, it sets up screening currents on its surface. These screening currents create an equal but opposite magnetic effect, thereby cancelling the magnetic field and leaving a net of zero inside the superconductor material. Reversing the sequence gives the same result; if you first place the material in a magnetic field and then cool it to the superconducting state, it sets up screening currents that expel the magnetic field. This phenomenon is known as the Meissner Effect.

A magnet placed near a superconductor will literally see its mirror image and , since like or similar poles repel, both the superconductor and the magnet will try to move away from each other. If you place a magnet above a superconductor, the superconductor will levitate the magnet.

Superconductivity was a mystery for many years. In 1935, the brothers Fritz and Heinz London devised two equations describing zero resistance and the Meissner Effect. Fifteen years later, the Russian scientists Vitaly Ginzburg and Lev Landan described superconductivity in terms of quantum mechanics. These theories were phenomenological, in other words they described the observable phenomena without explaining what was occurring on a microscopic level.

Although the London Equations and the Ginzburg - Landon Theory helped guide experimental researchers, a comprehensive microscopic theory didn't appear until 1957, 46 years after Onnes' discovery. John Bardeen, Leon Cooper, and Robert Schrieffer, then at the University of Illinois, won the Nobel prize for this theory, now known as ' The BCS Theory '. According to Schrieffer, ' all electrons condense into a single state, and they flow as a totally frictionless fluid '. The electrons, which normally repel each other, develop a mutual attraction and form ' Cooper pairs '.

The theoretical basis for electronic applications appeared five years later, in 1962. Brian Josephson, then a graduate student at Cambridge University, analyzed what would happen at the intersection of two superconductors separated by a thin insulating barrier (now called ' Josephson's Junction '). He predicted that a supercurrent would tunnel right through the barrier. He said there would be no voltage across the barrier as long as the supercurrent did not exceed a critical value. He also predicted that a voltage across the barrier would create a high - frequency alternating supercurrent. These predictions evoked considerable skepticism, but John Rowell, Phil Anderson and S.

Shapiro verified them experimentally in 1963.

The next 20 years saw no major scientific advances in superconductivity. In 1973, John R. Gavaler of Westinghouse found a transition temperature of 23 kelvin in Nb_3Ge, but Nb_3Ge has seldom if ever been used in any commercial device application.

IBM corporation, in particular, has devoted an enormous effort over a 14 - year period to the development of Josephson technology for computer logic and memory applications. Although IBM made considerable progress in solving problems with lead - alloy junctions for operation at liquid helium temperature, the effort was finally halted in 1983 after the apparent advantages in performance over more conventional (nonsuperconducting) technologies appeared to be narrowing. It would be more prudent to invest in Gallium Arsenide and high - speed silicon semiconductor technology.

The modern age of the so - called high-T_c superconductors began in April 1986 with a report by IBM - Zurich's K. Alex Müller and J. Georg Bednorz of superconductivity in lanthanum - barium copper oxide at 30° K. The confirmation of the IBM - Zurich findings were reported by the group headed by Paul Chu in Houston as well as by Bertram Batlogg and his colleagues at AT & T Bell Laboratories.

Late in 1986, Paul Chu and his colleagues at the University of Houston demonstrated that, under tremendous pressure, La-Ba-Cu-O showed unmistakable signs of superconductivity well above 40° K. The T_c , in fact, increased at about 1° K per kilobar, reaching 57° K at 12 kbar. This work suggested to the groups at Houston and Alabama that the Barium should be replaced with smaller Strontium, to simulate the effect of high pressure. The substitution of Strontium worked, the onset of superconductive behavior was as high as 42.5° K. At about the same time (December 1986), a team at AT & T Bell Laboratories at Murray Hill, New Jersey, had also demonstrated superconductivity in La-Sr-Cu-O at 36° K.

In Huntsville, M. K. Wu and his two graduate students, Jim Ashburn and Chuan-Jue Torng, made several attempts to improve the T_c in La-Sr-Cu-O. Various substitutions for the barium (in La-Ba-Cu-O) were attempted. Magnesium proved to be a total failure. Calcium substitutions resulted in a superconductor, but the T_c was lower than that for La-Ba-Cu-O.

Later, on January 17, 1987 these researchers were considering substitutions for both La and Sr in La-Sr-Cu-O. Considering effects on size of the unit crystal, they generated a chart that suggested Yttrium was an appropriate element to substitute for lanthanum, likewise Barium for Strontium. The fact that the Y-Ba-Cu-O ceramic superconductor can now be produced almost routinely.

There was a hitch: Professor Wu had no yttrium and it was hardly a time to wait weeks for delivery. Fortunately, the Alabama group was able to obtain a modest supply from friends at the Marshall Space Center in Huntsville. By January 28, Professor Wu and his students had prepared a mixture of Y-Ba-Cu-O. That evening, the new compound went into the oven for overnight processing. The next day, the black and green charcoal - like materials were removed from the oven and made ready for a test of resistance versus temperature.

On January 29, the first resistivity measurement was completed at precisely 2:08 p.m.. The results were far beyond the expectations of the investigators. This first sample tested began to exhibit superconductivity at 90° K. Later that day, Wu called his colleague in Houston, Paul Chu. Wu's message was direct: ' We've hit the jackpot '.

On January 30, the group in Houston, using a sample hand-carried to Houston by professor Wu, reproduced the results from Alabama. The Alabama groups earlier conclusion was unmistakable.

Paul Chu provided a sample to Robert M.Hazen, an experimental mineralogist at the Geophysical Laboratory of the Carnegie Institution of Washington. Hazen found that the historic sample was actually composed of two major phases. The largest proportion consisted of a transparent, emerald-green crystalline structure, which had 2-1-1 ratios of yttrium, barium, and copper. This material (Y_2BaCuO_5) is now commonly referred to as ' that green stuff '. The remainder was an opaque, black structure. Since all known superconductors are opaque, the black material was suspected of harboring the superconducting properties and it did. The proportion of yttrium to barium to copper in this orthorhombic crystalline structure was 1-2-3; a label that has stuck to this remarkable material or compound.

While the previous announcement by Muller and Bednorz of a 30° K T_c had created interest in the advancement of superconductor applications, the news of superconductivity above 90° K galvanized the attention of the technological and commercial sectors. The ' old ' superconductors required expensive liquid helium for cooling, but these new 1-2-3 materials could be cooled with relatively inexpensive liquid nitrogen at 77° K. The potential advantages in simplicity of refrigeration and lower costs were enormous.

During early 1988, when it seemed that the revolutionary developments had reached a plateau, most physicists, chemists, and engineers had settled down to the tedious task of producing better 1-2-3 materials. The theorists, somewhat less subdued, were publishing literally hundreds of papers asserting their varied speculations about the mechanism of superconductivity in these oxygen deficient perovskites.

Then, when matters seemed almost back to normal, Hiroshi Maeda's group at Tsukuba Laboratories in Japan reported the bismuth-strontium-calcium-copper oxides, which began superconducting at about 110° K, reaching zero dc resistance at about 80° K. The discovery at Tsukuba Laboratories on December 25, 1987, was the result of persistence and hard work.

These early bismuth compounds, the Bi-Sr-Ca-Cu-O compounds exhibit several superconducting phases that are not trivial to separate. Even this remarkable report about the bismuth-based 100° K materials was not to be the last word in new superconductors.

The next big surprise came at the World Congress on Superconductivity in Houston during late February 1988. On Monday, February 22, Zhengzhi Sheng and Allen Hermann from the University of Arkansas presented a poster that electrified the other investigators. Sheng and Hermann reported success with a thallium compound (Tl-Ca-Ba-Cu-O) that showed an onset of superconducting behavior at about 120° K, and zero dc resistance at 106° K. Sheng and Hermann had made an announcement several days earlier in Arkansas, but their discovery went virtually unnoticed. Following their poster report, the claim was immediately confirmed by S. Parkin at IBM-Almaden. As with the bismuth compounds, there were a variety of superconducting phases present.[1] ~ [5]

1.2 Superconductor Applications

Since the discovery of high-T_c superconductors, there has been much interest in their possible applications. One of the first applications of high-T_c superconducting thin films will be for passive devices for high frequency and microwave applications. The scene of superconductor microwave applications is exciting: Thin-film microwave circuits with Qs of 100,000 or more; devices that operate to frequencies of 3000 GHz; sensors that can detect the magnetic energy of a single photon; zero resistance lines; superconductor transistors that are smaller, faster, more efficient and extremely reliable. These are only some of the reasons scientists are pursuing the development of high temperature superconductors.

Low temperature superconductors have laid dormant for 75 years because they required liquid helium cooling. Working at 4° K is very difficult. Liquid helium is expensive and inefficient as a coolant. Mechanical refrigerators at temperatures of 4° K are expensive and bulky.

The high temperature superconductors (HTSCs) enthusiasm increased when transition temperatures of superconductors went above 77° K because liquid nitrogen, which has a boiling point of 77° K could be used as a coolant; passive cooling in satellites is feasible in the 80° K to 100° K range. The ability to use liquid nitrogen solved many of the earlier helium problems. Liquid nitrogen is much cheaper and denser, and has a heat of vaporization that is 60 times higher than that of liquid helium. Using liquid nitrogen instead of liquid helium reduces the cost of cooling by a factor of 1200.

In spacecraft, passive cooling is achieved using black body radiators that dissipate heat into deep space with a background temperature of 2° to 3° K. Small heat loads, such as passive microwave devices and small active components, can be cooled easily to 80° K by this method. For other platforms, several companies have developed compact light-weight cryocoolers that can cool components to 77° K.

Passive microwave devices will be the first area to take advantage of the HTSCs. Microwave quality materials are available today and numerous design activities are underway to take advantage of their properties.

Superconductors will impact the microwave business in three phases. In phase I, they will improve exciting circuits by replacing normal metals with low loss superconductors. This will result in lower insertion losses, higher Qs, and much smaller circuits. In phase II, they will spawn new circuits that can be fabricated only with superconductors. In phase III, they will be used in active devices with a new class of chips fabricated from a combination of the superconductors and GaAs and /or silicon. These superconducting transistors with switchable zero voltage junctions will have an impact on computer design.

While the curves of resistivity and susceptibility define a superconductor, they describe only low frequency behavior and give little indication of how a material will perform at higher frequencies. Zero resistance at dc is somewhat misleading, since the loss of a superconductor increases rapidly with frequency. For a normal metal, loss increases as the square root of the frequency. For a superconductor, it increases as the square of frequency.

At the higher frequency, the figure of merit for superconductor quality is the microwave surface resistance (Rs). Superconductors made today that are 10 to 50 times more conductive than copper at 10 GHz and 77° K.

Superconductors differ from normal metals in other important ways. Normal metals have a skin depth that is a function of frequency, that is, as frequency increases the skin depth decreases. As a consequence of the Meissner-Ochsenfeld effect, waves cannot penetrate the depth of a superconductor. Hence, the penetration depth of a superconductor is a function of the material and not a function of frequency. For a high quality thin-film superconductor, the penetration depth is 2000 A. For gold at 1 GHz, the skin depth is 20,000 A. Low loss HTSC circuits can be fabricated from very thin films even at lower frequency[2].

Another property of superconductors is dispersion. In transmission lines of normal metals, there is considerable dispersion, that is the phase velocity is a function of frequency. This becomes a significant factor at higher frequencies and broad bandwidths. For a superconductor transmission line, dispersion is negligible. This property has important consequences when handling very short pulses or building delay lines.

Superconductors currently are being fabricated in several distinct forms: bulk, wire, thin films and thick films. Wires are used for magnets, motors and generators. Bulk materials are desirable since they can be machined into complex structures and shapes. Thin films and thick films are used mainly for microwave and microelectronic applications.

The HTSC films have critical fields and currents that are well within the operating ranges of microwave or microelectronic circuits. In addition, they provide lower resistance than either copper or bulk SCs and are able to carry much higher currents per cross section than other metals. Narrow SC lines are not lossy. This makes thin SC films a good choice for extremely small conductor patterns and circuits.

1.3 Objectives of theThesis

High temperature superconductors and their applications in engineering are significant and in fashion in recent years.

This thesis work is based on a literature review, learning, and understanding for the superconductivity behavior. One of the primary goals of this work is to investigate the thin film superconductor material realization and characterization.

The S-gun magnetron sputtering system was used with different sputtering conditions, different substrates (aluminum oxide, single crystal magnesium oxide, zirconium oxide, and yttria stabilized zirconia) in the work. In this thesis, work involving the design, simulation, construction, and measurement of microstrip delay lines including superconducting microstrip line is presented.

1.4 Thesis Structure

This thesis consists of five chapters. Chapter I provides a history of superconductivity discoveries. Chapter II is the brief literature review about the superconductors. Chapter III and chapter IV present the author's own work, which describes superconducting material characterization and delay lines. Chapter V is the conclusion chapter, summarizing the work in this thesis and providing future directions of the work.

Chapter II

Literature Review

2.1 Introduction

Since the discovery of the new class of ceramic superconductors with critical temperature Tc above the boiling point of liquid nitrogen [1]~[4], there has been much interest in the superconducting research fields. There are extremely large amounts of technical papers published in various journals and Conference Proceedings . The interest areas mainly included new material investigation, preparation methods of the thin film and bulk superconductors, and their applications in various engineering fields.

2.2 Concept of superconductivity

Materials in their superconducting state offer a means to circulate direct electric currents (DC) with no resistive loss. A superconductor carries a current without any noticeable resistance. A current flowing around a superconducting ring will flow indefinitely as long as the temperature is maintained below the temperature of the transition from the normal state to the superconducting state. Materials in their superconducting state also offer a means to convey low-frequency alternating currents (i.e., AC at 60 Hz) with unusually small losses. The absence or significant reduction of losses prompts universal interest in superconductors as energy savers.

Materials become superconducting only in certain circumstances which differ for each material. Superconducting materials only remain superconducting as long as they are kept below a certain temperature, and they do not experience too large a magnetic field, and as long as the current passing through them is not excessive. Thus, there are three important numbers associated with each superconductor material, the critical temperature (Tc), the critical magnetic field (Hc), and the critical current (Jc). Each quantity represents a maximum value above which superconductivity ceases to exist.

There are two basic methods of determining if a material is, in fact, superconducting. One method is to measure the electrical resistance of the material as the temperature is lowered. Even non-superconducting materials will exhibit a decrease of the resistance value as the temperature is decreased. However, only a superconductor will have its resistance drop dramatically to zero at some transition or critical temperature.

The second method to determine the nature of materials is the Meissner Effect. This effect shows that a superconductor (below the critical temperature) will expel a magnetic field by an internally induced electric current. However, if the external magnetic field exceeds the critical value, the material reverts to a non-superconducting state.[6] ~ [15]

2.3 Low - Temperature Superconductors

Professor Heike Kamerlingh Onnes discovered, when examining the electrical resistance of pure mercury at low temperature, that the resistivity, instead of decreasing continuously as expected upon cooling, vanished abruptly and completely at 4.15° K. Even early in the 1980's, more than 60 years after the discovery of superconductivity, there was no comprehensive theory which enabled accurate predictions about superconductivity to be made. However, there were well developed theories of superconductivity and a consistent description of the superconductivity phenomenon. The number of materials discovered to be superconducting grew continuously and rapidly since 1911, but the maximum transition temperature attainable remained always in the very low temperature range. These materials were called **low temperature superconductors**. These low temperature superconductor materials are : Vanadium (V, 5.3° K),Lead (Pb, 9.2° K), Niobium-Zirconium (Nb_2Zr , 10.8° K), Silicon-Vanadium (SiV_3 , 16.9° K), Niobium-Tin (Nb_3Sn, 18° K), and Niobium-Aluminum-Germanium ($Nb_{12}Al_3Ge$, 20.8° K).

An interesting property of a superconductor material is its diamagnetism, or impermeability to a magnetic field. When cooled in a magnetic field less than the critical value, a superconductor expels magnetic flux when the critical temperature is reached. The way in which this process takes place divides superconducting materials into two types. In type I materials, the magnetic field is totally excluded from the interior up to the value Hc, and above Hc the magnetic field penetrates fully into the sample and normal electrical resistance is restored. In type II materials, the perfect superconducting state exists for fields less than a lower critical magnetic field, Hc, and partial penetration of the magnetic field occurs and increases up to a higher critical field at which penetration is complete and normal electrical resistance is finally restored. Type I materials are restricted to those which show low values of Tc and Hc, whereas materials which are of interest in technological projects are invariably of type II, and in particular those which exhibit high field superconductivity.

