WIDEBAND ELECTRICAL CHARACTERIZATION OF MULTILAYER LOW-LOSS DIELECTRIC MATERIALS

by

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(ABSTRACT)

The objective of this thesis is to conduct research work on wideband characterization of the complex dielectric constant of low-loss dielectric materials used in multilayer structures. The materials include Pyralux®, a polymer, and Green Tape^{TM}, a low-temperature co-fireable tape ceramic (LTCC). The study is composed of three main tasks. First, the effects of operating temperature and relative humidity on the complex dielectric constant of these materials, at low frequencies, will be investigated. Next, the complex dielectric constant of these materials will be determined for high frequencies, under ambient conditions. Finally, lifetime tests will be performed in order to determine the effects of aging on the complex dielectric constant of these materials, at low frequencies.

Since experiments will be performed at both low and high frequencies, both capacitor and T-resonator microstrip structures will be used. Analysis of the low frequency environmental test results indicate that both the dielectric constant and the dissipation factor of the polymer increase with both temperature and relative humidity. The results for the tape ceramic indicate that the dielectric constant and dissipation factor increase with temperature, but decrease with rising humidity. The results of the high frequency testing are compared to earlier experimentation in order to evaluate several test structures and techniques. Results of the aging experiments indicate that both the dielectric constant and the dissipation factor of the polymer are increasing over time. Conversely, both the dielectric constant and the dissipation factor of the tape ceramic are decreasing over time.

ACKNOWLEDGEMENTS

This section is dedicated to all of the individuals who have assisted me in the completion of this thesis. First, I would like to thank my principal advisor, Dr. A. Elshabini-Riad, for her valuable support and guidance throughout this project. I would also like to thank my other committee members, Dr. S.M. Riad and Dr. W.A. Davis for their constructive input and suggestions. In addition, I would like to thank DuPont Electronics for their sponsorship, as well as Dr. Wilson Whitehead and my fellow colleagues at DuPont Electronics in RTP for the valuable learning experience that they provided me during my summer employment. I would also like to thank the many members of the Microelectronics and Time Domain Laboratories who have supported me with their thoughtful advice and assistance. I would like to thank my best friend, Adele Mountz of the Biochemistry Department, for her caring companionship during my undergraduate and graduate work at Virginia Tech. Finally, special thanks go to my parents, Ronald J. and Betty R. Addington, for their loving support and encouragement throughout my education.

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CHAPTER 1

INTRODUCTION

The objective of this research is a wideband characterization of the complex dielectric constant of two DuPont low-loss dielectric materials used in multilayer structures - Pyralux® and Green Tape^{TM}. Pyralux® is a polyimide-based system that utilizes layers of copper, Kapton® (the polyimide), and WA (adhesive) materials. Through selected etching of the copper layers, and the correct application of heat and pressure, these materials may be laminated together to form many types of flexible structures and circuits. Green Tape^{TM} is a low temperature co-fireable tape ceramic (LTCC) system that combines traditional thick-film processing with the more recent multilayer techniques. Green Tape^{TM} is a dielectric tape on which patterns may be screen-printed using specially formulated pastes. Layers of printed Green Tape^{TM} are laminated together using a combination of heat and pressure to form a flexible circuit. Interconnections between layers are made possible through vias (small-diameter holes in a tape layer that are filled with a special conductive paste). Unlike Pyralux®, the flexible Green Tape^{TM} laminate is subsequently burned-out and fired to produce a rigid structure, similar to a pre-fired alumina substrate.

As outlined earlier, this research study is composed of three main tasks, organized into six chapters. Chapter 1 is a general introduction. Chapter 2 presents a literature survey of published work in the area.

Chapter 3 investigates the effects of various environmental operating conditions on the complex dielectric constant of these materials at low frequencies. This chapter also includes a detailed outline of the processing of Pyralux® and Green Tape^{TM} circuits, as well as a discussion of the designed and in-house constructed equipment required to accurately perform these measurements.

The manufacture of the polymer laminate begins by blanking out 3" x 3" squares of 1 oz. copper, Kapton® (the polymer material), adhesive, and copper-clad Kapton®, according to the specifications of the structure. Since experiments will be performed at both low and high frequencies, capacitor and T-resonator structures will be implemented through selected etching of the copper. Various thicknesses of Kapton® are used to build up the dielectric layer of the structures. The manufacture of the Green Tape^{TM} laminate begins by blanking out 3" x 3" squares of the tape ceramic. A special silver paste is screen-printed on the blanks to form the upper and lower electrodes of the capacitor array, and the T-pattern and ground plane of the resonator structure. For both materials, connection between the bottom electrodes of the capacitor array and the upper surface is provided through conductor-filled vias, so that only the upper surface of the laminate needs to be probed for measurement purposes.

For the environmental testing, a special lazy-susan test fixture was designed and constructed. This device eliminated the need to open the environmental chamber door, thus disturbing the set environmental test conditions, in order to perform the proper calibrations and sample measurements. The environmental chamber was set to various temperature and relative humidity combinations to expose the samples to a range of conditions. Both open circuit and short circuit calibrations were performed, and all measurements were made using an impedance analyzer.

Chapter 4 determines the complex dielectric constant of the two materials at high frequencies, under ambient conditions. For the high frequency testing, Pyralux® and Green Tape^{TM} T-resonator microstrip structures were analyzed. Due to the acute sensitivity of high-frequency measurements, it was very difficult to repeat the environmental tests, without seriously interrupting the set conditions. Thus, to maintain repeatability, these measurements were performed at ambient conditions.

Chapter 5 analyzes the effects of aging on the complex dielectric constant of Pyralux® and Green Tape^{TM} at low frequencies. In this chapter, both long-term (natural) aging, as well as short-term (forced) aging are investigated and compared. In order to test the natural aging of the materials, polymer and tape ceramic laminates were constructed and subsequently enclosed in a small desiccant chamber under vacuum conditions. Over a span of several months, in two week intervals, these laminates are removed from the chamber, tested, and immediately returned to the chamber. To further simulate the lifetime testing, additional laminates are forced to undergo rapid aging through thermal cycling.

Finally, Chapter 6 provides a general summary of the research work.

CHAPTER 2

LITERATURE REVIEW

2.1 INTRODUCTION

The purpose of this chapter is to highlight other studies that are of interest to this research work. As mentioned in Chapter 1, there are three main areas addressed in this study. Two of these topics, environmental testing and high frequency testing, are discussed in literature more often than the third, lifetime testing. Section 2.2 surveys the research work conducted in the area of environmental testing. Section 2.3 discusses research work involving high-frequency electrical characterization.

2.2 ENVIRONMENTAL TESTING

Reference [2], by D.D. Denton, et al, is an extensive discussion of moisture diffusion in polyimide films. Although the current research does not involve a study of the diffusion process for Pyralux® and Green Tape ^{TM} materials, it is useful to understand some of the theory that underlies the environmental effects observed in Chapter 3. According to Denton, a change in the capacitance value is proportional to a change in the moisture content. Thus, the capacitance element as a function of time may be modeled using a one-dimensional moisture diffusion coefficient. One limitation of this model, however, is that it deviates somewhat for polyimide films of greater thicknesses, due to several one-dimensional assumptions.

Reference [1], by H. Sadri, will be highlighted for two reasons. First, it demonstrates the effect of temperature on the complex dielectric constant of aluminum nitride, a ceramic material. Secondly, the paper provides an example of the difficulties involved in performing environmental measurements at high frequencies. Again, it will be useful to introduce some theory behind the environmental effects observed in Chapter 3. According to Sadri, the increase in the dielectric constant upon an increase in the temperature is attributed to an increased polarizability due to expansion of the material. In addition, the observed increase in the dissipation factor upon an increase in the temperature is attributed to losses associated with the softening of residual glassy phases in the material. These measurements were performed at frequencies in the microwave range using free-wave transmission and reflection methods. However, this procedure requires that the sides of the oven, containing the test sample, be opened, thus disturbing the set temperature conditions. This result illustrates the difficulty of performing accurate environmental measurements at high frequencies.

2.3 HIGH-FREQUENCY ELECTRICAL CHARACTERIZATION

References [3], [4], [6], [7], and [9] illustrate the significant amount of research that has been devoted to the microwave characterization of thick-film and multilayer materials. These studies have utilized a variety of test structures and measurement techniques. For example, a dielectric filled cavity has been conceived to provide wideband characterization of dielectric materials in the microwave region. Terminating and through cavity configurations have been designed and tested in both the time-domain as well as the frequency-domain. The cavity structure is modeled as a given geometry of the test material surrounded by a finite-conductivity boundary. Using this model, the measured transmission and reflection signals are then related to the characteristics of the dielectric material. In addition, various simulation techniques have been developed to model striplines in both the time-domain and the frequency-domain in order to characterize dielectric materials at high frequencies. Reference [9], by W. Su, et al, uses line resonators and ring resonators to determine the complex dielectric constant of Pyralux® at high frequencies, under ambient conditions. A line resonator consists of a linear section of stripline (or microstripline) that is separated from two launching-probe regions, at each end, by small gap discontinuities. Resonance occurs when the length of the linear section is an integer multiple of the half-wavelength of the (micro)stripline. The ring resonator, on the other hand, is simply a line resonator that has been bent into a closed circle. Again, small coupling gaps separate the ring from two linear launching regions. Resonance occurs when the circumference of the ring is an integer multiple of the wavelength of the (micro)stripline. Although the ring structure removes the end effects (i.e. fringing) that plague the line resonator, difficulties arise in defining the circumference length of a ring having a finite line thickness. Results obtained from these structures will be compared to the results obtained from the current research that will utilize the T-resonator. The T-resonator holds several advantages over the line and ring. First, the design of the T-resonator is very simple, and thus may be easily processed in both Pyralux®

and Green Tape^{TM}. The design of line and ring resonators, on the other hand, involves critical gap dimensions and line widths in order to obtain accurate results. In addition, the measurement technique associated with the line and ring resonators involves the construction of several samples, having different line and dielectric dimensions, in order to obtain data on the loss tangent. The T-resonator requires only one design if appropriate approximations are made [7]. It also appears that a typical waveform obtained from a T-resonator is much clearer than one obtained from the line or ring. A comparison of the data obtained from the three resonator structures will demonstrate that all three resonator types provide equally reliable results.

2.4 CONCLUSIONS

This chapter has presented a review of other studies which are relevant to this research work. A survey of environmental testing research has provided theories which may explain the results obtained in the current study. A review of high-frequency electrical characterizations has provided alternative measurement techniques, as well as various results to which the current data may be compared.

CHAPTER 3

ENVIRONMENTAL TESTING AT LOW FREQUENCIES

3.1 *INTRODUCTION*

Dielectric materials such as Pyralux® and Green Tape^{TM} have a wide variety of uses, and thus become exposed to many different environmental conditions. The purpose of this chapter is to study the effects of operating temperature and relative humidity on the complex dielectric constant of these materials at low frequencies. The parameters of interest in this experiment are the standard quantities involved in most tests of this nature. Environmental studies almost always include varying temperatures and relative humidities, although dew point temperatures sometimes replace the relative humidity value. Also, studies of dielectrics tend to focus on the relative permittivity and loss tangent as the characteristic parameters of the material. The decision of this study to opt for temperature and relative humidity as variables is based on the familiarity of and the ease with which laboratories and industries can relate to (and measure) these quantities.

3.2 *BACKGROUND*

It is not surprising that recent studies have focused on the environmental effects on polymer materials, such as Pyralux®, rather than on ceramics such as Green Tape^{TM}. This is mainly due to the sensitive nature of the polymer as opposed to the more sturdy reputation of ceramics. According to a report on the properties of aluminum nitride over a wide temperature range [1], almost all polycrystalline ceramics experience an increase in the dielectric constant value and loss tangent upon an increase in the operating temperature. Although this data was taken at very high frequencies (35-45 GHz) and over a much larger temperature range, it is useful as well as interesting to see if similar trends develop for the Green Tape^{TM} material, at low frequencies. Judging from the lack of studies on the effects of moisture on ceramics, one may expect to find a negligible effect of humidity on the dielectric properties of Green Tape^{TM}. On the other hand, extensive work has been done in the area of characterization of polyimide materials under various environmental conditions. [2] The relative findings of this report indicate an increase in the dielectric constant value upon an increase in the humidity (at a fixed temperature). However, no data has been provided for increasing temperature (at a fixed relative humidity), and no consideration is given to loss tangent variations.

3.3 PROCEDURES

3.3.1 MATERIAL PROCESSING

Before conducting any experimentation, it is necessary to first become familiar with the processing of Pyralux® and Green Tape^{TM} materials. This is a two-fold procedure, in that the processing steps must correlate closely with those prescribed by the DuPont procedure manuals, and yet be flexible enough to account for processing in a different laboratory, having different equipment. General processing steps will be discussed in the following sections; however, these processing lists are explained in full detail in Appendices A and B. [3]

3.3.2 EXPERIMENTAL DESIGN

Since these experiments will be performed at low frequencies (100 kHz and 1 MHz), capacitor laminates with varying dielectric thicknesses are designed and constructed with the Pyralux® and the Green Tape^{TM} 851-AT systems. The manufacture of a Pyralux® laminate begins by blanking out 3" x 3" squares of 1-oz. copper, Kapton®, WA (adhesive), and copper-clad Kapton®, according to the specifications of the capacitor structure. Various thicknesses of Kapton® and WA are available, and are used to build up the dielectric layer of the capacitor. Copper will form the upper and lower electrodes. The squares are stacked in the correct arrangement, placed in a laminating die, and then exposed to heat and pressure. One of the copper sides of the laminate is then selectively etched to form an array of four upper electrodes so that data is obtained from four capacitors instead of just one. A sample containing four capacitors, under the same set of environmental conditions, increases the level of confidence in the experimental results. The Pyralux® capacitors used in this experiment have dielectric layers consisting of 1 mil, 2 mil, and 5 mil Kapton® layers, each sandwiched between two 1 mil WA layers. The size of the upper electrodes, 200 mil x 200 mil, is selected so that four electrodes may be etched in the 3" x 3" area with sufficient separation, and fringing effects may still be neglected. [4] So that measurements can be made by probing the upper surface only, the copper regions around the etched upper electrodes are connected to the ground plane by drilling 32 mil diameter vias and filling them with conductive epoxy (Epo-tek P-10). The manufacture of a Green Tape^{TM} laminate begins by blanking out 3" x 3" squares of 851-AT tape. A special silver paste (DuPont 6142D) is screen-printed on the blanks to form the upper and lower electrodes of four capacitors. A connection between the internal ground and the upper surface is again provided by vias, so that only the upper surface of the laminate needs to be probed. The vias are 15.7 mil in diameter after processing and filled with a special via-fill paste (DuPont 6141D). Each tape square will be 3.7 mil thick after processing, and so dielectric thickness may be built up by increasing the number of tape layers between the electrodes. The squares are correctly stacked in a laminating die and exposed to heat and pressure. The resulting flexible laminate must then be burned-out in an ashing furnace, and fired in a multizone furnace to produce the final ceramic laminate which has shrunk by approximately 12% (2.63" x 2.63"). The Green Tape^{TM} capacitors used in this experiment have dielectric layers consisting of one and two layers of 851-AT. The size of the electrodes is again chosen to provide replications and still minimize fringing. The top electrodes are 220 mil x 220 mil pre-fired and 200 x 200 mil post-fired. The lower electrodes are 420 mil x 870 mil pre-fired and 375 mil x 750 mil post-fired. Figure 3.1 illustrates the capacitor structures.

3.4 EQUIPMENT AND MODIFICATIONS

The two pieces of equipment used in the low frequency environmental testing are a commercial Blue-M (model CFR-7652C-2) environmental chamber, and an HP 4192A impedance analyzer. However, this basic arrangement provides no means of performing the calibration and the measurements without opening the chamber door to reposition the probes, and thus disturbing the environmental test condition. A lazy-susan test fixture was designed and constructed to alleviate this problem. [3] The test fixture consisted of a sample holder, capable of holding up to four 3" x 3" samples, and a probe platen, capable of probing four capacitors per sample. The probe is raised and lowered via a manual crank that fits through a small hole in one side of the environmental chamber. A switch, also brought to the outside of the chamber, controls the rotation of the sample holder. A position limit switch, mounted beside the sample holder, stops the rotation of the holder at the location of the next measurement. Coaxial cables connect the probes to the impedance analyzer, again through a series of small holes in the side of the environmental chamber. Figures 3.2-3.4 illustrate this fixture in greater detail.