The most important use of low temperature superconductivity is in the production of large magnetic fields over appreciable volumes without a large consumption of electrical power. In this area of use are superconducting solenoids which can produce 100 k Gauss continuously in bores of about 1 inch, and larger volume magnets, for instance similar to that constructed at the Argonne National Laboratory with a diameter of 16 ft and height 10 ft producing 20 k Gauss[16]. It has been proven that superconducting magnets can be fabricated that would replace adequately conventional iron core magnets with economies in both capital and running costs. For very large bubble chambers, the power dissipation in a conventional design is very high, about 11 MW, whereas using superconducting cables, the energy required little more than that finally stored in the magnetic field, and in the persistent mode only the refrigeration power to keep the magnet running.

The low temperature superconductors can be used in the construction of delicate instruments designed to measure extremely small quantities. The amount of superconducting material used is usually small in comparison with magnets, but the zero resistance to the flow of current gives such devices an advantage over those of conventional design[17] ~ [23].

2.4 High-Temperature superconductors

One of the most exciting developments in science in recent times is the discovery during the past few years of high-temperature superconductors. These new superconductors are ceramic oxides, not metals, and have the mechanical properties of ceramics. They are brittle, not ductile like metals. Further, the conduction is highly anisotropic. High current densities are obtained only when the flow is along two-dimensional sheets of copper oxide. The sheets are separated by the divalent barium and trivalent lanthanum. Limiting current densities are much lower when the flow is perpendicular to the sheets. High current densities are not found in the usual polycrystalline material in which the crystallites have random orientation. Real innovation in materials technology will be required to make the oxides practical for most potential applications.[24]

Superconductivity is a macroscopic quantum phenomenon. Quantum effects are exhibited on a macroscopic scale rather than the scale of atoms and molecules. There was no possibility of explaining the phenomenon before the advances of quantum mechanics in the mid-twenties.[25]

It is convenient to divide the oxide superconductors into three classes. Class 0 materials are compounds such as $LiTi_2O_4$[26], and $BaPb_{1-x}Bi_xO_3$[27]. These materials, although they do not possess remarkably high values of Tc, are very puzzling in that their transition temperatures are high despite their relatively low carrier concentrations. The class 1 materials are those related to La-Ba-Cu-O, which was first reported to be superconducting by Bednorz and Muller.[28] This material and several related compounds with the same K_2NiF_4 crystal structure have transition temperatures which apparently do not exceed 40° K. The class 2 materials exhibit superconductivity above liquid nitrogen temperatures, in the range of 90° K to 120° K, and have the chemical formula $MBa_2Cu_3O_{7-x}$,where the metal M is Y or any of the rare earths except Ce, Pr or Tb.[29]~[33]

2.5 A brief description of the theory of superconductors

For many decades, superconductivity was the shame and despair of theoretical physics. The phenomenon was discovered by Kamerlingh Onnes in 1911. The quantum theory of normal metals was developed fully by the early 1930's. A successful set of phenomenological equations for superconducting metals was given by F. London in

1935. Yet, in 1950, almost 40 years after the original discovery, there was not even a proper beginning of an adequate microscopic theory of superconductivity.

By accident, the role of the electron - phonon interaction was discovered by Frohlich in the year 1950. The ideas which he introduced have been so fruitful that , within a decade of his paper, a broadly satisfactory theory of superconductivity has come into existence.

A further important step in the theory of superconductivity was made by Bardeen, Cooper, and Schrieffer (BSC theory, the first successful microscopic theory of superconductivity). They examined a simplified model of a Hamiltonian in which the electron - phonon interaction is replaced by direct interaction between electrons. This interaction is constant in a narrow layer close to the Fermi surface and only terms are retained which correspond to the interaction of electron pairs with opposite momentum and spin. Thus, they have, in essence, taken into account only pairs found in the " condensate ". For such a Hamiltonian they have calculated a mean (and free) energy with the help of trial wave functions, taking into account correlations between electrons of opposite momentum and spin. Elementary excitations in the system were calculated, and it was shown that, as in the one - dimensional model, these have an energy gap that depends on temperature and goes to zero at a certain critical temperature. These findings have made it possible to give an explanation of thermal and electric properties of superconductors which is in satisfactory agreement with experiments.

Although BCS 's calculations are based on a rather idealized model, they give a reasonably good account of the equilibrium properties of superconductors. When the parameters of the theory are determined empirically, they find that they get agreement with observed specific heats and penetration depths to within an order of 10 %. Only the critical temperature is involved in the superconducting phase; the other two parameters required (density of states and average velocity at the Fermi surface) are determined from the normal phase.[34]

Bardeen, Cooper, and Schrieffer were awarded the 1972 Nobel Prize in physics for their landmark accomplishment.

2.6 Realization of high-Tc superconductors with RF magnetron sputtering

High Tc superconductors may not be fully understood, and may involve complex processing and take several days to heat treat properly, but these high-Tc superconducting materials, in both bulk and thin-film forms, are starting to be developed into real word products. The three primary high-Tc materials that researchers are working with are Y-Ba-Cu-O (with Tc = 95° K) , Bi-Pb-Sr-Ca-Cu-O (with Tc = 108° K) , and Tl-Ca-Ba-Cu-O (with Tc = 125° K)[35]-[37]. A vanadium oxide-based material with a 130° K Tc has been developed, but it is unstable and in addition it loses its superconductivity after a few days when exposed to air.

Development of these materials into thin films for electronic applications is slow, but demonstrated a relatively steady output, without the major engineering problems that the bulk materials have had to overcome. Bulk high-Tc problems involve the inherent brittleness of the ceramic components, which makes them hard to form into wire. They also have a limited current carrying capacity, and they exhibit " flux creep " , which expends electrical energy by opposing lines of magnetic force[38].

High quality thin Y-Ba-Cu-O films can be produced by several techniques, such as sputtering[39],[40], electron beam co-deposition[41] ~ [43], molecular beam epitaxy[44], and laser deposition[45] ~ [47]. The basic requirements to obtain ideal films are (1) an exact composition with a 1:2:3 atomic ratio of the metallic elements, (2) an epitaxial film growth and (3) an optimized alloy to satisfy these requirements.

In the Hybrid Microelectronics laboratory at Virginia Tech, thin films of Y-Ba-

Cu-O were deposited using an S-gun type sputtering system. It is necessary to deposit interface layer (buffer) for alumina substrates. During the sputtering of the interface layer and the superconductor layer, the substrate was held at a temperature of 480 ~ 540° C, and the chamber atmosphere was held at a pressure of 0.27 pa (2 mtorr) pure argon with a flow rate of 3 sccm. After deposition of Y-Ba-Cu-O layer, the sample was pre-annealed in the sputtering chamber with a pure oxygen atmosphere at a total pressure of 173 pa (1.3 torr) until the sample turned opaque. Next, the samples were annealed in a tube furnace with an oxygen flow of 1400 sccm (0.05 scfm) at 900 ~ 915° C for 120 seconds then cooled to room temperature at a rate of 2° C / min.

2.7 Concept and Design of Delay Lines

One of the first applications of high temperature superconductivity is expected to be related to microwave delay lines. The superconducting delay lines will allow much smaller, lighter, and cheaper devices, with less frequency dispersion and much lower insertion losses. Microstrip is the most widely used and most practical transmission line structure for microwave applications; as a consequence, the superconducting delay lines have been made with microstrip line structures[48].

It is necessary to account for the time when electromagnetic wave propagates on the transmission lines. For a TEM mode wave propagating in a homogeneous dielectric medium, the propagation time per unit length is [49]

$$T = \frac{1}{v} = \frac{1}{\frac{1}{\sqrt{\mu\epsilon}}} = \sqrt{\mu\epsilon} \qquad (2\text{-}1)$$

where

T = propagation time per unit length

v = velocity of electromagnetic wave,

μ = permeability of the medium,

ε = permittivity of the medium

$$\mu = \mu_o \mu_r = \mu_o$$

$$\epsilon = \epsilon_o \epsilon_r$$

where

$$\mu_o = 4\pi x10^{-7} \ \ henry/m = 3.83x10^{-7} \ \ henry/ft.$$

$$\epsilon_o = 8.85x10^{-12} \ \ farad/m = 2.69x10^{-12} \ \ farad/ft$$

Therefore,

$$T = \sqrt{\mu\epsilon} = \sqrt{\mu_o\epsilon_o}\sqrt{\epsilon_r} = 0.0847\sqrt{\epsilon_r} \ \ ns/inch. \qquad (2\text{-}2)$$

Practically, the modes on microstrip lines are only quasi-Tranverse Electric and Magnetic (TEM). Thus the result of the delay time which is independent of the frequency is approximate. The microstrip cannot support a pure TEM, or any other simple electromagnetic field mode, and the necessary longitudinal field components which exist lead to the propagation of hybrid modes. All metallic microstrip lines are dispersive. For TE and TM modes, the group velocity V_g is the function of the frequency :

$$V_g = V_m \sqrt{1-(\frac{f_c}{f})^2} \qquad (2\text{-}3)$$

The propagation time per unit length is the form

$$T = \frac{1}{V_g} = \frac{1}{V_m\sqrt{1-(f_c/f)^2}} = \sqrt{\mu_o\epsilon_o}\,\sqrt{\epsilon_r}\,\frac{1}{\sqrt{1-(f_c/f)^2}} \tag{2-4}$$

where

V_g = group velosity of electromagnetic wave,

f_c = cutoff frequency,

f = operating frequency,

V_m = medium phase speed.

The delay lines are special transmission lines. The time that waves take to propagate through the delay line is called delay time, and is referred to by T_d. The delay lines have been widely applied in pulse technologies, electromagnetic measurement instruments, and also some digital circuits. Delay lines today are made by winding a length of low loss coaxial cable into a tightly wound coil. For a 100 ns delay , the coil could weigh two pounds and have an insertion loss of 20 to 30 db. A thin film superconductor delay line can have loss of a fraction of one db and size and weight reductions of 100 to 1.[50]

Microstrip delay lines are series in nature and their performance degrades with any film imperfection along the line. Long delay lines require dense packing and, consequently, narrow linewidths and small dielectric thicknesses. Both these requirements are severely constrained by the presently available materials. Fabrication yield is reduced due to the requirements of film uniformity over the whole substrate. For linewidths smaller than 100 μm, YBaCuO film quality is difficult to maintain over large substrate areas. Furthermore, compatible substrates, mostly $LaAlO_3$, only very recently became available in 250 μm thicknesses. Attempts to deposit high quality YBaCuO films on both sides of the substrate have long been undertaken but reports of success are only now starting to emerge.[51] ~ [54]

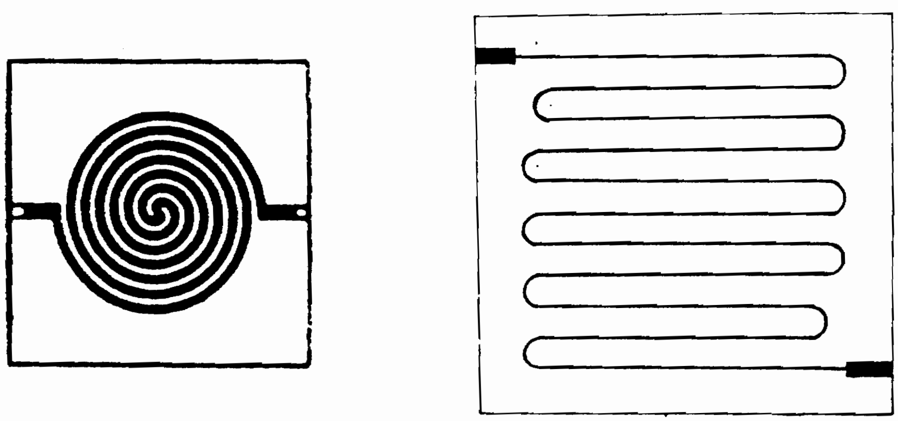
There are two different winding shapes for practical delay lines; serpentine shape and spiral shape,respectively, shown as figure (2-1), with the serpentine as a periodic structure. It is possible to get resonance for a periodic system. The serpentine structure is not often used in order to avoid the parasitic resonance in circuits. Perhaps the double screw structure (spiral) is much more suitable to use.

The amount of delay is obtained as measured by Time Domain Reflectometry (TDR) techniques. The TDR system consists of a generator, an oscilloscope system, and a reference transmission line in which only the principal mode can propagate and observed at x = 0 (see figure (2-2)).

Assume that no load resistance is connected to the transmission line output terminals ($Zl = \infty$) and that $Zg' = Zo$. As the zero-impedance step generator applies its 1 - volt step signal to Zg'. the oscilloscope voltmeter indicates 0.5 volt. The oscilloscope voltmeter will continue to indicate a 0.5-volt signal until the wave travels down the line to the open end, doubles in amplitude due to no current into $Zl = \infty$ and is reflected back to the generator end of the line. A 1-volt signal is finally indicated on the oscilloscope after the measurable time required for the step signal to travel down and back in the finite length of open-ended transmission line.

If $Zl = Zo$, there is no wave reflected back and the oscilloscope voltmeter will always indicate a 0.5-volt signal. If $Zl = 0$, the oscilloscope voltmeter will indicate 0-volt signal when the wave travels down the line to the short end. Figure (2-3) illustrates TDR oscilloscope (voltmeter) displays related to the value of Zl versus the value of the transmission line Zo.

Assume that a delay line with impedance of 50.5 Ω (slightly larger than Zo) is connected to the oscilloscope voltmeter shown as in figure (2-4,a) , the time domain displays would be the waveform shown as in figure (2-4,b). It is easy to determine the delay time from figure (2-4,b).



Spiral Serpentine

Figure (2-1) Layout of the various lines

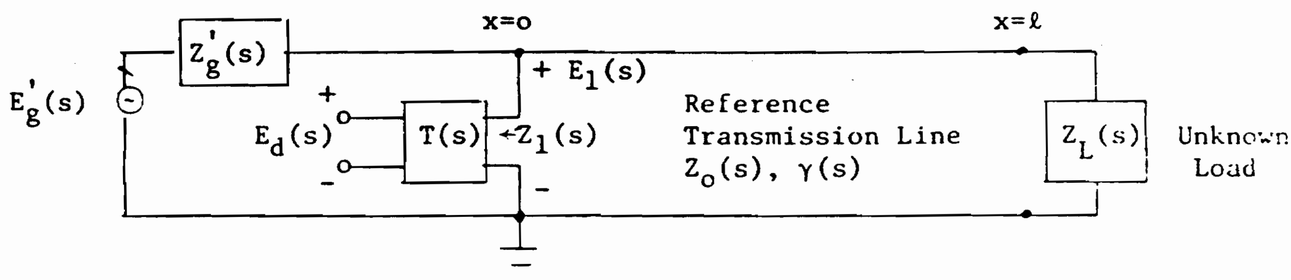


Figure (2-2) TDR system

The transmission loss (S_{21}) is measured by using a HP 8510 Network Analyzer. The Microwave Network Analyzer system consists basically of three blocks: a sweep oscillator, a transducer / harmonic frequency connector combination, and a display unit. The reflection - transmission test unit in combination with a sweep oscillator and a network analyzer makes up a system for measuring transmission and reflection - phase and magnitude for microwave components in a wide frequency range. With this transducer in the system, transmission or reflection parameters can be measured. In terms of S - parameters, both S_{11} and S_{21} can be measured with one setup. By merely reversing the device under test, the other two S - parameters, S_{22} and S_{12} can also be determined.

The calibration of the Microwave Network Analyzer test setup is of prime importance to obtain accurate and valid data. When adjusting the system prior to any measurement, one has to be sure that all connections and adapters necessary for the actual setup should be used. Once the system is calibrated and the device to be tested is connected in the system, no further change in test setup need to be made to measure all S-parameters.