A typical measurement proceeds as follows. First, the samples, including a calibration sheet (plain copper sheet for the Pyralux® samples, and an alumina substrate screen-printed with silver paste for the Green Tape^{TM} samples), are placed on the sample holder and the environmental chamber is closed. The desired temperature and relative humidity are then set using a pair of wet-bulb and dry-bulb controllers. Stabilization of the chamber, checked using a chart recorder, occurs in approximately 1.5 hours. However, in order to allow stabilization of the samples to the conditions, at least 24 hours are allowed to elapse before making any measurements. According to DuPont literature [5], polyimides can absorb their maximum water content within 30 minutes in a high humidity (> 70%) environment. After this time, the probe is lowered down onto the calibration sheet, and a short calibration is made. Next, the probe is lifted up off the calibration sheet, and an open calibration is performed. The sample holder is then rotated until the first sample to be tested is directly under the probe, at which point the limit switch stops the rotation. The probe is lowered down onto the first sample, and measurements of the capacitance value and the loss tangent value are made for all four capacitor elements on the sample by simply connecting the corresponding pair of coaxial connectors to the impedance analyzer. When all four capacitors on the sample have been measured, the probe is raised, and the holder is rotated as before. This process continues until all samples in the chamber have been measured at that environmental condition. Then, the environmental chamber is set to the next set of environmental test conditions, and the process repeats. Note that due to the shrinkage factor of the Green Tape^{TM} samples, the probe assembly was unable to connect to all four capacitors at the same time. Thus, so as not to sacrifice repeatability of the data by only measuring some of the capacitors, the chamber door is opened for a short time while the measurements are taken manually. Comparison of some of the data obtained with the door closed with those obtained with the door open indicates that, at least for the Green Tape^{TM} material, opening the chamber door for this short period of time does not significantly disturb the stabilized condition of the samples.

3.5 MEASUREMENTS

Measurements of the Pyralux® and Green Tape^{TM} samples were made for an array of operating temperatures and relative humidities (29°C, 50°C, 75°C; 50%, 75%, 95%). The test procedure started with the lowest temperature and lowest humidity settings (29°C/50%). Then, holding the temperature constant, the humidity was raised to 75% and then 95%. Next, the temperature was raised to 50°C, and the humidity was again increased from 50% to 75% and then to 95%. Finally, the temperature was set at 75°C, and the humidity was increased as before. The decision to make the measurements in this order was based on a concern that moisture may linger inside the samples between measurements. Thus, by making the measurements in increasing order of humidity, moisture remaining inside the samples actually aids in the stabilization to the higher humidity level. In addition, concerns about making measurements at 50% humidity after the samples have just been exposed to 95% humidity are quieted by the fact that the 50% humidity case will be at a higher temperature than the previous 95% humidity case. Thus, the samples will again be assisted in the stabilization process as the high humidity is dried up by the increased temperature.

The four capacitance values obtained from each sample are averaged, and the dielectric constant for that sample is determined using Equation 3.1:

$$\mathrm{C} = \frac{\epsilon_r \epsilon_o A}{\mathrm{d}}, \qquad (3.1)$$

where C is the capacitance value, ϵ_r is the relative permittivity (dielectric constant), ϵ_o is the permittivity of free space, A is the area of the upper electrode of the capacitor, and d is the dielectric thickness of the capacitor. The dissipation factor is obtained directly from the impedance analyzer. All the results have been summarized in Figures 3.5-3.24.

3.6 RESULTS

Through careful inspection of Figures 3.5-3.24, a number of trends can be discovered which relate material type and thickness to the operating temperatures, relative humidities, and frequency. Figures 3.5-3.10 show that the dielectric constant of Pyralux® increases with both temperature and humidity. This increase is less however for the laminates having the thicker dielectrics. In addition, as the temperature and humidity get higher, the increase in the dielectric constant becomes more significant. However, at the higher frequency (1 MHz) these trends become slightly less evident. Figures 3.11-3.16 show that the dissipation factor of Pyralux® also increases with both temperature and humidity, and that this increase is again generally less for the laminates with the thicker dielectrics. Note also that the 50% and 75% curves remain relatively close together across the temperature range. That is, compared to the case for the dielectric constant, the increase in the dissipation factor due to an increase in humidity is much more apparent for very high (95%) humidities. As was true for the case of the dielectric constant, these trends also appear to diminish at the higher test frequency, although less significantly. Figures 3.17-3.20 show that the dielectric constant of Green Tape^{TM} increases with temperature, but decreases slightly with rising humidity. Again, these trends are diminished at the higher frequency. Figures 3.21-3.24 show that an the dissipation factor of Green Tape^{TM} increases with rising temperature, and again becomes less apparent at the higher test frequency. The trend for the dissipation factor with respect to humidity is not as clear; however, a slight decrease in the dissipation factor with increasing humidity appears possible. Note that for all of the Green Tape^{TM} figures, there is no noticeable trend related to the thickness of the dielectric.

3.7 CONCLUSIONS

This chapter has addressed the environmental testing of Pyralux® and Green Tape^{TM} materials at low frequencies. Knowledge of the behavior of these materials under various temperature/humidity conditions is critical to the design phase of circuits utilizing these multilayer technologies. Unless this information is taken into account, the circuit design may be limited in its applications. This chapter has also formally introduced a processing scheme for Pyralux® and Green Tape^{TM} circuits, outside the industrial domain. In addition, the difficulties involved in performing low frequency environmental measurements, without disturbing the set conditions, have been alleviated by the design of a custom-made probe assembly.

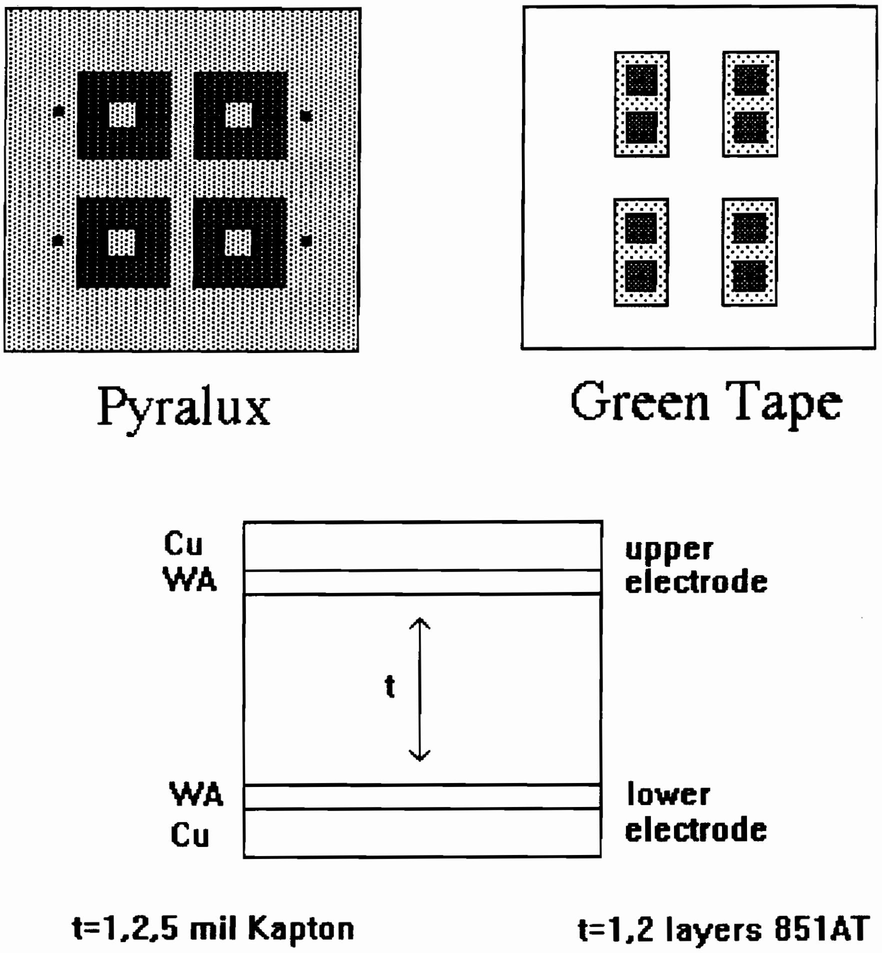


Figure 3.1 - Laminate Drawings

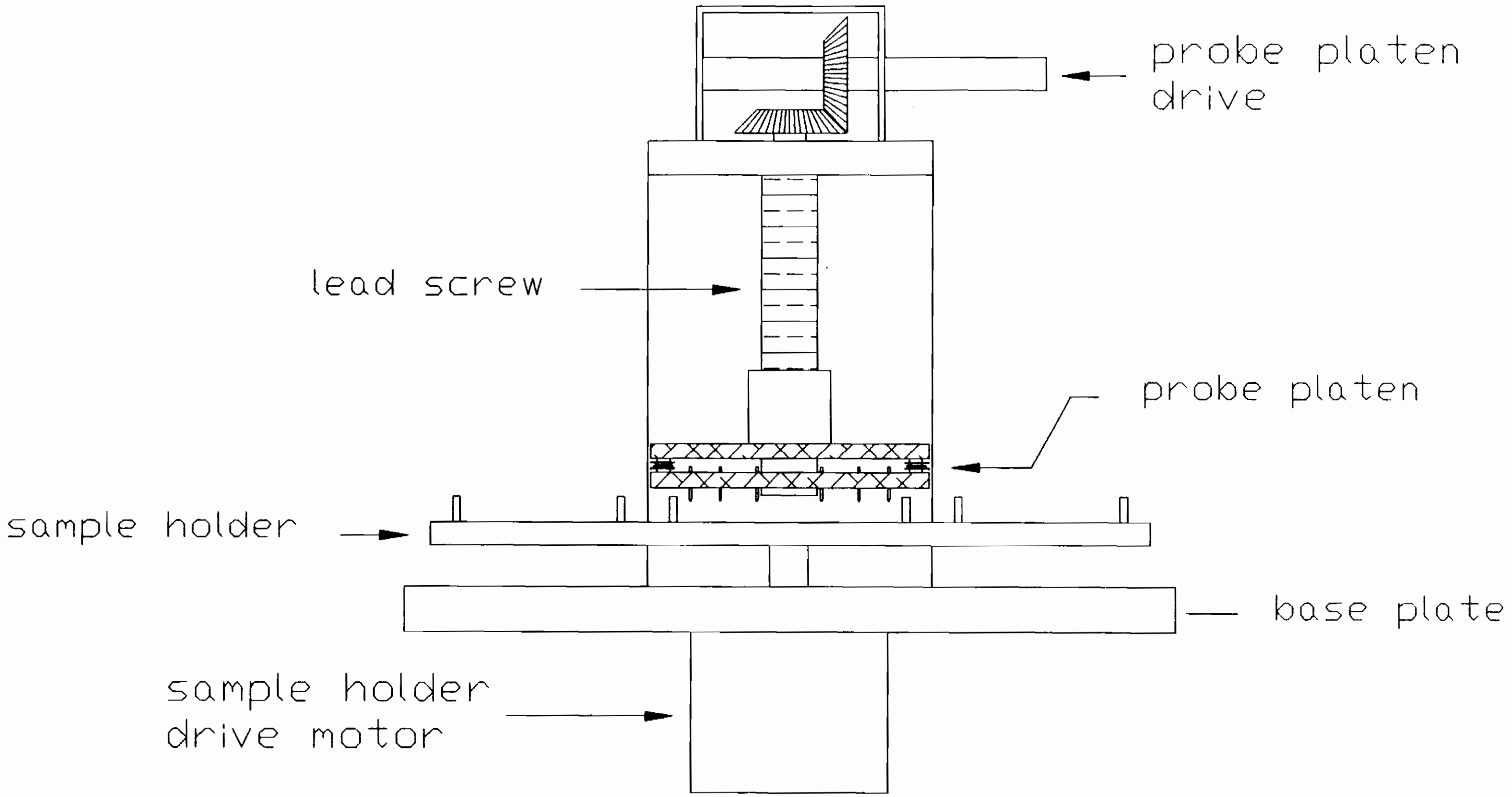


Figure 3.2 - Test Fixture - Sample Changer

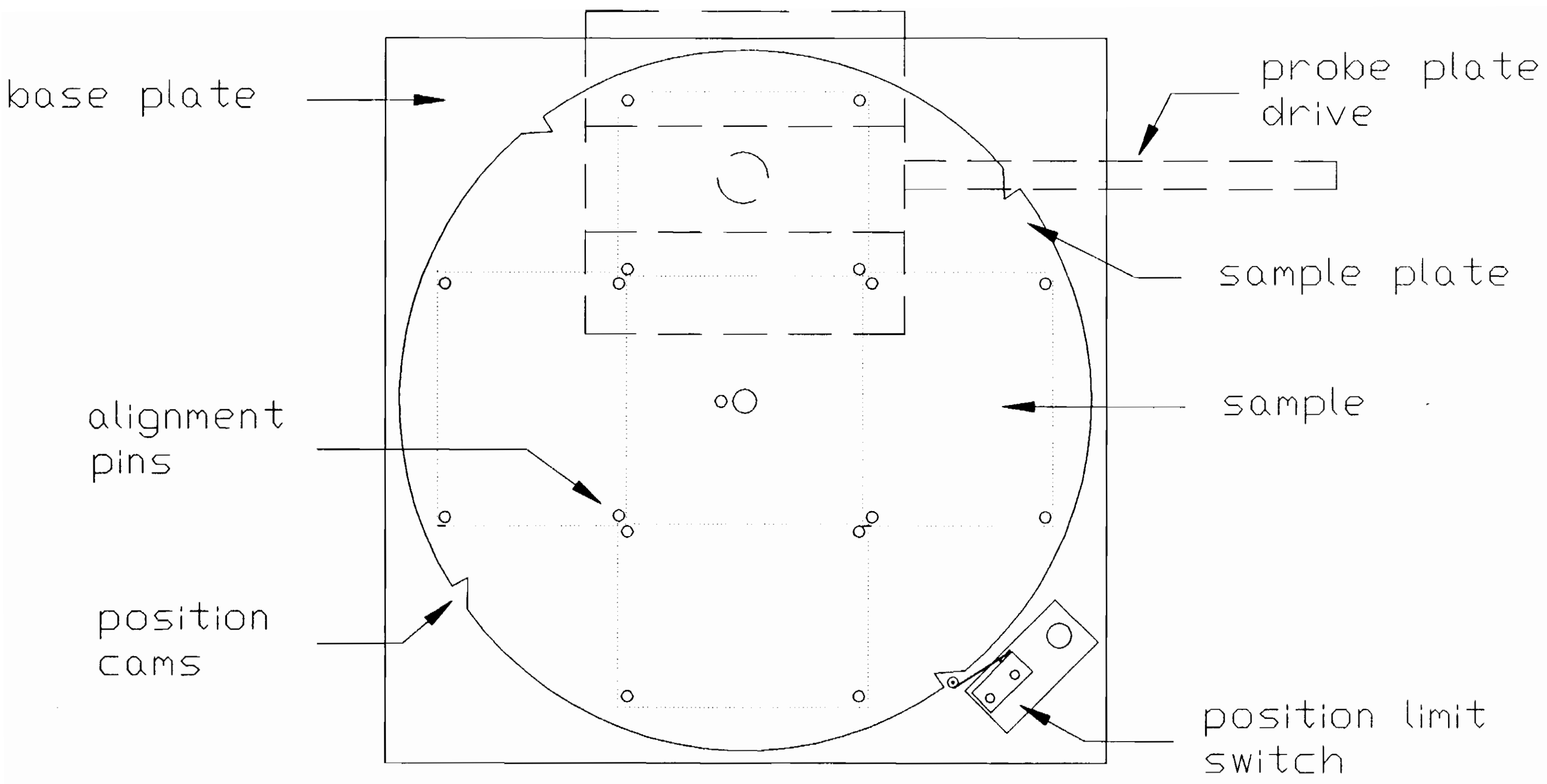


Figure 3.3 - Test Fixture - Sample Changer (Top View)