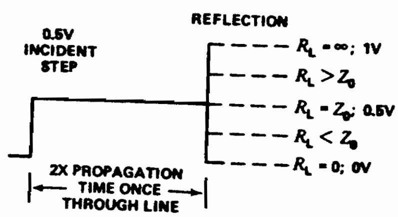
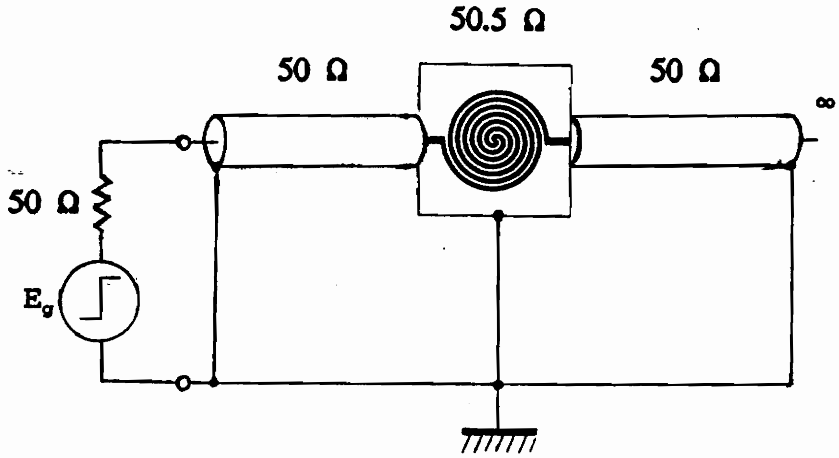
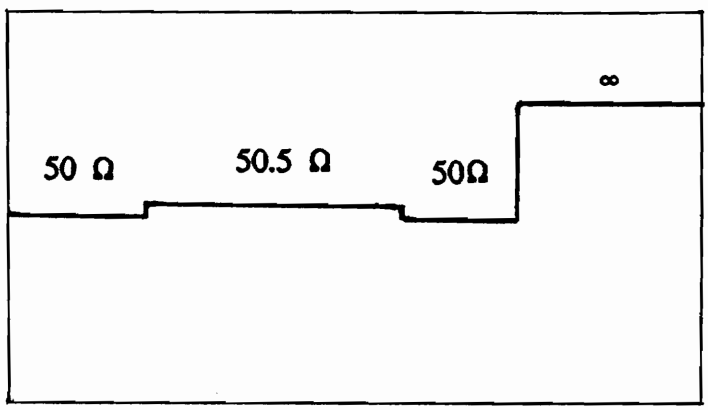


Figure (2-3) Oscilloscope voltmeter displays for TDR system of figure (2-2), dependent upon value of Zl versus Zo



(a)



(b)

Figure (2-4) Delay line measurement circuit and TDR oscilloscope displays

2.8 Conclusion

A superconductor shows no impediment to the flow of steady electrical current. The state of superconductivity cannot be present above a certain critical temperature (or transition temperature), T_c . The superconductivity can be destroyed by imposing a magnetic field above a certain critical field, H_c . The superconductivity is limited to less than a critical current, J_c .

The modern age of the so called high-T_c superconductors began in April 1986. The critical temperature T_c was risen from 30° K (LaBaCuO) to 90° K (YBaCuO), and then 125° K (TlCaBaCuO).

Thin film superconductors are the best form for electronic applications. The high quality thin film superconductors can be produced by several techniques including RF magnetron sputtering.

One of the first applications of high temperature superconductivity is expected to be in microwave delay lines. They will allow much smaller, lighter, and cheaper devices, with less frequency dispersion and much lower insertion losses.

Chapter III

Superconducting Material Characterization and Superconductive Delay Line Realization

3.1 Introduction

After reviewing the literature, we can begin to mention the practical investigation work from this chapter.

During the past few years, a number of research groups have prepared superconducting thin films of YBaCuO on a variety of substrates by several techniques, such as sputtering, electron beam co-deposition, molecular beam epitaxy, and laser deposition. In this work, an off-axis single-target magnetron sputtering technique is used.

Inexpensive amorphous/ceramic noncrystalline substrates have emerged as an important substrate material for potential high-frequency hybrid microelectronics applications. Since the substrates have problems of substrate-film diffusion and no crystal lattice for epitaxial growth, it is difficult to grow an orthorhombic superconductor structure on such polycrystalline substrates. Therefore, a buffer layer needs to be deposited in order to act as a diffusion barrier during the superconductor growth process.[55] ~ [59]

There are various factors, such as substrate surface parameters, the sputtering conditions (temperature, gas pressure, RF power, time and so on), and the annealing condition, that are known to have an effect on the superconducting properties of YBaCuO thin film.

3.2 Superconductor Material Realization and Characterization

(1) The steps of making the superconductor thin films

The steps of making the superconductor thin films consist of substrate processing, sputtering superconductor films, pre-annealing, post-annealing, and finally conducting measurements.

a) Substrate process

Usually, the substrates need to be polished (both mechanically and chemically). This is expected to yield a smooth surface ready for film growth. In some cases, the substrates are heated in air or oxygen to 1100-1200° C for 12 - 24 hours, a process that should results in the formation of a high density of atomic steps on the substrate surface.[60]

The substrates have to be cleaned completely before they are sputtered. In general, there are a lot of dust particles and oil residue on the substrates. These dust particles and oil residues make the thin films easy to be separated from the substrates, affecting their adhesion strength.

b) Sputtering superconductor films

The method of High-Temperature Superconductor thin film growth which has been used with considerable success is sputtering. The sputtering process can be performed using either multiple metal (or oxide) targets or a single oxide target. The general principles are the same in either case. Basically, sputtering involves the use of energetic ions (such as Ar^+) to erode a target. The target species then are deposited onto a heated substrate which is located near the target.

In on-axis sputtering, the substrate faces the target[61]. This position gives reasonable deposition rates, but this geometry places the substrate within the plasma region, so that the deposited film is bombarded by ions. This may result in film damage and, more importantly in the case of High-Temperature Superconductor films, the preferential resputtering of some atomic species. The films deposited also tend to be quite nonuniform.

Because of these problems, off-axis sputtering has become quite popular for High - Temperature Superconductor film deposition. In this configuration, the substrate is oriented at an angle with respect to the target, so that it is not in the plasma region as shown as figure (3-1) . This position results in uniform and stoichiometric films with state-of-the-art properties. The major drawback of the off-axis geometry is the relatively low deposition rate, requiring a long period of time to achieve one run.

A far off-axis single target magnetron sputtering system to deposit YBaCuO thin film is used in this work. The substrate was located away from the axis of the target, as shown in figure (3-1) . The angle θ is taken to be equal to or little more than 45°. The substrate temperature should be set to a temperature of the order 540° C. One should vary very slowly the temperature going up or down in value.

The pure argon gas at a flow rate of 3 SCCM and with a total pressure of 2.0 mTorr is used for sputtering from the copper rich ($YBa_2Cu_3O_X$) ceramic target. A typical deposition rate of 1.5 μm / hr of the compound is obtained with an RF power

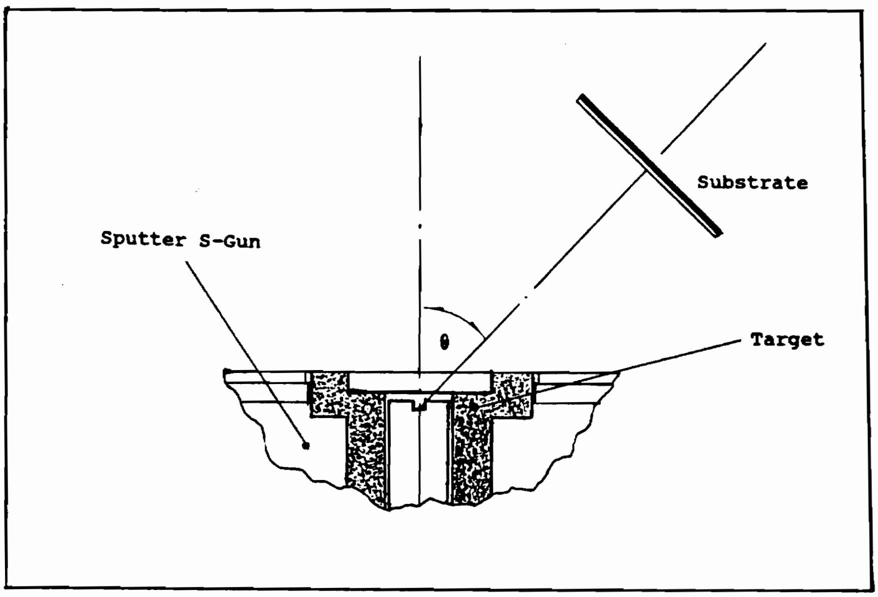


Figure (3-1) The substrate location and orientation.

of 100 W.

c) Pre-annealing

The deposited thin film is pre-annealed in the pure oxygen atmosphere of 1.3 Torr at about 540° C just prior to removal from the vacuum chamber. During pre - annealing, the color of the superconductor changes from semitransparent black to its normal color.

d) Post-annealing

The samples are annealed in an oxygen atmosphere at the temperature of about 900° C for 120 seconds and then cooled to room temperature at rate of 2° C / min. These post-annealing treatments are necessary to transform the thin films so that they will become superconducting at these low temperatures.

For YBaCuO superconductor films, the annealing temperature has to be very close to 900° C. At the temperature of 900° C, large amounts of the films transformed to the high Tc phase. If the annealing temperature is too low, most of the films remained in the low Tc phase.

e) Measurement

A dc four point probe measurement is used to determine the resistance. Four silver pads were evaporated onto the YBaCuO films to form low resistivity contacts[62].

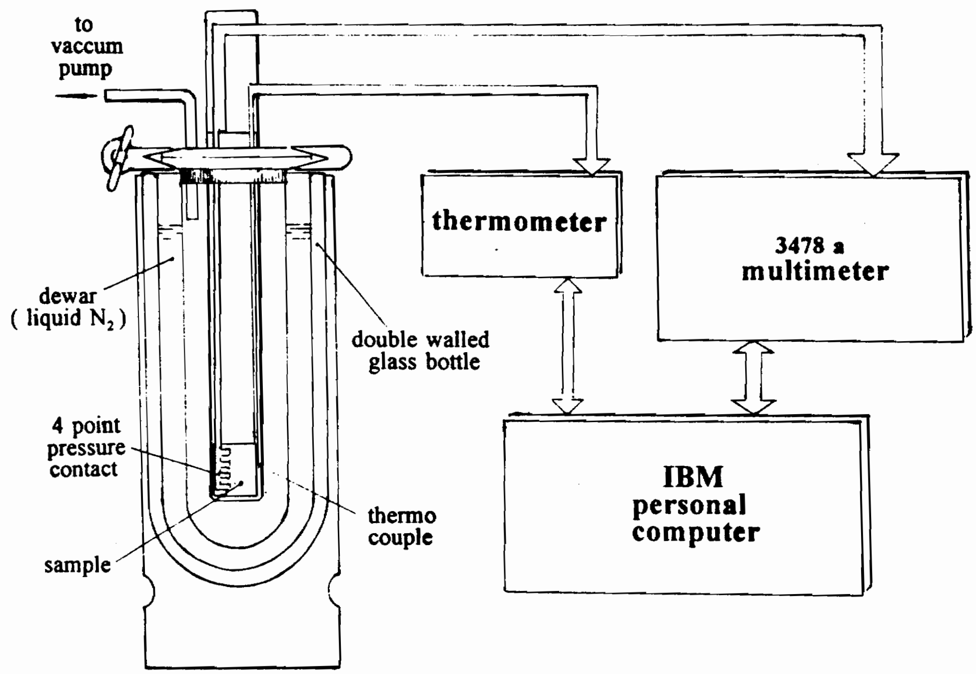


Figure (3-2) Four point probe measurement system.

The sample was mounted in an evacuated glass chamber and lowered into a dewar of liquid nitrogen. A type T thermocouple and a microprocessor controlled thermocouple thermometer were used to measure the temperature. The computer controlled acquisition of temperature versus resistance measurements were performed in the microelectronics laboratory (see figure (3-2)).

(2) The effect of interface layer between YBaCuO thin films and substrates

In the case of superconductive film deposition on polycrystalline substrates such as alumina ceramics (Al_2O_3), hetero-epitaxial growth cannot be expected. Crystallization has to be achieved by spontaneous crystallization similar to bulk superconductors. The layers start with very poor quality crystals and gradually improve; thus, the ' transition layers ' are formed. Then, the high quality crystals are deposited on top of these transition layers. Because the effective thickness of the crystals remains poor, the thick transition layers between the high quality surface layers and the substrates become necessary. The thickness of the transition layers is a very important parameter in the applications of thin film superconductors. The interface enhances crystallization significantly. This has been demonstrated through successful experimental work. The effects of two different buffer layers, Niobium (Nb) and Zirconia (ZrO_2) on superconductivity of YBaCuO films were investigated in the course of this work.

(3) The effect of substrate surface parameter

The substrate surface parameter is very important for the superconductive properties of thin films. Single crystal magnesium oxide (MgO), zirconium oxide (ZrO_2), and yttriastabilized zirconia (SYZ) were used as the substrates under study. All the substrates were mechanically polished to a normally scratch-free optical finish with 1/4 or 1 μm diamond grit. The first group of substrates was standard cleaned, the second group was chemically etched in a hot phosphoric acid (50° C). This is expected to yield an atomically smooth surface. The third group was heated in oxygen to 950-1000° C for 12 hours, a process that resulted in the formation of a high density of atomic steps on the substrate surface [63],[64].

(4) Experimental results

Various observations were made in the course of this experimental investigation :

a. The resistance-temperature profiles of the deposition results on Zirconia (ZrO_2), Magnesia (MgO), Alumina (Al_2O_3), Aluminum-Nitroge (AlN), and Silicon (Si) substrates are shown in figure (3-3) through (3-7). The onset temperature varies from 98 to 105° K and the critical temperature (Tc) varies from 90 to 95° K for Zirconia and Magnesia substrates. These results indicate that the good quality YBaCuO thin films can be deposited on these substrates by using magnetron S-gun sputtering systems. The Zirconia (ZrO_2) and Magnesia (MgO) substrates are much better than other substrates.

b. Figure (3-8) shows the effect of ZrO_2 buffer layer thickness on the resistance versus temperature profile of YBaCuO compound on ZrO_2/Al_2O_3. The profile has demonstrated an optimum condition after one hour of ZrO_2 deposition (5.4 μm). Increasing the ZrO_2 sputtering deposition time to two hours has been found to have the resistance characteristic unaltered, as illustrated in figure (3-9).

c. Figure (3-10) shows the resistance-temperature profile of the deposition results on Zirconia ceramics (ZrO_2) for different sputtering deposition time and temperatures. It is seem that the best condition to deposit is at a temperature of about 540°C for 9 hrs.

d. Figure (3-11) shows the effect of the different gas pressure on the resistance versus temperature profiles of YBaCuO compound on Ti doped MgO substrate.

e. Corresponding effects are observed for the deposition of the YBaCuO compound on Alumina with Niobium buffer layer, as illustrated in figure (3-12).

f. figure (3-13) shows the resistance-temperature profiles of the deposition results on Silicon with ZrO_2 buffer layer for the same sample with different annealing temperatures. The results show that the annealing temperature is very important. The annealing temperature has to be very close to 900° C. At this temperature, Large amounts of the films transformed to the high-T_c superconductor phase. If the annealing temperature is too low (say, 800° C), Most of the films remained in the low Tc phase (like semiconductor).

g. The effect of substrate surface process with different methods on the critical temperature is illustrated in table (3-1). According to table (3-1), the most likely best process to prepare the substrate surface for thin-film superconductor growth is thermally annealing these substrates, especially true for MgO and ZrO_2.

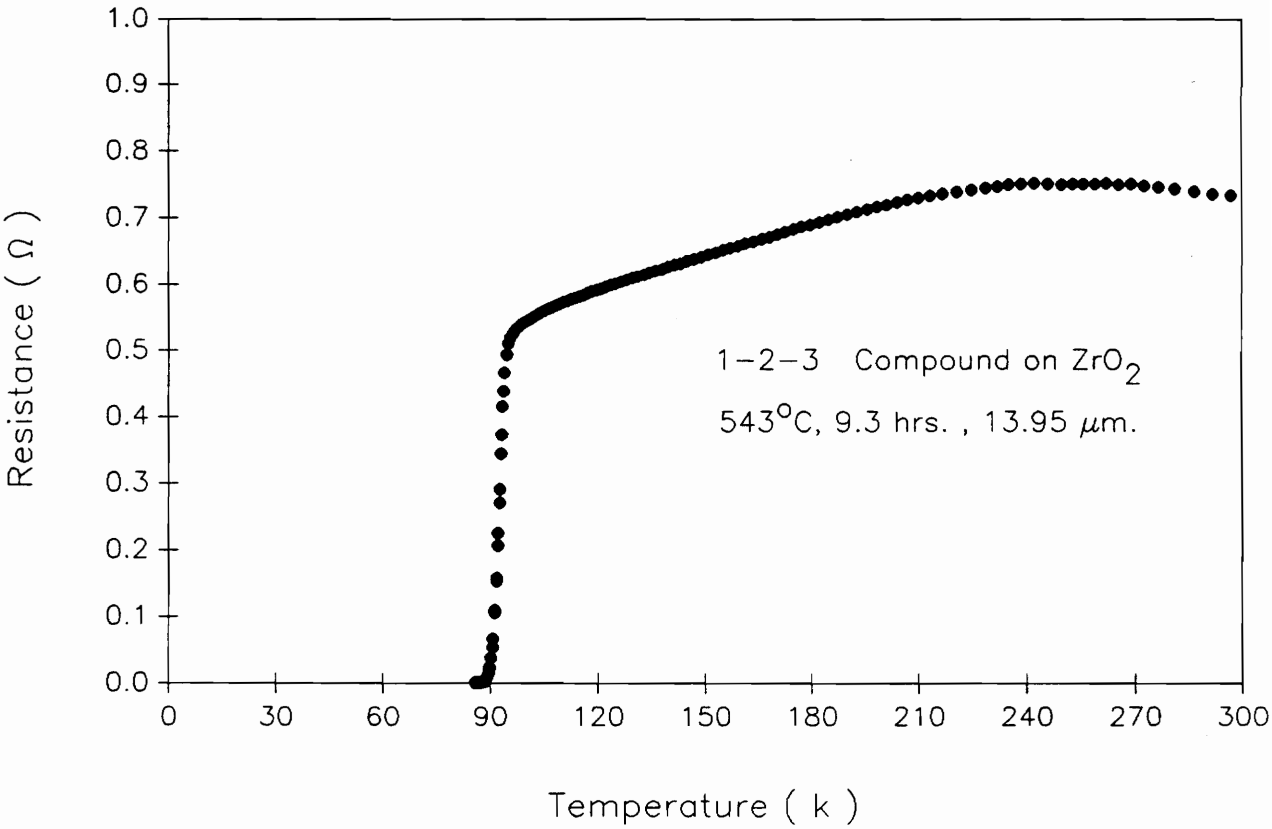


Figure (3-3) Resistance - temperature profile of 1-2-3 compound on Zirconia ceramics (ZrO_2)

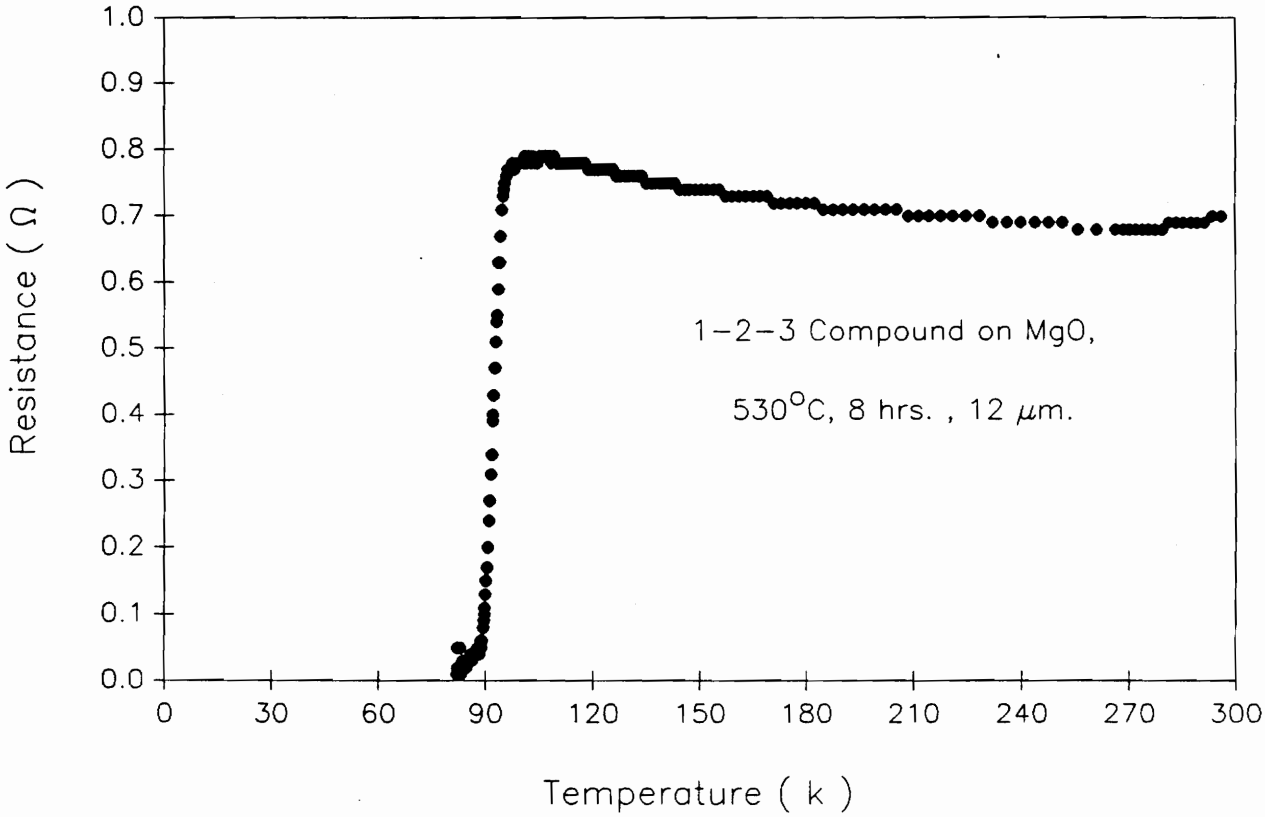


Figure (3-4) Resistance - temperature profile of 1-2-3 compound on Magnesia crystal (MgO).

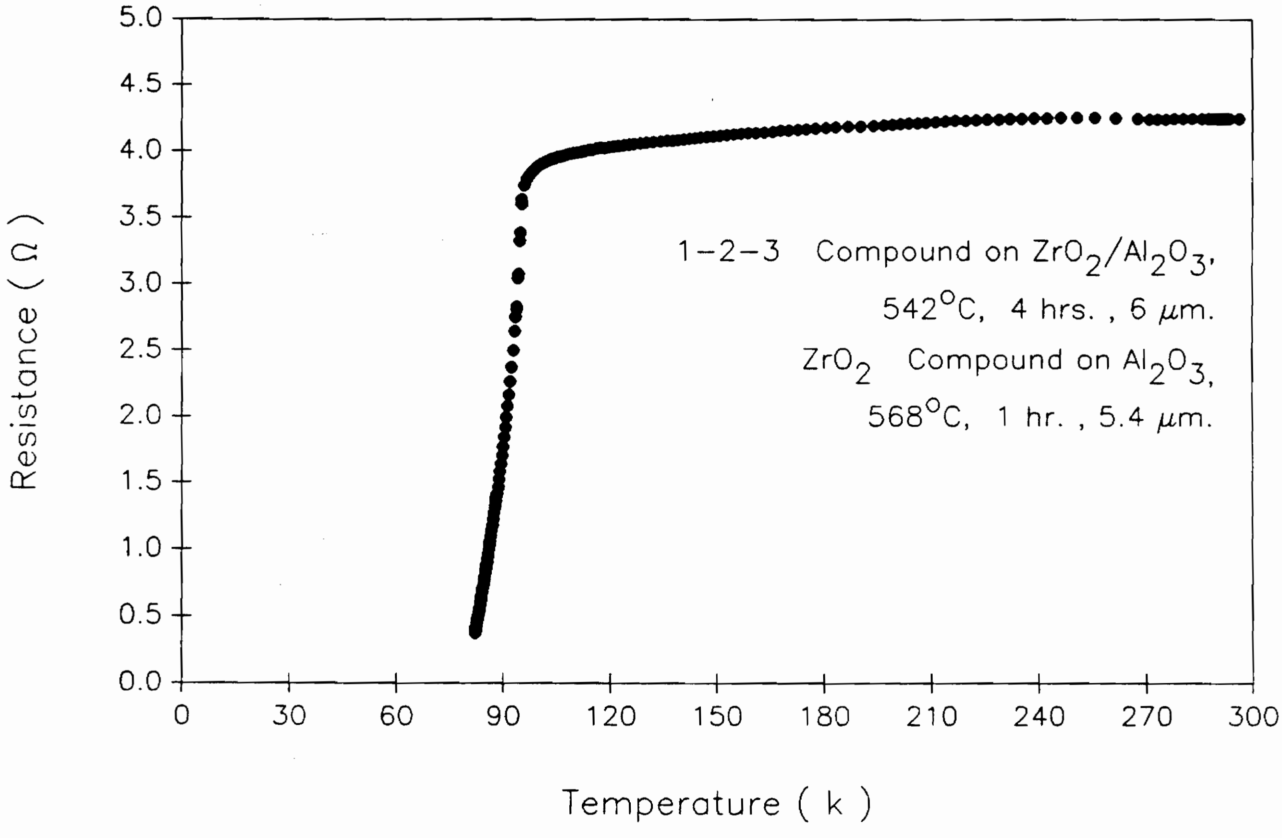


Figure (3-5) Resistance - temperature profile of 1-2-3 compound on Alumina with ZrO_2 buffer layer.

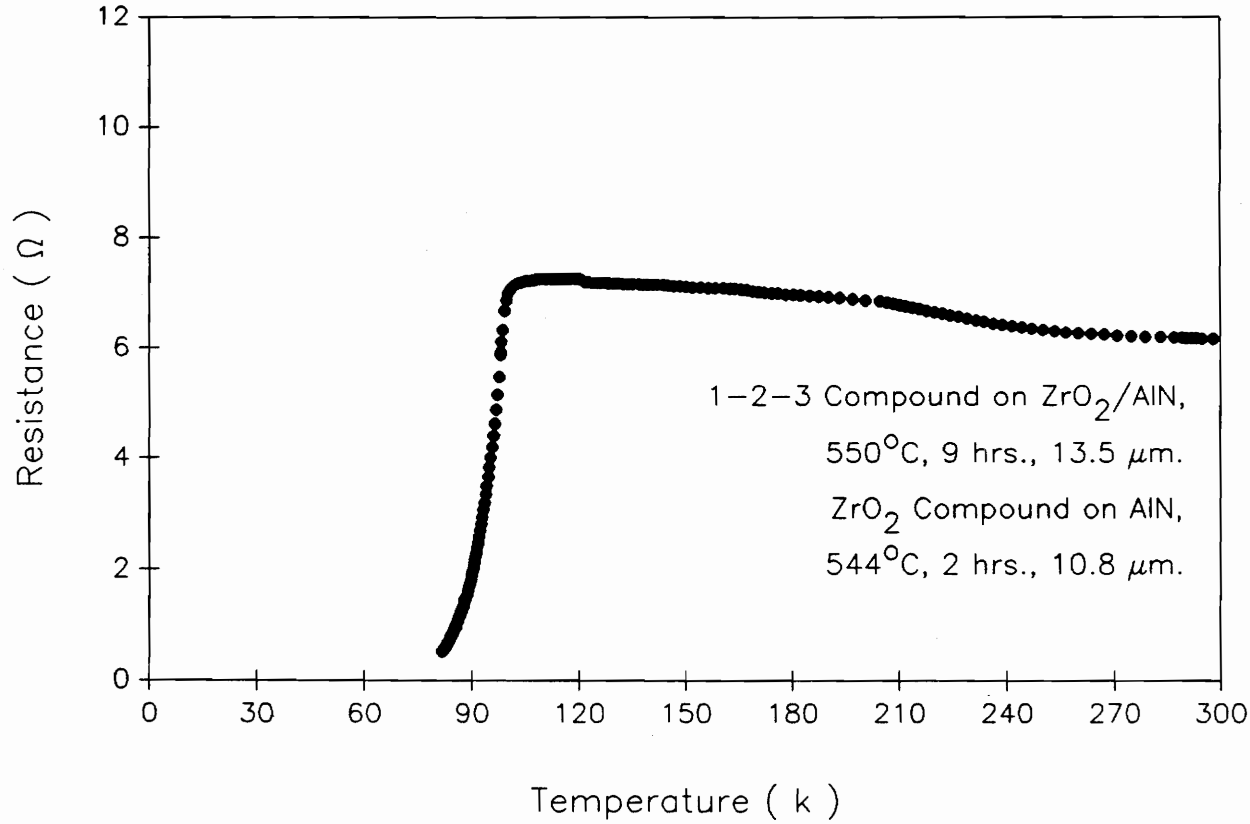


Figure (3-6) Resistance - temperature profile of 1-2-3 compound on Aluminum - Nitrogen (AlN) with ZrO2 buffer layer.

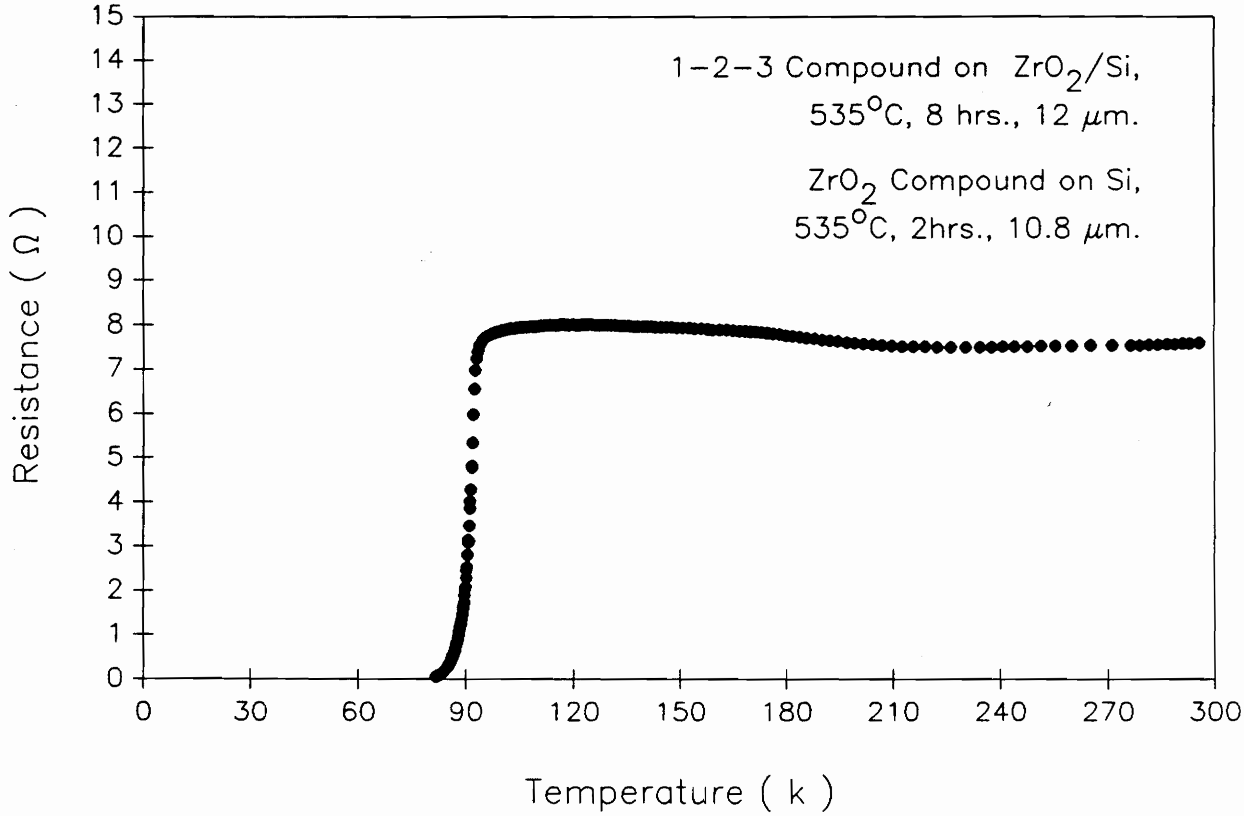


Figure (3-7) Resistance - temperature profile of 1-2-3 compound on Silicon with ZrO_2 buffer layer.