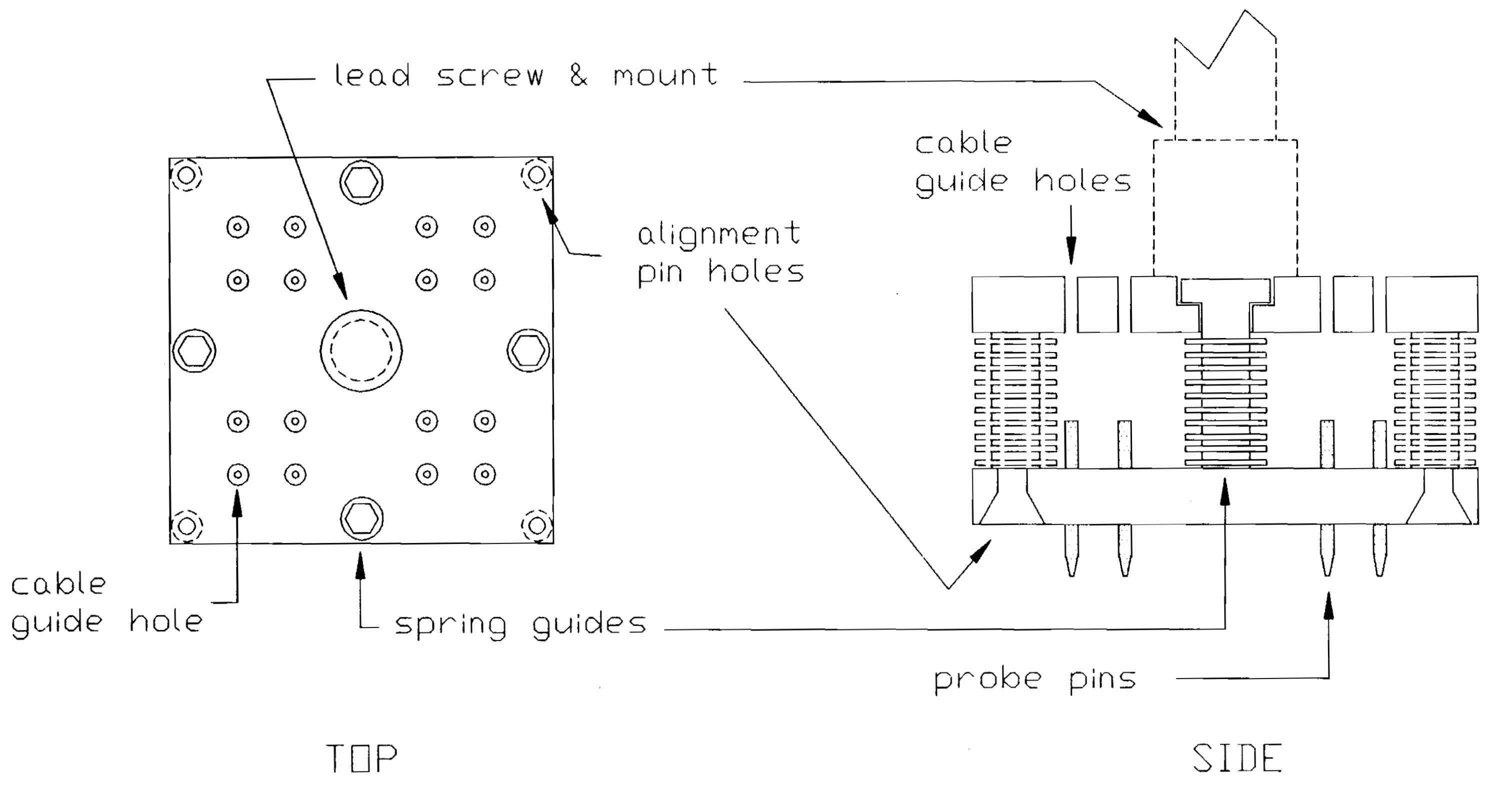


Figure 3.4 - Test Fixture - Probe Platen

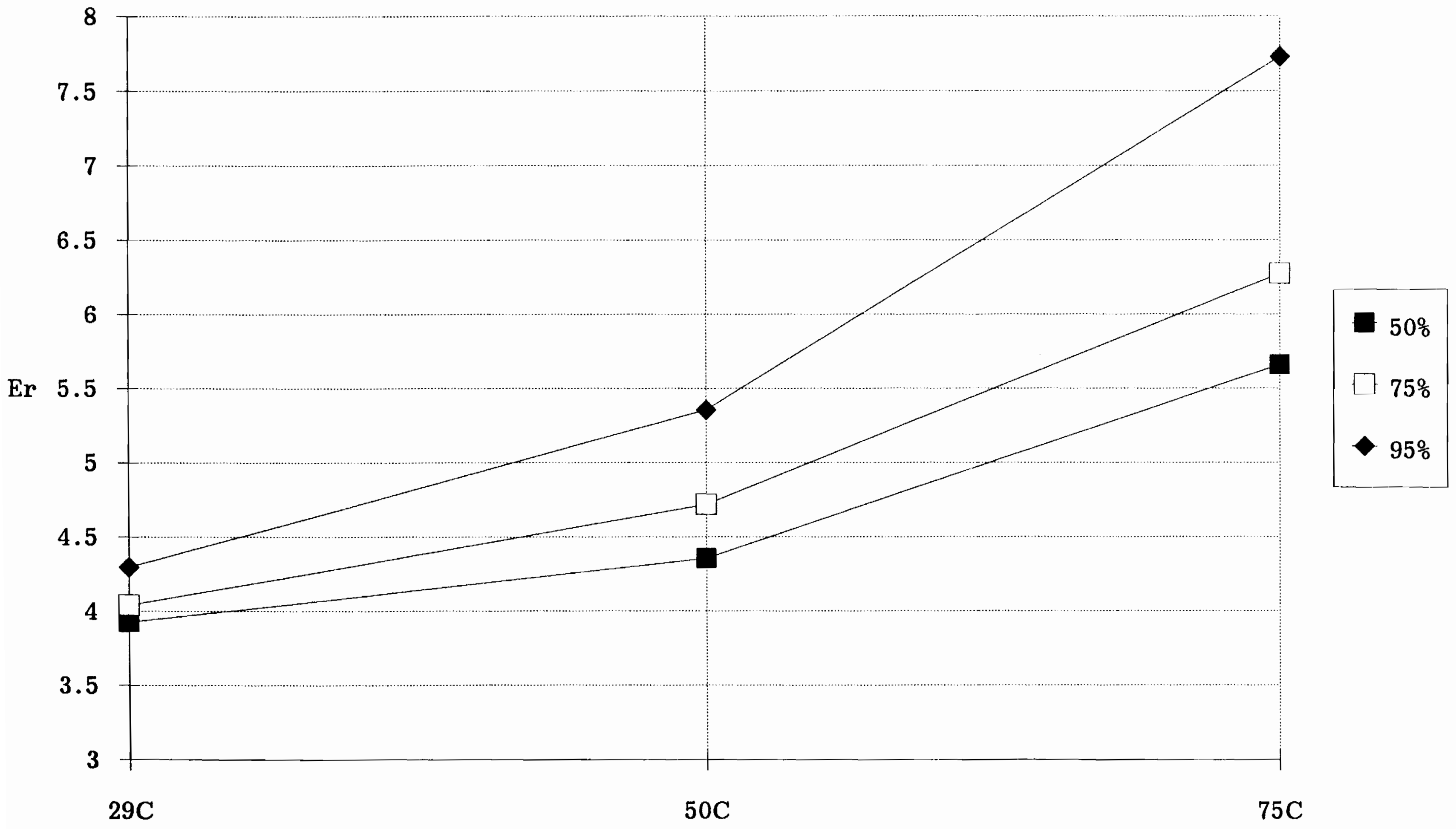


Figure 3.5 - Pyralux Dielectric Constant (d=3 mil, f=100kHz)