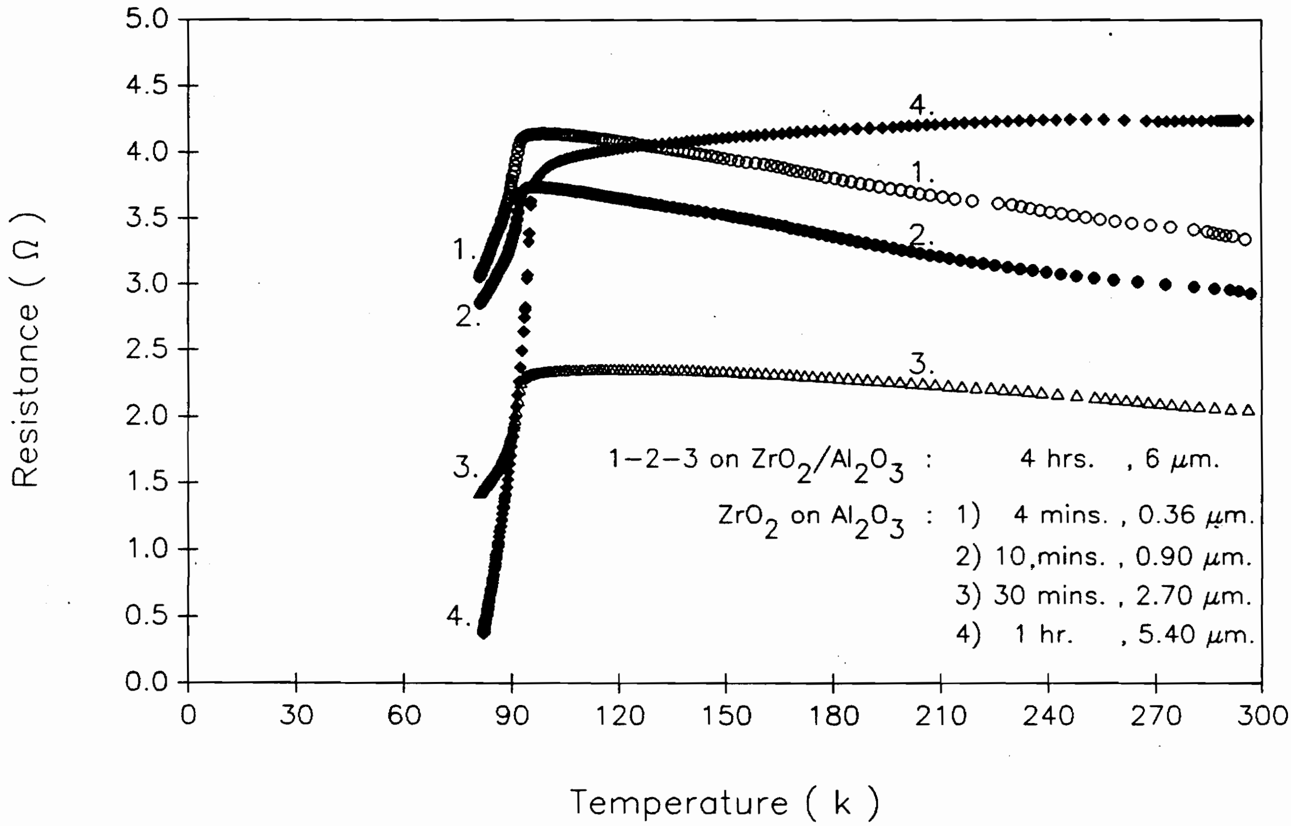


Figure (3-8) Effect of ZrO_2 buffer layer thickness on the resistance - temperature profile of 1-2-3 compound.

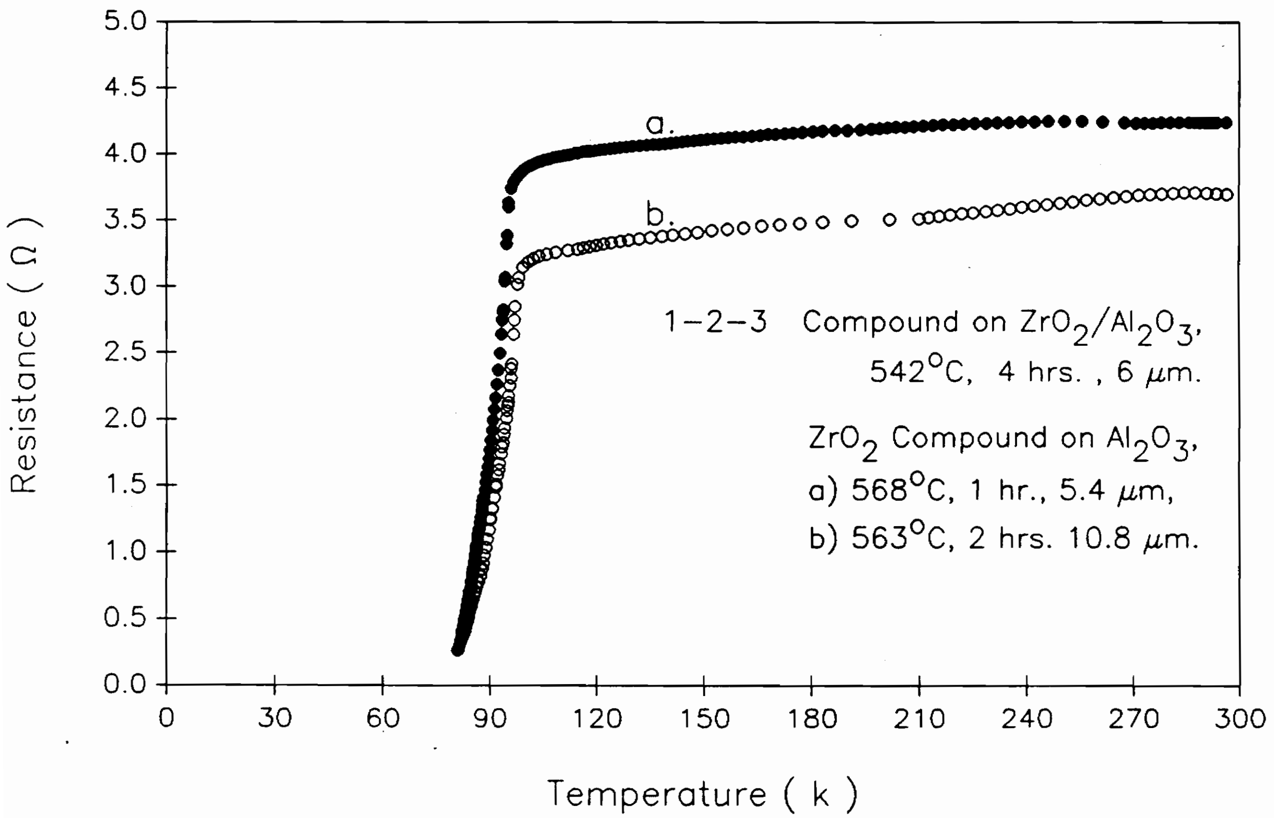


Figure (3-9) **Effect of ZrO_2 buffer layer thickness on the resistance - temperature profile of 1-2-3 compound.**

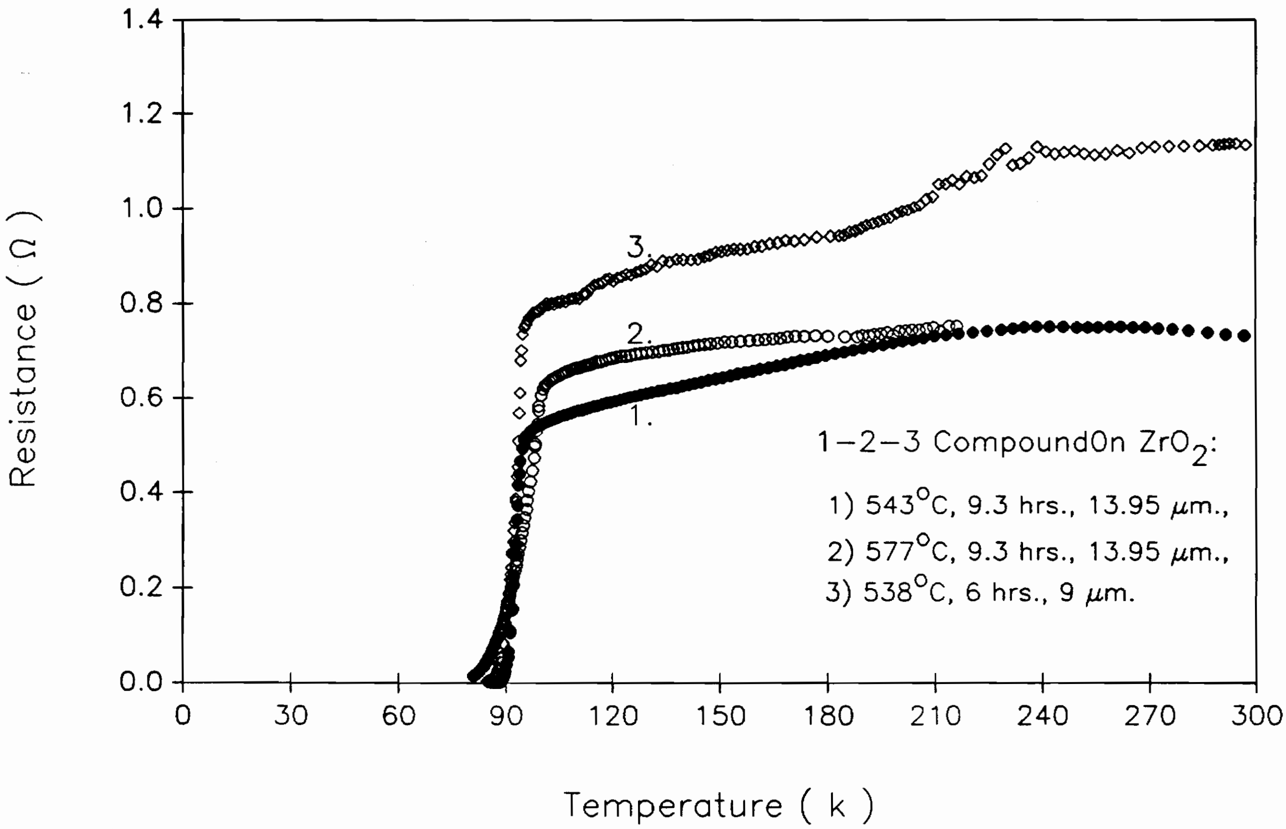


Figure (3-10) **Effect of sputtering time/temperature of YBaCuO compound (on ZrO_2) on the resistance-temperature profiles.**

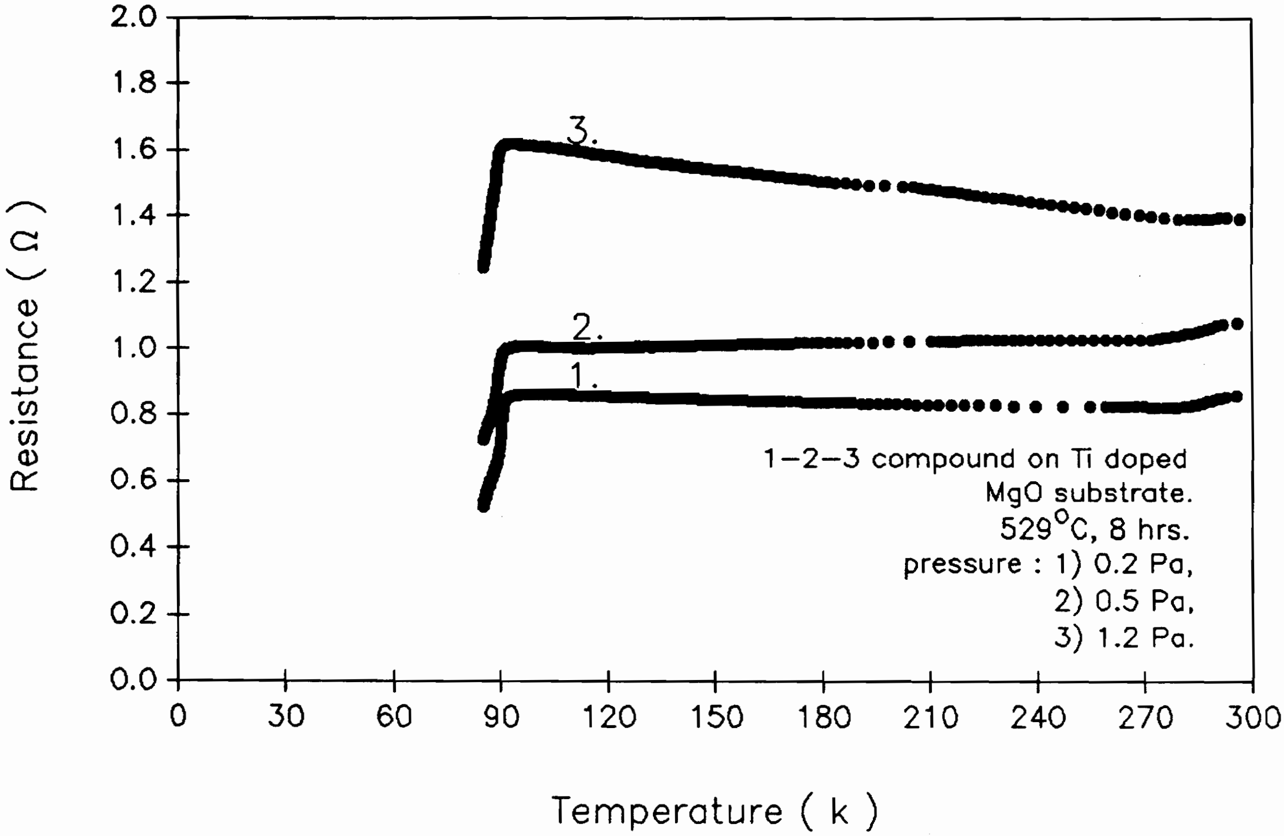


Figure (3-11) The resistance versus temperature profile of YBaCuO compound on Ti doped MgO substrate (pressure : 0.2 ~ 1.2 Pa.)

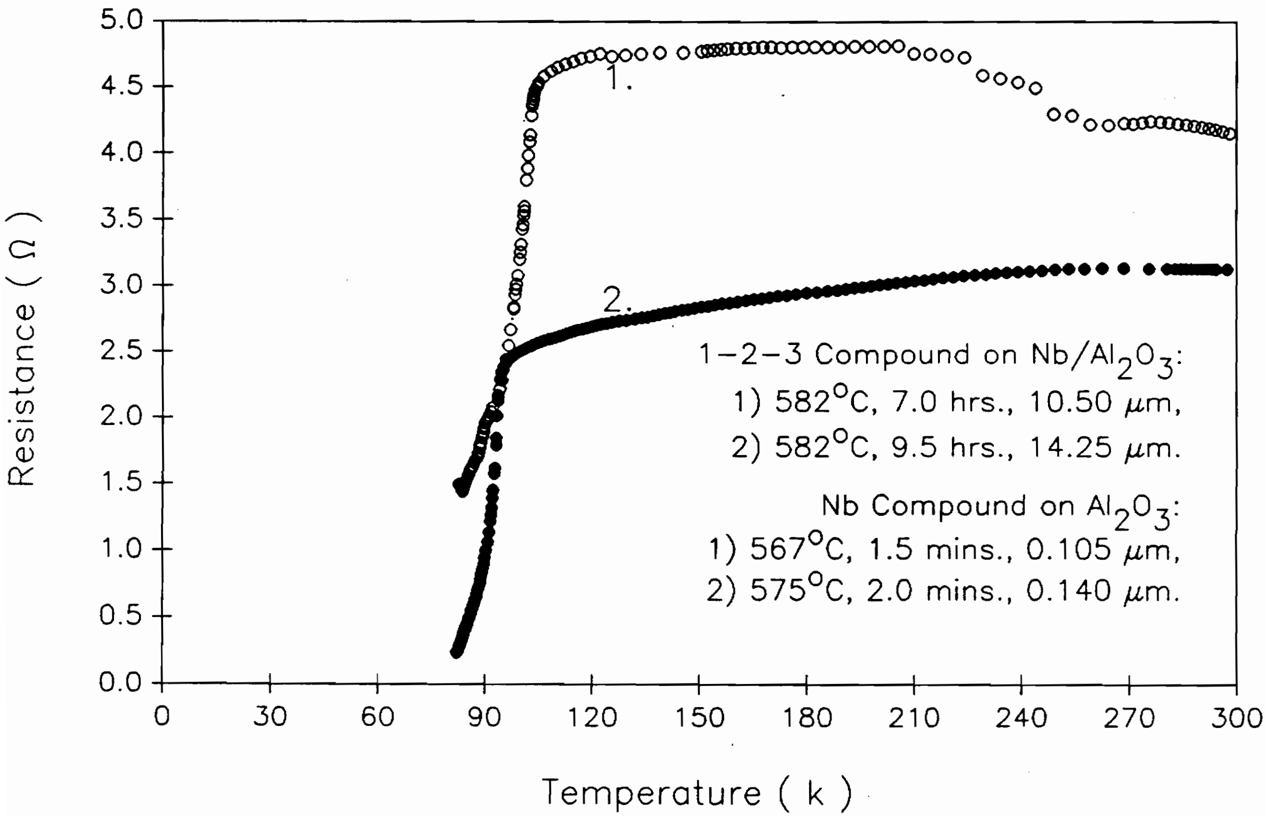


Figure (3-12) Effect of Nb buffer layer thickness on the resistance - temperature profile of YBaCuO compound.

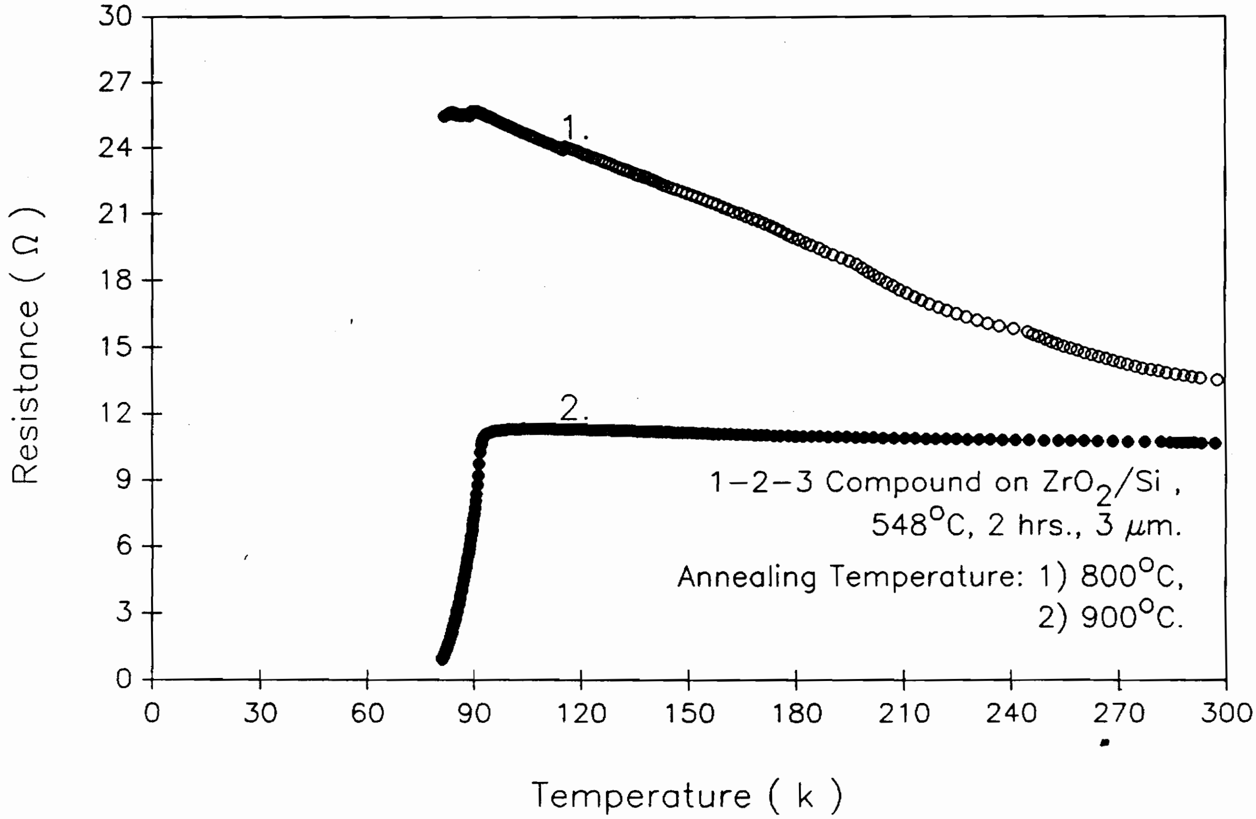


Figure (3-13) Resistance-temperature profiles of the deposition results on Silicon with ZrO2 buffer layer for the same sample with different annealing temperature.

Table (3-1) The effect of substrate surface process with different methods on the critical temperature.

Substrate	Standard cleaning	Chemically etching	Thermally annealing
MgO	78° K	75° K	80° K
ZrO_2	80° K	79° K	83° K
YSZ	75° K	78° K	67° K

3.3 Delay Line Realization

The superconductor thin film quality,the substrate property, design , and fabrication are important parameters for superconductor delay line realization.

(1) Substrates

The quest for substrate materials that are capable of supporting excellent films of high- Tc superconductor (HTS) materials has been in progress for nearly as long as HTS thin films have been prepared[65]. A good thermal expansion match is necessary, whether or not one is dealing with an epitaxial system. In the case of the YBaCuO compound, this requirement is particularly important because of the brittleness of the superconductor material. The best HTS films grown to date, as determined by a multitude of metrics including critical current density, morphology, and stability over time, are epitaxial in nature on their substrate surface. This most likely dictates that the lattice mismatch between the film and the substrate should be as small as possible, although high quality films have been grown on MgO substrate, which has a mismatch of the order of 9 % [66].

Of particular concern for the growth of high quality YBaCuO films, whether or not they are epitaxial, is the chemical compatibility of the film with the substrate material. The constituents of the YBaCuO compound are reactive with many substrates that might otherwise be good candidates (such as unbuffered Si)[67]. The issue of chemical compatibility has generally meant that the substrates that support reasonably high quality YBaCuO films are themselves oxides.

An ideal substrate would have a flat surface and be free of abrasions and other structural inhomogeneities, although a number of materials in current use as YBaCuO substrates do have such problems. It would be desirable, at the very least, to grow films on a substrate that has no phase transitions within the temperature regime required for film processing. In the case of microwave applications, where the dielectric properties of the substrate have an important effect on device performance[68].

Device applications impose a number of other property requirements on the HTS substrate material. Microwave applications are not generally very sensitive to the dielectric constant of the substrate (as long as it is uniform and, preferably, isotropic), but they do depend on having a low value of the loss tangent.

The compatible substrates - mostly $LaAlO_3$ - very recently became available in 250 m thicknesses but they are very expensive. The author chose MgO as the substrate material for delay line realization. It is well established that at normal growth temperatures there are no obscuring chemical reactions between the film and the MgO substrate[69]

(2) Microstrip delay line calculation and design

a) Determination of the microstrip width W

The microstrip width W is in relation to the characteristic impedance Zo and thickness h of substrate. For an infinite thin strip, in the case of the narrow strip, i.e. Zo > (44 - 2 ε_r) , the following equation is provided,[70]

$$\frac{W}{h} = \left[\exp \frac{H'}{8} - \frac{1}{4 \exp H'} \right]^{-1} \tag{3-1}$$

where W - width of strip;

h - thickness of substrate.

and

$$H' = \frac{Z_o\sqrt{2(\epsilon_r+1)}}{119.9} + \frac{1}{2}\left(\frac{\epsilon_r-1}{\epsilon_r+1}\right) . \left(\ln\frac{\pi}{2} + \frac{1}{\epsilon_r}\ln\frac{4}{\pi} \right) \tag{3-2}$$

The characteristic impedance Zo is of the form,

$$Z_o = \frac{119.9}{\sqrt{2(\epsilon_r+1)}}[\ln(4\frac{h}{W}+\sqrt{16(\frac{h}{W})^2+2})$$

$$-\frac{1}{2}(\frac{\epsilon_r-1}{\epsilon_r+1})(\ln\frac{\pi}{2}+\frac{1}{\epsilon_r}\ln\frac{4}{\pi})] \qquad (3\text{-}3)$$

The effective microstrip permittivity ε_{eff} is given by

$$\epsilon_{eff} = \frac{\epsilon_r+1}{2}[1+\frac{29.98}{Z_o}(\frac{2}{\epsilon_r+1})^{1/2}(\frac{\epsilon_r-1}{\epsilon_r+1})(\ln\frac{\pi}{2}+\frac{1}{\epsilon_r}\ln\frac{4}{\pi})]^2 \qquad (3\text{-}4)$$

When the strip thickness becomes significant, the effects of finite thickness on the electric field should be taken into account. An indication of the change in the electric field distribution is provided by Figure (3-14), and several workers have investigated the effects of finite strip thickness.[71]

For W/h < 1/2 π :

$$\frac{W_e}{h} = \frac{W}{h}+\frac{1.25t}{\pi h}(1+\ln\frac{4\pi W}{t}) \qquad (3\text{-}5)$$

where W_e is the effective microstrip width. The characteristic impedance Zo is given by

$$Z_o = \frac{60}{\sqrt{\epsilon_{eff}}}\ln(8\frac{h}{W_e} + 0.25\frac{W_e}{h}) \qquad (3\text{-}6)$$

When the strip thickness increases, the characteristic impedance Zo decreases, Therefore, the strip width has to be properly adjusted (decrease).

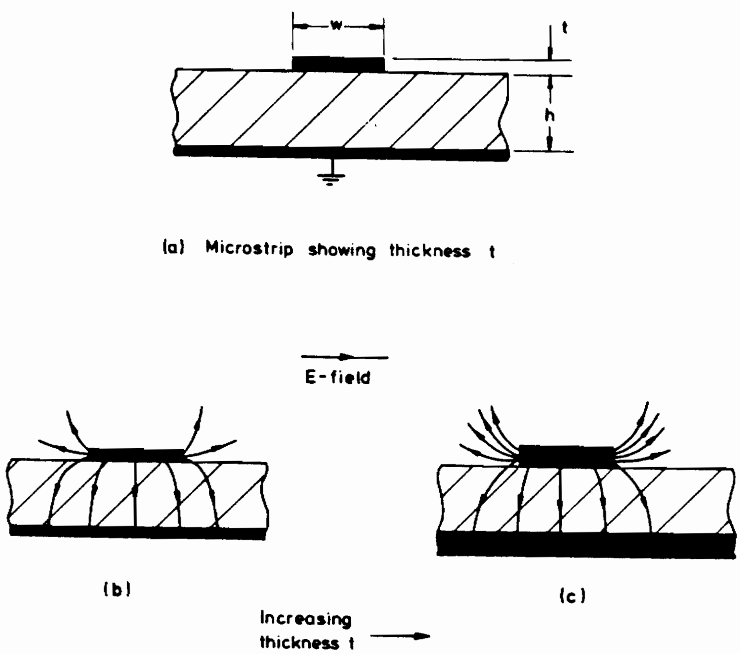


Figure (3-14) Changes in the distribution of electric field (transverse cross-section) as the thickness of microstrip is altered.

For a MgO substrate,

$$\epsilon_r = 9.65, \qquad h = 19.7\ mils.,$$

$$H' = \frac{50\sqrt{2(9.65+1)}}{119.9} + \frac{1}{2}\left(\frac{9.65-1}{9.65+1}\right) . \left(\ln\frac{\pi}{2} + \frac{1}{9.65}\ln\frac{4}{\pi}\right) = 2.12$$

$$\frac{W}{h} = \left(\frac{\exp 2.12}{8} - \frac{1}{4 \exp 2.12}\right)^{-1} = 0.99$$

$$W = 0.99\ h = 0.99\ x\ 19.7 = 19.5\ mils.$$

W is selected to be 20 mils.

The characteristic impedance Zo is given by

$$Z_o = \frac{119.9}{\sqrt{2(9.65+1)}}\left[\ln\left(4\frac{19.7}{20} + \sqrt{16\left(\frac{19.7}{20}\right)^2 + 2}\right)\right.$$

$$\left. - \frac{1}{2}\left(\frac{9.65-1}{9.65+1}\right)\left(\ln\frac{\pi}{2} + \frac{1}{9.65}\ln\frac{4}{\pi}\right)\right] = 49.4\ \Omega.$$

b) Delay line design

The MgO substrate has the geometry value of 787x787x20 mils. Let the lead line Lo (see Figure (3-15)) be

Lo = 59 mils.

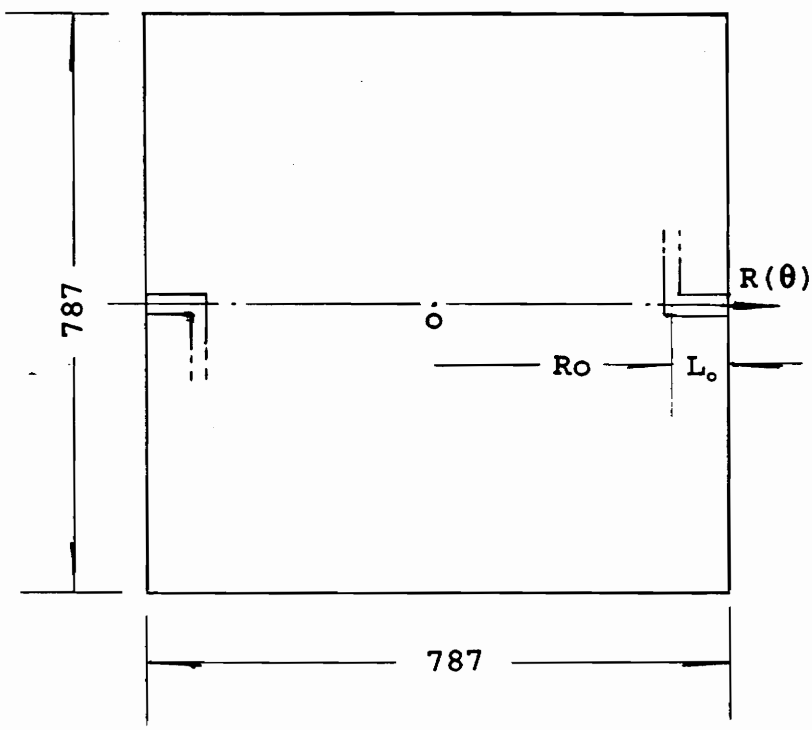


Figure (3-15) The delay line layout.

The maximum radius of the spiral Ro is

$$R_o = \frac{787}{2} - 59 = 334.5 \ mils \approx 335 \ mils.$$

The expression for spiral is

$$R(\theta) = \gamma\theta e^{j\theta} \qquad (3\text{-}7)$$

Let

$$\theta = 0 \rightarrow 5\pi$$

$$R_o = | \ R(5\pi) \ | = 5\pi\gamma = 335$$

$$\gamma = \frac{335}{5\pi} = 21.3 \ mils/rad.$$

Therefore,

$$R(\theta) = 21.3 \ \theta \ e^{j\theta} \qquad (3\text{-}8)$$

Figure (3-16) is the designed double spiral delay line profile. The distance between two strip lines d_o should be larger than 3 times the thickness of the substrate in order to avoid the coupling between them, i. e.

$$d_o \geq 3h = 60 \ mils.$$

Let's check it :

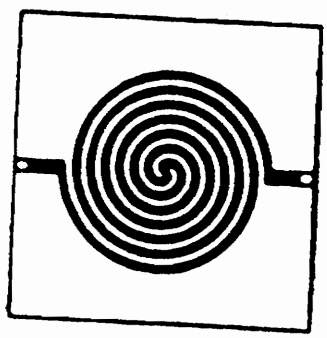


Figure (3-16) Designed double spiral delay line

$$d_o = \frac{|R(\theta+2\pi)|-|R(\theta)|}{2} = \frac{|R(2\pi)|}{2} = 21.3\ \pi = 66.9\ mils > 3h.$$

c) The propagation-delay time

Equation (2-2) gives the propagation-delay time T per unit length for a homogeneous dielectric medium. The total length of this double spiral line is

$$L = 2\int_0^{5\pi} |R(\theta)|\, d\theta = 2\int_0^{5\pi} 21.3\ \theta\ d\theta = 21.3\ \theta^2 \Big|_0^{5\pi} = 5255.6\ mils.$$

$$= 5.2556\ inch.$$

The delay time T_d is equal to,

$$T_d = TL = 0.0847\ x\ 5.2556\ x\ \sqrt{9.65} = 1.383\ ns.$$

(3) The fabrication of superconductor microstrip delay lines

After getting a high quality high - Tc superconductor thin film, one can begin to build the delay line.

a) Make delay line patterns

The superconductor thin films on one side of the sample was etched to make the microstrip delay line patterns using wet chemical etching in ethylenediaminetetraacetic acid (EDTA).