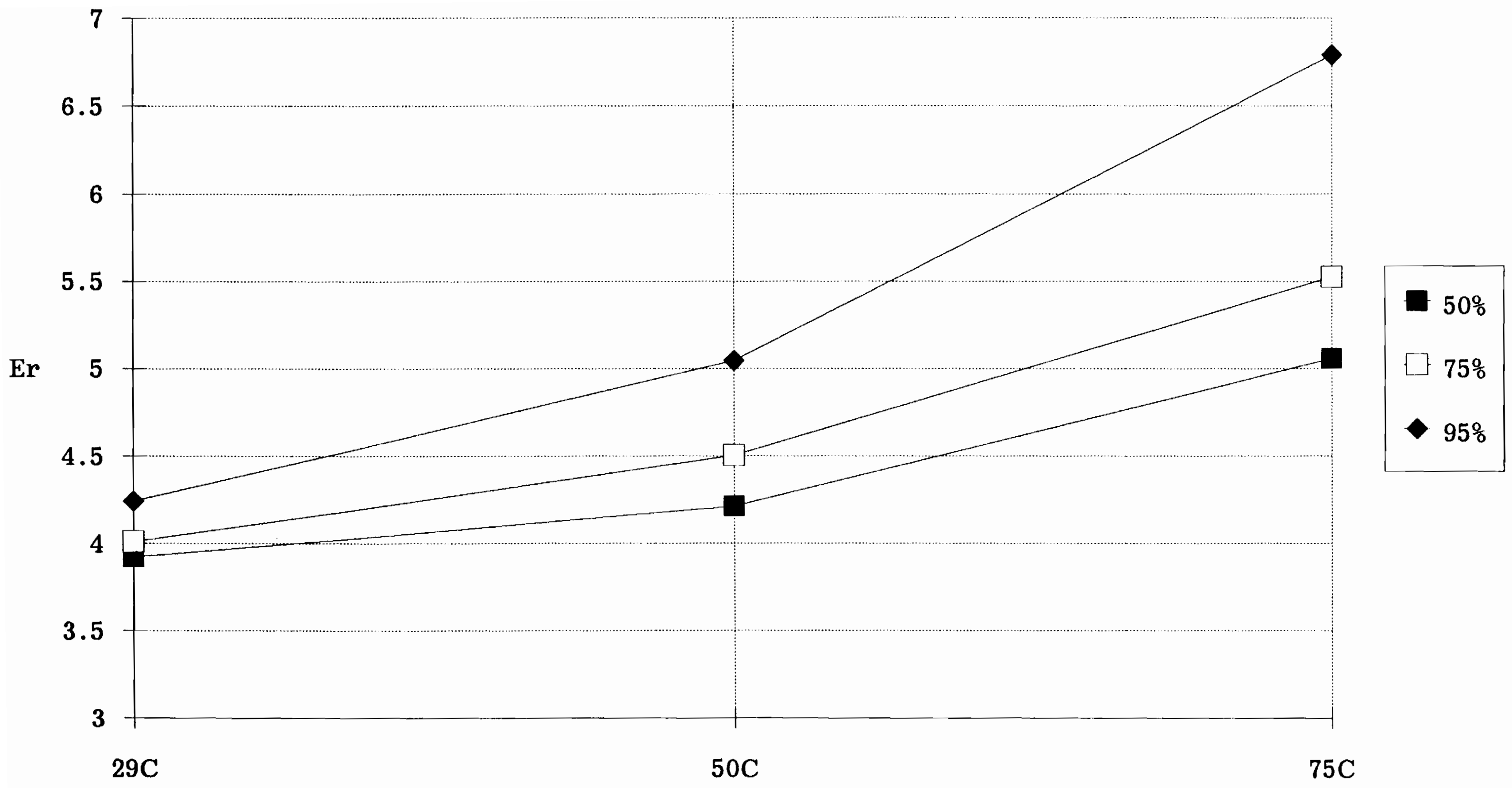


Figure 3.6 - Pyralux Dielectric Constant (d=4 mil, f=100kHz)

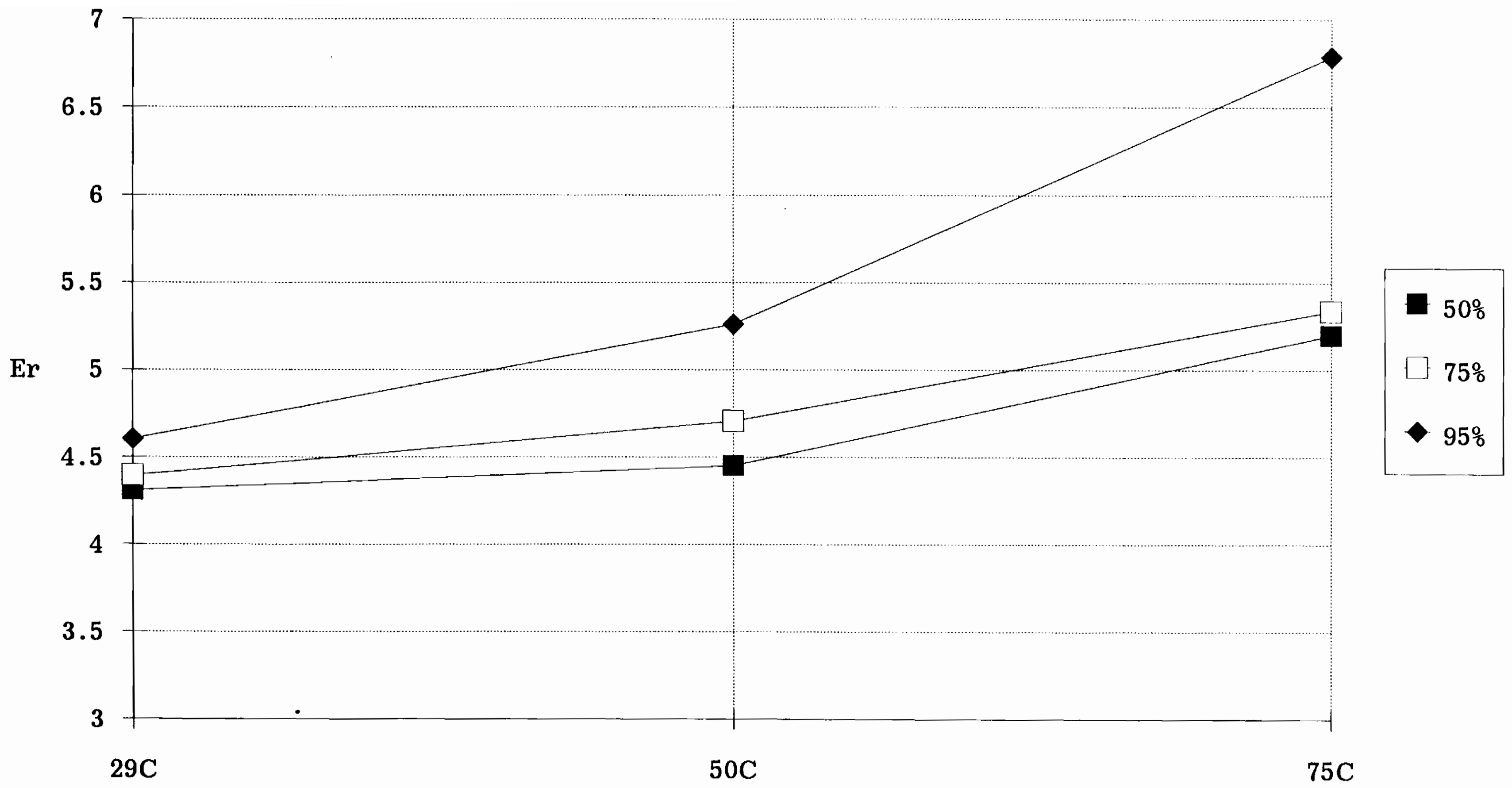


Figure 3.7 - Pyralux Dielectric Constant (d=7 mil, f=100kHz)