F. K. Shoroohi et al. presented a novel chemical wet etch which readily removes

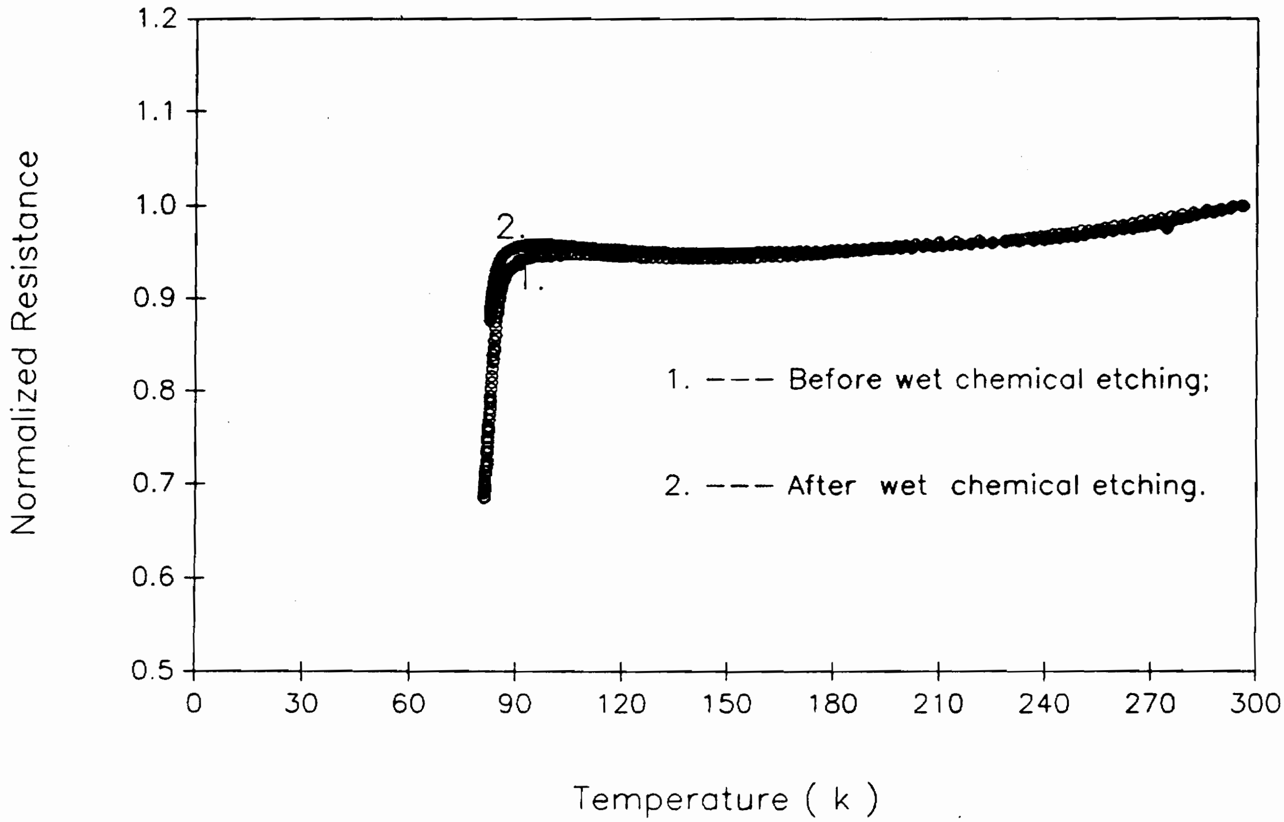


Figure (3-17) **Normalized resistance versus temperature profile of the 1-2-3 compound to illustrate wet chemical etching.**

YBaCuO, leaving the substrate material intact.[72] The method is quite suitable for use in standard photoresist lithography microfabrication of superconducting devices. With this etchant, a 20 mil wide double spiral pattern which exhibits no measurable degradation in the superconducting transition temperature as shown in Figure (3-17) was fabricated.

A positive photoresist was used with spun at 5000 rpm for 30 second. The saturated solutions of EDTA/H_2O were stirred with spun at 350 rpm and heated at 50° C during etching. Very smooth sidewall of the strip line under these conditions were obtained.

b) Packaging

The lead lines of the double spiral patterns have to be connected with the interior conductor of the coaxial connector, as shown in Figure (3-18). It is very difficult to have superconductor films sealed on the copper conductors. Before they are sealed, the superconductor films have to be cleaned with ion beam and then deposit the silver or gold layer, and put it into oven to bake at 550°C with oxygen.

c) Measurement

As describing in chapter II, the amount of delay is obtained by Time Domain Reflectometry (TDR) measurements. The insertion loss (S_{21}) is measured using an HP 8510 Network Analyzer.

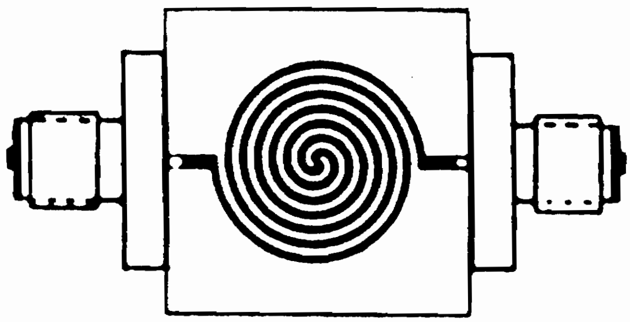


Figure (3-18) The superconductor delay line package.

d) Discussion

All the necessary procedures to achieve superconductor delay lines were followed. The only result is the delay time which is equal to 1.4 ns. We didn't get the results of the delay line insertion losses. The quality of the superconductor thin films was the main reason. The author was not able to obtain the high quality YBaCuO films due to contamination of the sputtering chamber. The critical temperature Tc of the 1-2-3 films on MgO substrate was not high enough for microstrip delay line operation.

Almost all of the good samples with good results were done in 1991. The current sputtering chamber is too small to have the substrate holder located far away from the axis of the S-gun. The electron bombardment can't be avoided efficiently so that the superconductor thin films deposited are quite nonuniform.

Perhaps the YBaCuO target quality is another problem. The old target was used out. The new one is a product of lower quality. If the starting material is bad, the thin films produced will not be good.

3.4 Conclusion

A superconductor thin film was realized and characterized. For Zirconia and Magnesia substrate, the onset temperature varies from 98 to 105° K and the critical temperature (Tc) varies from 90 to 95° K. The buffer layer thickness, sputtering time, gas pressure, substrate temperature, annealing temperature, substrate surface process will effect the thin film superconductor properties.

Superconductor delay lines can be built. These devices require that high quality and uniform superconductor thin films are produced.

Chapter IV

Delay Lines : Various Technologies

4.1 Introduction

Various technologies (like thin-film sputtering, thick-film screen printing, etching...) can be used to make delay lines successfully. Thick-film technology has become well established in the low-cost manufacture of hybrid microcircuits at lower-than-microwave frequencies and for densely packaged digital subsystems.[73] Thick-film materials and techniques have been developed for microwave integrated circuits (MIC) operating at frequencies up to about 20 GHz[74].

Two methods are commonly used for the manufacture of ' thick-film ' circuits including delay lines.

(a) Thick-film patterns are printed and fired to on the ceramic substrate (usually alumina).

(b) A printed circuit technique is used to etch the desired pattern in the copper cladding substrate.

Method (b) is very well known and need not be described here. The first method is used for MIC manufacture to an increasing extent and therefore it will be briefly described.

Three samples of thick-film delay lines were made. Two of them were copper lines on Teflon-ceramic (Duroid 6010). The delay line patterns were made by chemical etching. The other was printed silver on alumina substrate.

4.2 Thick Film Delay Line Realization on Alumina Substrate

(1) Thick-film delay line design

96 % alumina substrate was used in this work. The relative parameters are given below :

$$\epsilon_r = K = 9.6$$

$$h = 25\ mils,$$

$$a \times b = 1000 \times 1000\ mils.$$

From equation (3-1) ~ (3-5), we can determine the width of microstrip lines.

$$H' = \frac{50\sqrt{2(9.6+1)}}{119.9} + \frac{1}{2}\left(\frac{9.6-1}{9.6+1}\right)\left(\ln\frac{\pi}{2} + \frac{1}{9.6}\ln\frac{4}{\pi}\right)$$

$$= 2.11$$

$$\frac{W}{h} = \left[\frac{\exp 2.11}{8} - \frac{1}{4\exp 2.11}\right]^{-1}$$

$$= 0.999$$

$$W = 0.999 \times 25 = 24.975 \approx 25 \ mils.$$

$$\epsilon_{eff} = \frac{9.6+1}{2}\left[1 + \frac{29.98}{Z_o}\left(\frac{2}{9.6+1}\right)^{1/2}\left(\frac{9.6-1}{9.6+1}\right)\left(\ln\frac{\pi}{2} + \frac{1}{9.6}\ln\frac{4}{\pi}\right)\right]^2$$

$$= 6.4216$$

For thick films, the effect of thickness of the metal strip should be considered (typically t = 0.55 mils) :

$$\frac{W_e}{h} = \frac{W}{h} + \frac{1.25\ t}{\pi h}\left(1 + \ln\frac{4\pi W}{t}\right)$$

$$= 0.999 + \frac{1.25 \times 0.55}{25\pi}\left(1 + \ln\frac{100\pi}{0.55}\right)$$

$$= 1.06332$$

$$Z_o = \frac{60}{\sqrt{6.4216}} \ln\left(8 . \frac{1}{1.06332} + 0.25 \times 1.06332 \right)$$

$$= 48.60\ \Omega < 50\ \Omega .$$

So, the strip width should be reduced. Let

$$W = 24\ mils,$$

$$\frac{W_e}{h} = \frac{24}{25} + \frac{1.25 \times 0.55}{25\pi} \left(1 + \ln \frac{96\pi}{0.55} \right)$$

$$= 1.02396 .$$

$$Z_o = \frac{60}{\sqrt{6.4216}} \ln\left(8 . \frac{1}{1.02396} + 0.25 \times 1.02396 \right)$$

$$= 49.44\ \Omega \approx 50\ \Omega .$$

So, choose

$$W = 24\ mils.$$

The delay line design :

Let

$$L_o = 20\ mils,$$

$$R_o = 500-20 = 480\ mils,$$

$$\theta = 0 \rightarrow 6\pi$$

$$R_o = |R(6\pi)| = 6\pi\gamma = 480\ mils$$

$$\gamma = \frac{480}{6\pi} = 25.46\ mils/rad.$$

$$d_o = \gamma\pi = 80\ mils \succ 3\ h.$$

The total length of this double spiral line is

$$L = 2\int_0^{6\pi}\gamma\theta d\,\theta = \gamma\theta^2|_0^{6\pi} = 9046\ mils = 9.046\ inch$$

The delay time is

$$T_d = 0.0847\sqrt{9.6}\ x\ 9.046 = 2.374\ ns.$$

(2) Thick-film delay line fabrication

The author made thick-film delay lines using the screen printing process. The metal, usually silver, which eventually forms the microcircuits, is initially available to the circuit manufacturer as a paste or ' ink ' contained in a jar. Du Pont 6160 silver paste was used. Some of this paste is positioned on a pine-mesh ' screen ', with areas open for the circuit pattern, and some of the paste is squeezed through these open areas and on to the surface of a substrate held rigidly just beneath the screen . Settling, drying, and firing sequences complete the ' thick ' deposit, a very high proportion of which is pure metal. Although the process has earned the generic name ' thick film ', the actual fired film thicknesses are only usually about 0.55 mils.[75]

The conductor pastes have to be special ' fritless ' materials, and manufacturers such as Du Pont or Ferro or Electro-science Labs provide such pastes. For substantial shelf life, pastes should be kept in a refrigerator. The artwork is prepared and photographic processing is then used to obtain a positive transparency. Suitable screens are available, often with dimension 15 x 15 cm and made of at least 305 mesh stainless steel or polyester. These are stretched to ' drum tightness ' over a rigid frame which will fit into the screen printer. They are coated with a suitable emulsion (for example, Ulano CDF-4) layer.

The positive transparency is held in intimate contact (image side) with the coated surface of a screen, and then standard ultraviolet exposure and wash-and-dry processes leave a screen with apertures for the required circuit. All other areas of the screen are then opaque with a durable emulsion layer.

This screen is placed in a printing jig (preferably a machine with well-defined adjustments and controls). The substrate which is to receive the circuit is placed beneath the aperture region of the screen - with accurate registration. This substrate is held firm by vacuum suction.

A few millilitres of conductor paste are placed on the screen, between the squeegee and the aperture region.

When the parameters have all been optimized, the aperture region of the screen is wet, and the action smooth, a satisfactory wet deposit of paste will be transferred on to the substrate.

This ' wet-circuit ' deposit must next be left to settle by placing it horizontally in a clean area for about 15 min. Following this it is dried at approximately 100° C for about 15 to 20 min under an infrared drying unit.

The firing process returns the material of the deposit to a predominantly metallic substance of high electrical conductivity. A four zone belt furnace was used. The setting temperatures of the different zones were 750° C, 860° C, 860° C, and 650° C.

4.3 Copper Lines on Teflon-Ceramic Composite Substrate

Duroid 6010 was used to make the delay lines.

$$\epsilon_r = K = 10.5,$$

$$h = 20\ mils,$$

$$t = 1.34 \; mils,$$

$$a \times b = 1000 \times 1000 \; mils.$$

Using the same method as described in section 4.2, we can obtain the results below :

The width of strip lines W

$$W = 15 \; mils,$$

The total length of double spiral line L

$$L = 9.046 \; inch,$$

The delay time T_d

$$T_d = TL = 0.0847\sqrt{10.5} \times 9.046 = 2.483 \; ns.$$

The delay line patterns were chemically etched by using standard copper etchant.

4.4 Electrical Measurements and Discussion of Results

In section 2.6 we mentioned that the delay time is obtained by TDR measurements. TDR is a good way to measure the delay time. But here we will use another method, Time Domain Transmission (TDT) measurements, to obtain the delay time. The TDT measurement system consists of a generator, two reference transmission lines in which only the principal mode can propagate, and an oscilloscope voltmeter as shown in Figure (4-1). Between two transmission lines, there are insertion terminals which allow to place the device to be measured. Before measuring, these two transmission lines are connected with each other and in the oscilloscope voltmeter will appear a step waveform (if the step generator is applied). Then, the delay line is placed in the insertion terminals and in the oscilloscope voltmeter will appear another step waveform which shifts an intermission corresponding to the first step waveform. This time intermission is the delay time (T_d) of the delay line.[76]

The insertion loss (S_{21}) of the delay line is measured using HP 8510 Network Analyzer as described in section 2.6.

Figures (4-2), (4-3) are the measurement results for thick-film printing (silver) delay line and Copper lines Teflon-Ceramic delay line, respectively. The delay time measurement results and calculation results are shown in table (4-1). The results indicate that the delay time depends on the substrate material dielectric constant and the delay line's total length. It does not depend on the width of the strip line. Two delay lines have the same line's length but Teflon's dielectric constant (10.5) is larger than alumina's (9.6) so that delay time of copper lines on Teflon-ceramic (2.7 ns) is larger than that of silver lines on alumina substrate (2.62 ns). The delay time's measurement values are larger than calculation values by about 0.2 ns for both of them.

Figures (4-4) ~ (4-6) show the insertion losses (S_{21}) of these delay lines. Samples 1 and 2 (Copper lines on Teflon- ceramic) are better than sample 3 (silver lines printed on alumina). Both of them have the reasonable small insertion losses at frequencies of 0 ~ 10 GHz.

4.5 Conclusion

This work has demonstrated 2.6 ~ 2.7 ns thick-film microstrip delay lines 9 inches long configured on one square inch alumina, and Teflon-ceramic composite substrates. The thick-film silver strip lines were printed on alumina successfully. The copper lines on Teflon-ceramic composite substrate were built by etching using standard copper etchant. These two kind of delay lines have reasonable small insertion losses and would operate at frequencies of 0 ~ 10 GHz quite well.