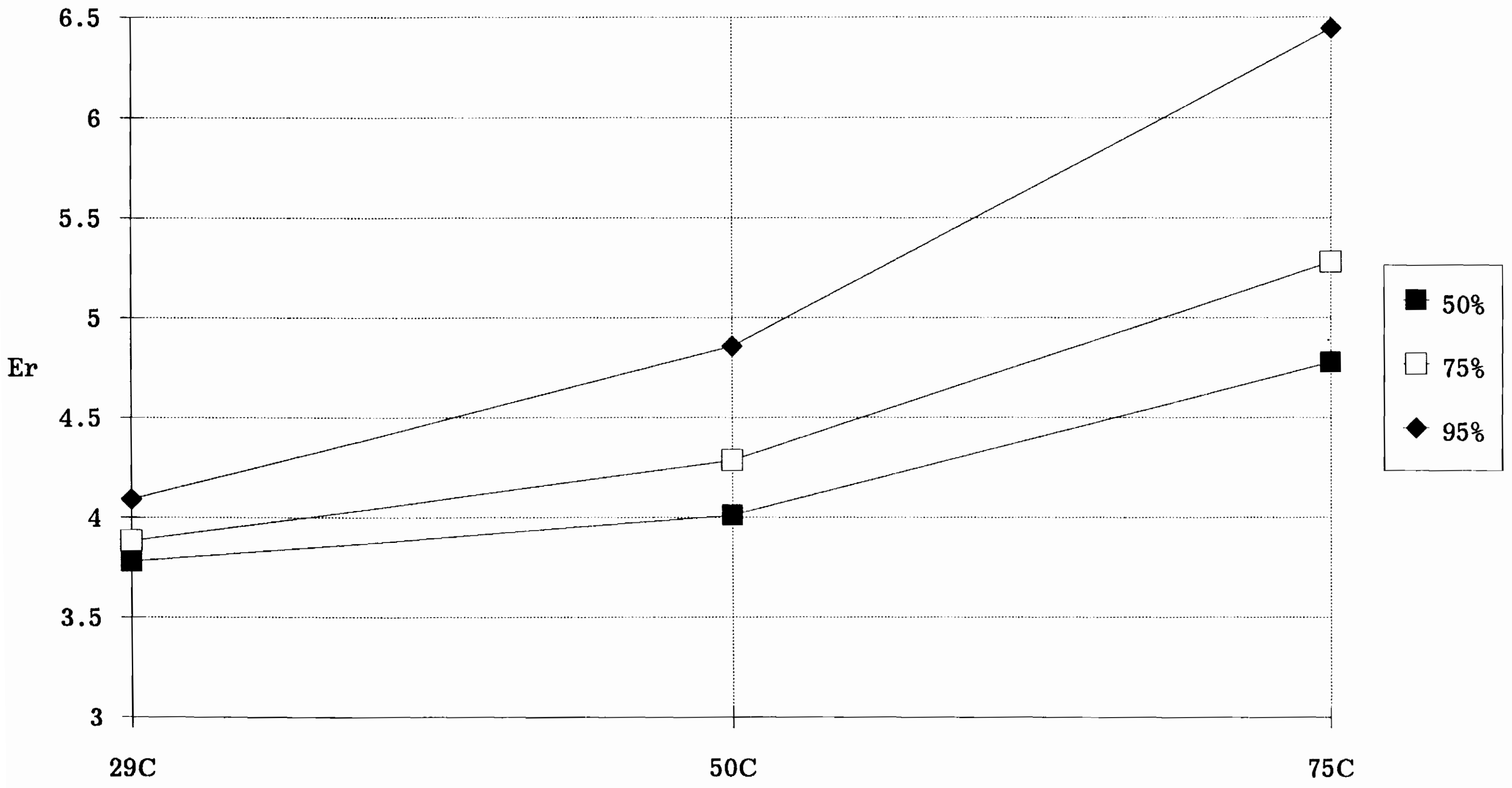


Figure 3.8 - Pyralux Dielectric Constant (d=3 mil, f=1MHz)

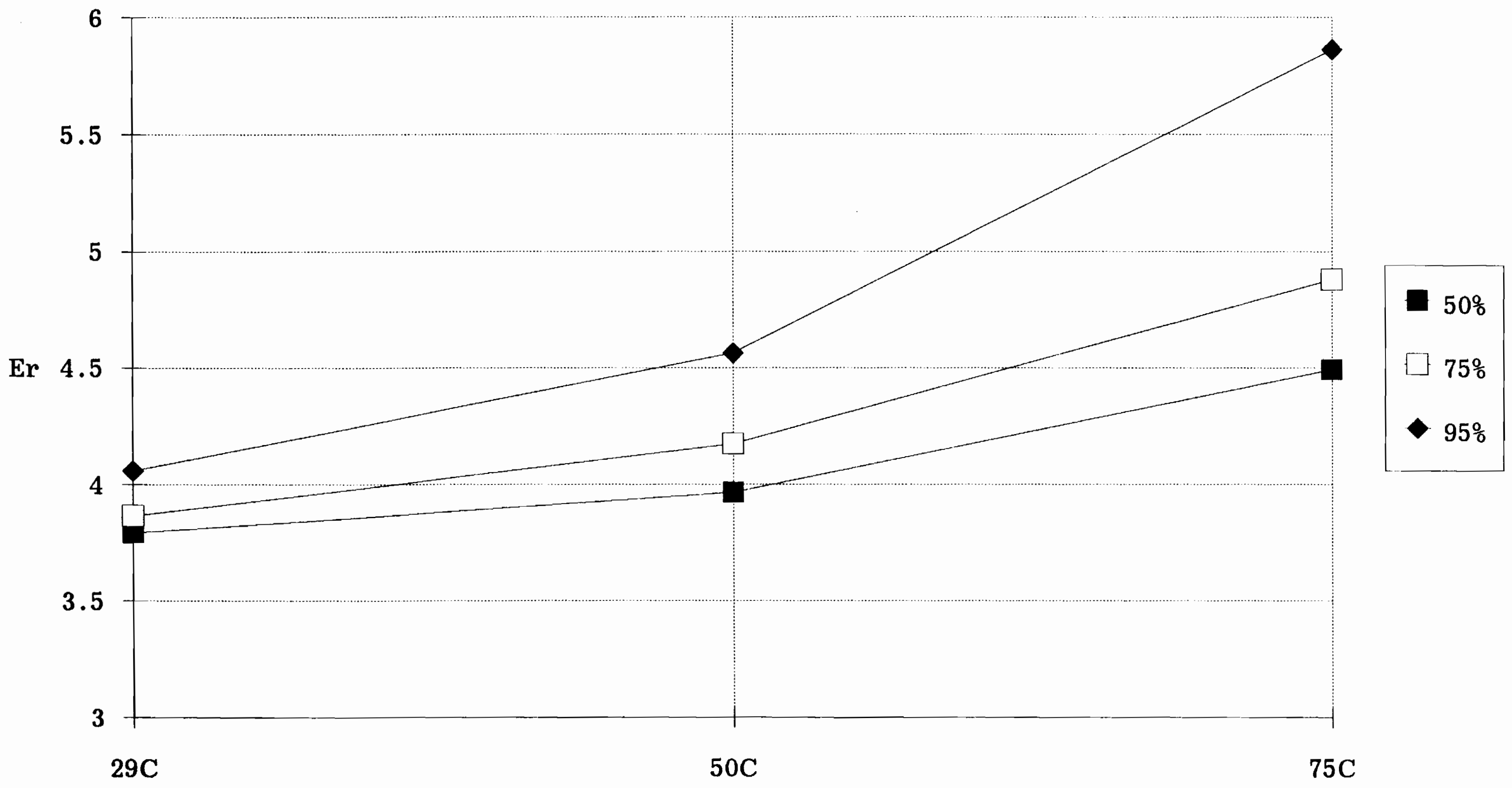


Figure 3.9 - Pyralux Dielectric Constant (d=4 mil, f=1MHz)

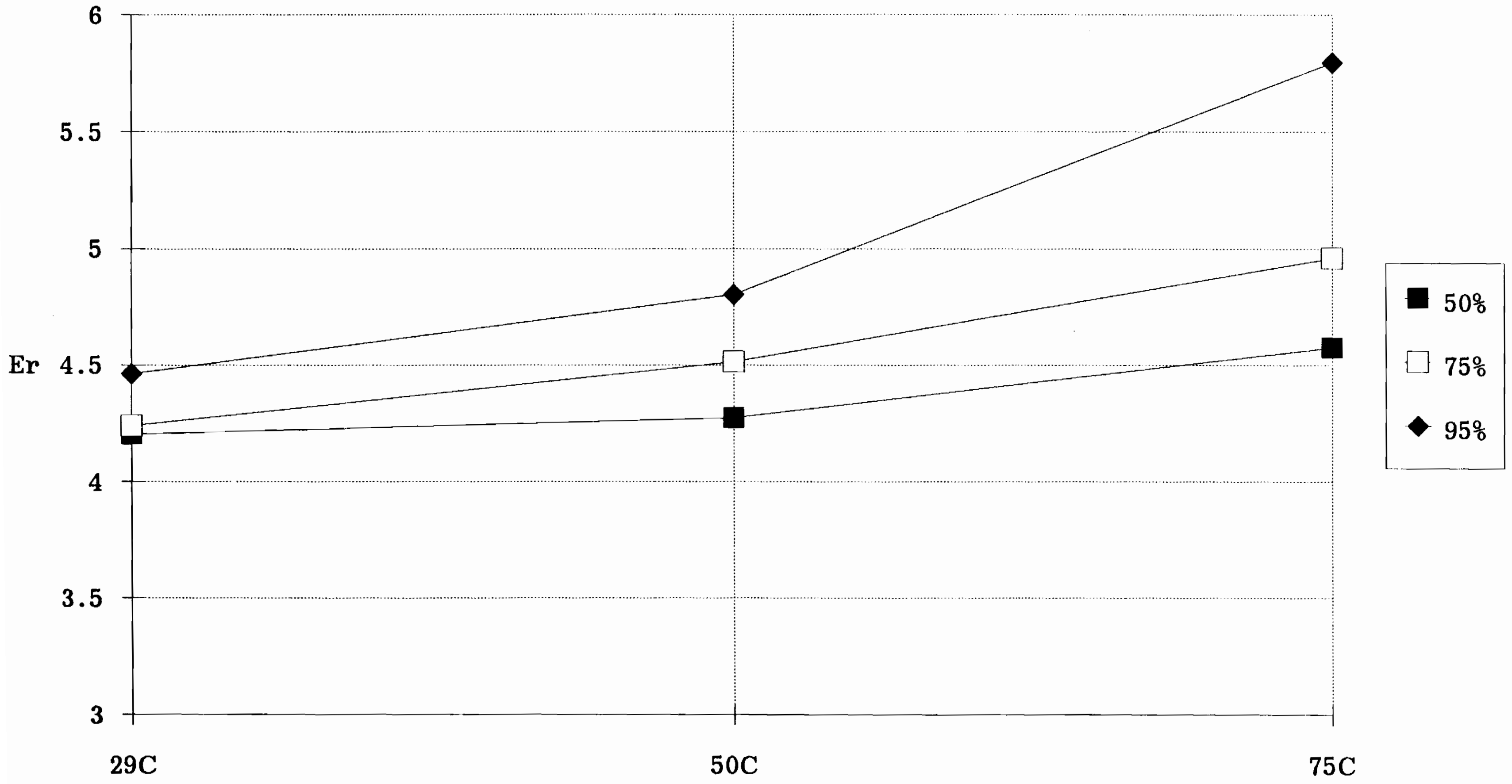


Figure 3.10 - Pyralux Dielectric Constant (d=7 mil, f=1MHz)