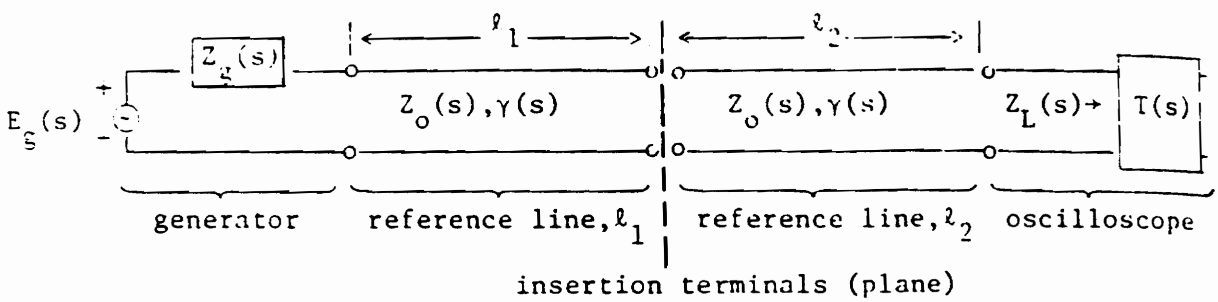
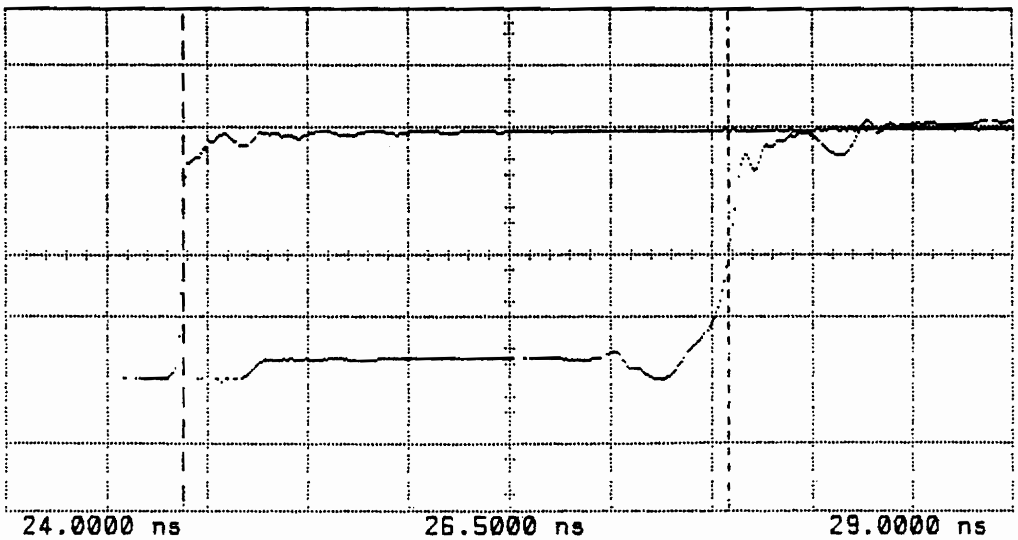


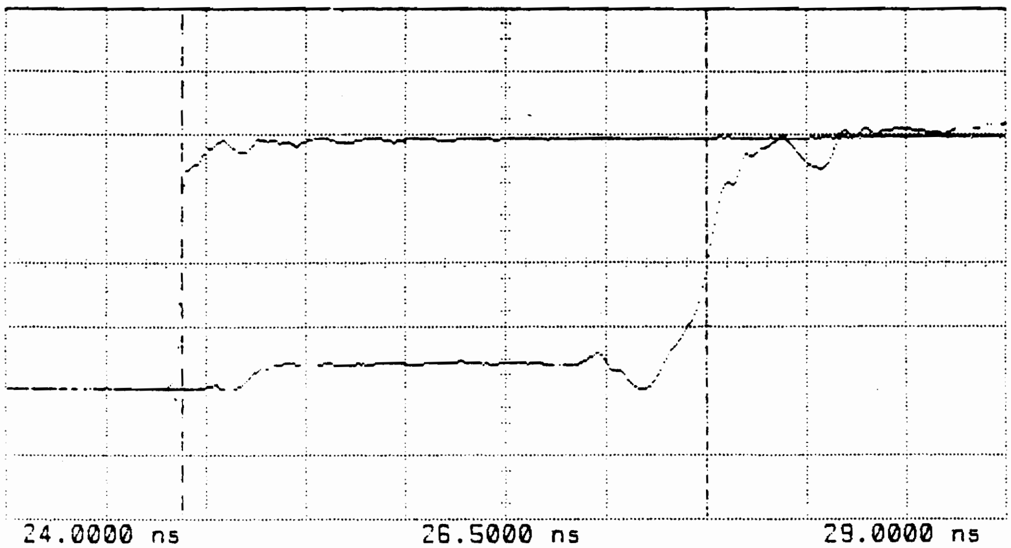
Figure (4-1) TDT system



Ch. 1	=	80.00 mVolts/div	Offset	=	-271.3 mVolts
Ch. 2	=	50.00 mVolts/div	Offset	=	100.0 mVolts
Timebase	=	500 ps/div	Delay	=	24.0000 ns
Memory 1	=	50.00 mVolts/div	Offset	=	100.0 mVolts
Timebase	=	500 ps/div	Delay	=	24.0000 ns
Delta T	=	2.7000 ns			
Start	=	24.8800 ns	Stop	=	27.5800 ns

Triqqer is Freerunninq at 500 kHz with Step on

Figure (4-2) The copper lines on Teflon-ceramic delay line delay time measurement



Ch. 1	=	80.00 mVolts/div	Offset	=	-271.3 mVolts
Ch. 2	=	50.00 mVolts/div	Offset	=	100.0 mVolts
Timebase	=	500 ps/div	Delay	=	24.0000 ns
Memory 1	=	50.00 mVolts/div	Offset	=	100.0 mVolts
Timebase	=	500 ps/div	Delay	=	24.0000 ns
Delta T	=	2.6200 ns			
Start	=	24.8800 ns	Stop	=	27.5000 ns

Trigger is Freerunning at 500 kHz with Step on

Figure (4-3) The silver lines printed on alumina substrate delay line delay time measurement.

Table (4-1) Delay line's delay time comparison (Silver lines on alumina, copper lines on Teflon, calculation and measurement)

	silver lines printing on alumina	copper lines on Teflon-ceramic
calculation	2.374 ns	2.483 ns
measurement	2.62 ns	2.7 ns

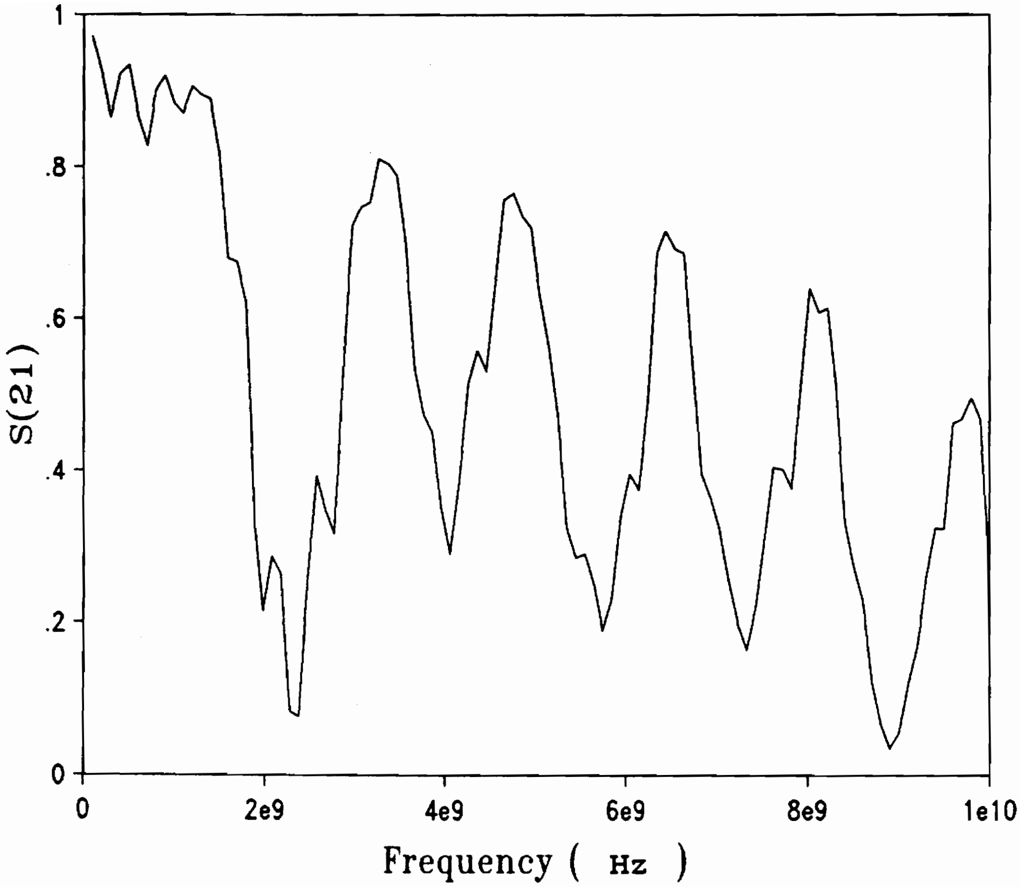


Figure (4-4) The delay line insertion losses (S_{21}) measurement (copper lines on Teflon-ceramic composite substrate).

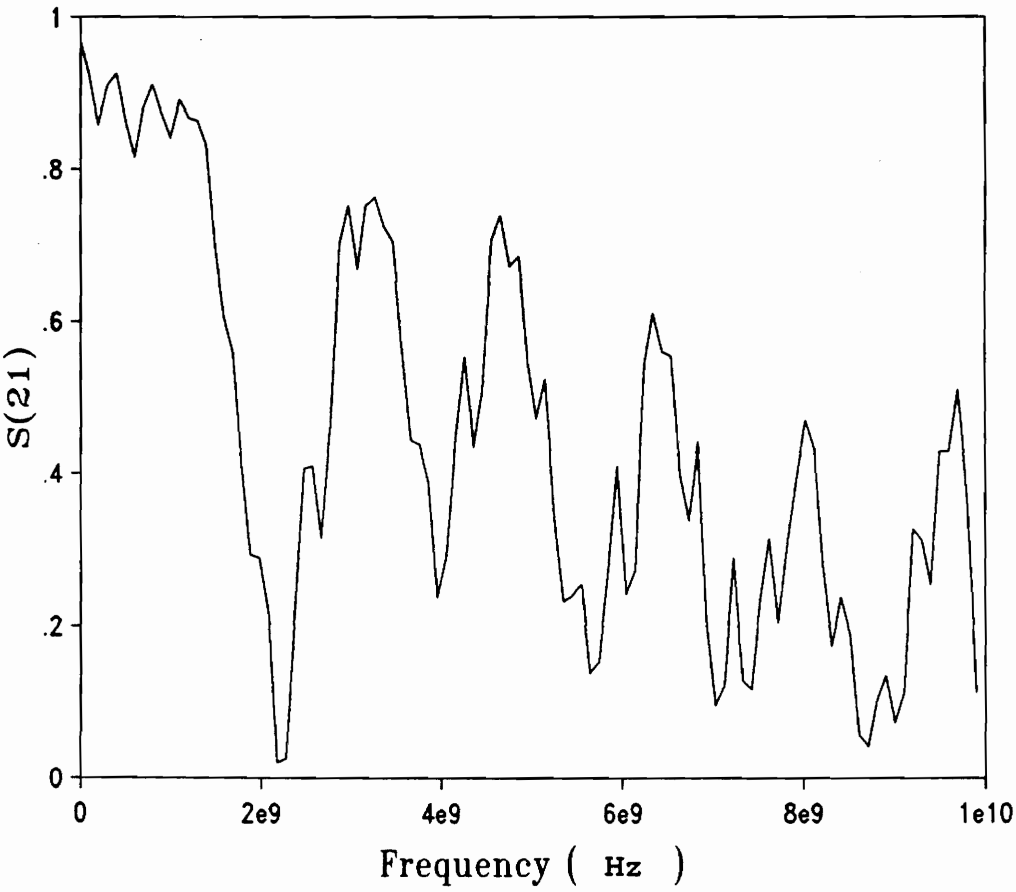


Figure (4-5) The delay line insertion losses (S_{21}) measurement (copper lines on Teflon-ceramic composite substrate).

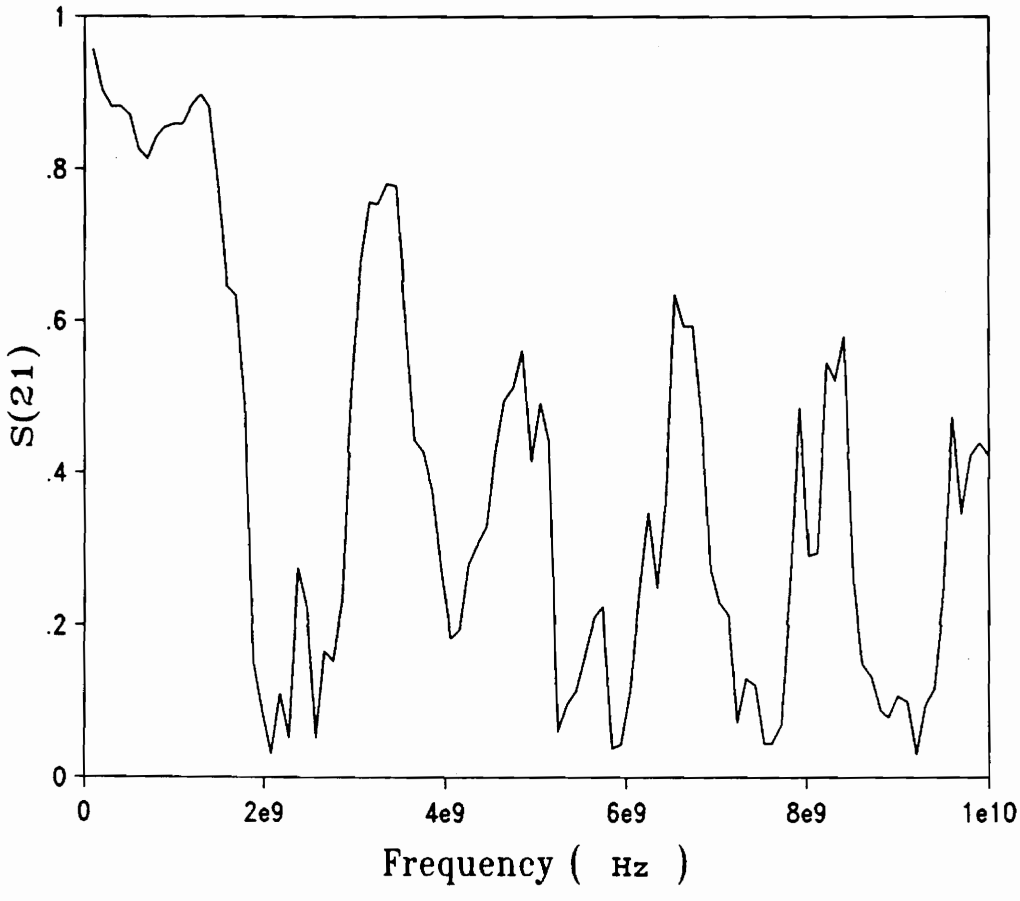


Figure (4-6) The delay line insertion losses (S_{21}) measurement (silver lines printed on alumina substrate).

Chapter V

Conclusions and Recommendations

This thesis addresses the realization of thin films sputtered YBaCuO on various substrate materials in order to realize superconducting microstrip spiral delay lines.

1. Superconducting thin-films of $YBa_2Cu_3O_x$ have been successfully sputtering on Alumina (Al_2O_3), Aluminum-Nitride (AlN), Silicon (Si), Zirconia (ZrO_2), Yttriastabilized Zirconia (SYZ), and Magnesia (MgO) substrates. The onset temperature varies from 98 to 105° K and the critical temperature (Tc) varies from 90 to 95° K for Zirconia and Magnesia substrates. For Alumina, Aluminum-Nitride, and Silicon substrates, the buffer layer is necessary. The ZrO_2 material was used as a buffer layer, and the thickness of about 5.4 μm (sputtering for one hour) is sufficient..

2. The sputtering temperature, gas pressure, substrate position and orientation with respect to the target, and annealing temperature are very important parameters and have to be controlled very carefully in order to have successful results of sputtered films.

3. The substrate surface has to be processed. The most likely best process to prepare the substrate surface for thin film superconductor growth is thermal annealing.

4. All the necessary procedures to achieve superconductor delay lines were followed. The work has successfully etched patterns in YBaCuO superconducting thin films using EDTA solution in water and standard photoresist lithography. No measurable degradation of the superconducting transition temperature is observed (see Figure (3-17)). The solution selectively etches YBaCuO films and appears totally nonabrasive with respect to the substrate materials.

5. 2.6 ~ 2.7 nanosecond silver and copper thick-film microstrip delay lines 9 inches long configured on one square inch substrates have been demonstrated. The silver thick-film strip lines were printed on alumina substrates. The copper lines on Teflon-ceramic composite substrate (Duroid 6010) were built by etching using standard copper etchant. These delay lines possess reasonable small

insertion losses at frequency of 0 ~ 10 GHz.

6. The superconductor thin film quality is a very crucial requirement for superconductor delay lines. The multilayer RF magnetron sputter-deposition of Y_2O_3 , $BaCO_3$, and CuO is a good procedure to prepare high quality superconductor thin films. $LaAlO_3$ substrates are much better for microwave applications. There is no difficult to make various superconductor microwave devices like microwave delay lines, microwave resonators, fin line filters if one get high quality superconductor thin films.

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