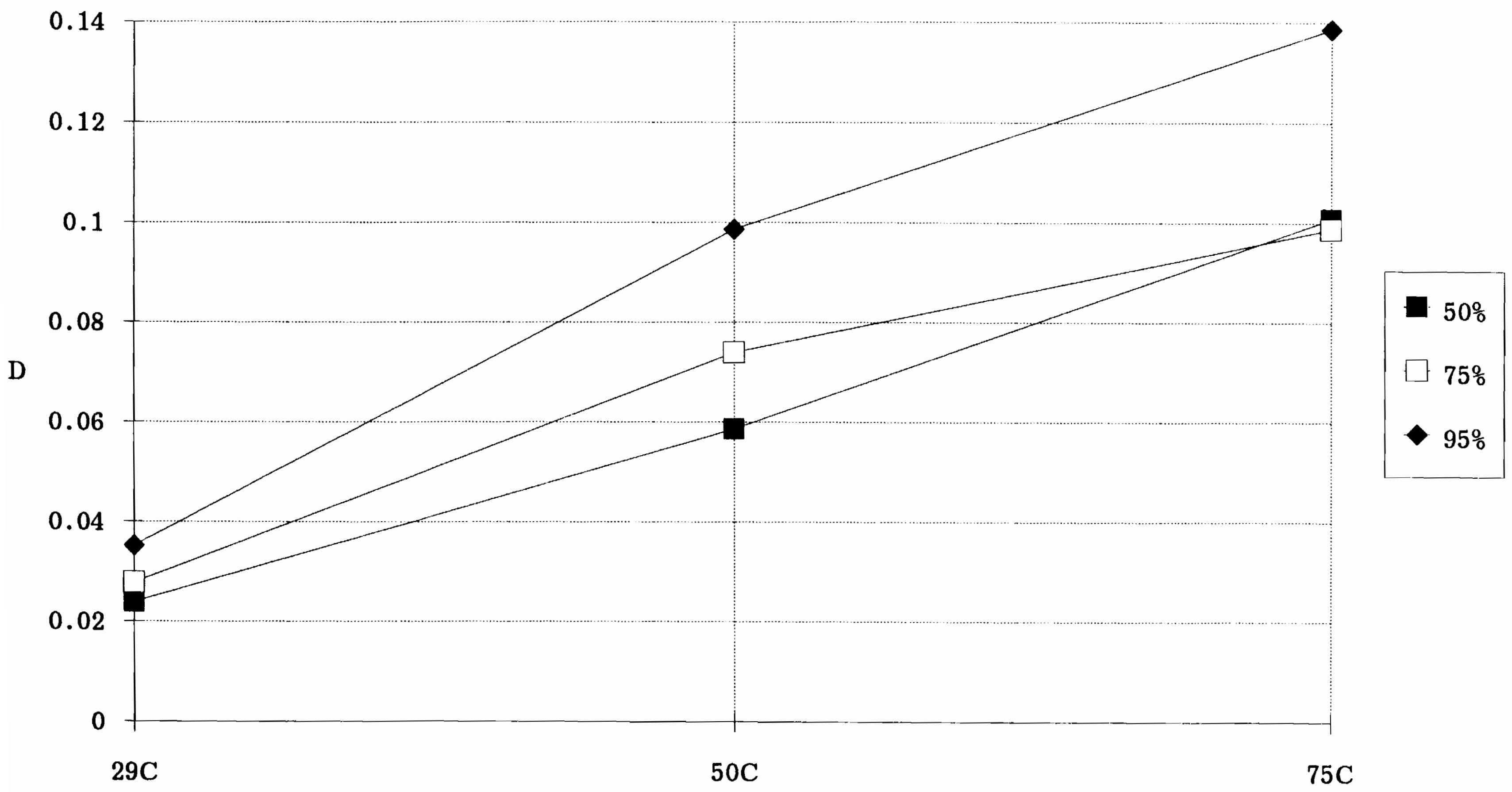


Figure 3.11 - Pyralux Dissipation Factor (d=3 mil, f=100kHz)

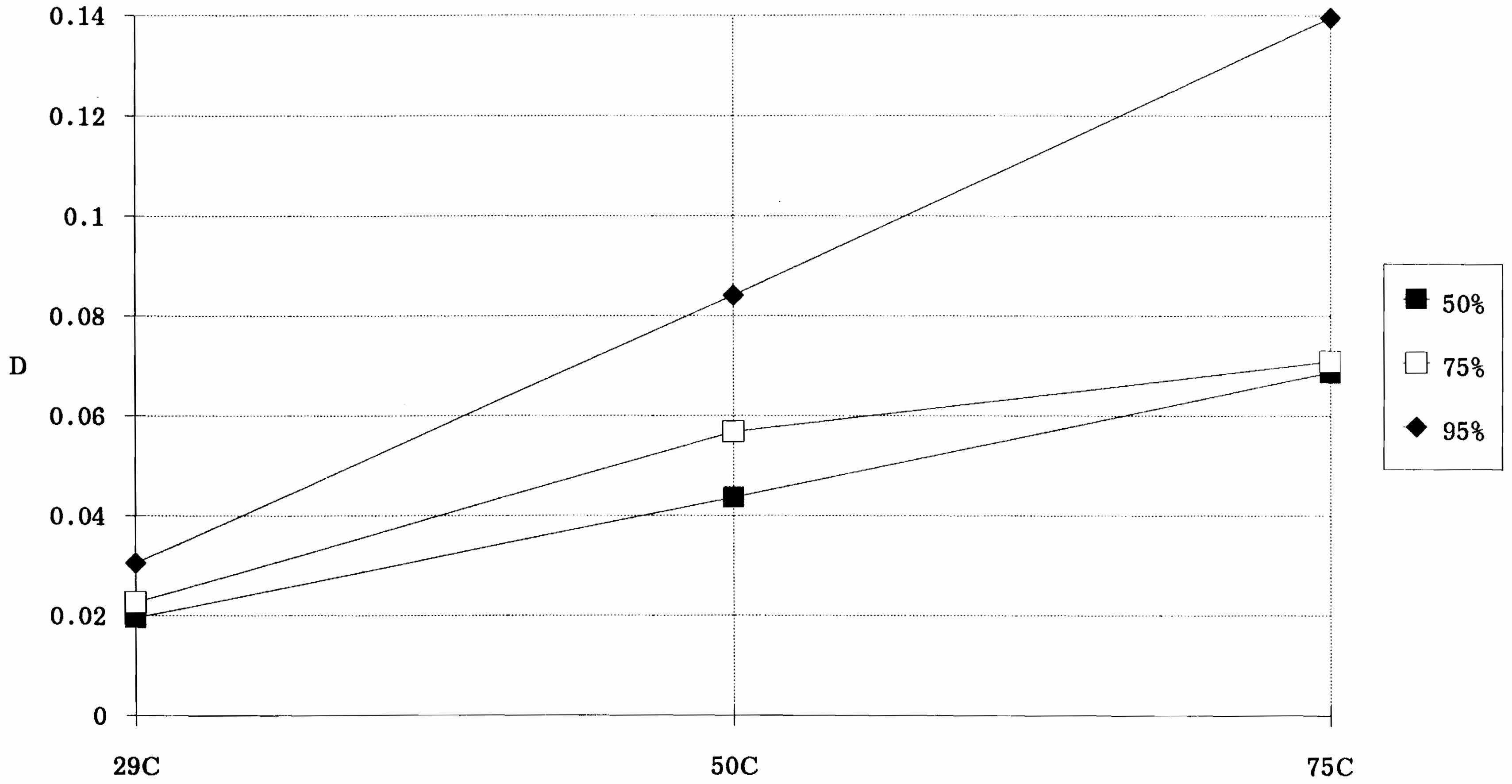


Figure 3.12 - Pyralux Dissipation Factor (d=4 mil, f=100kHz)

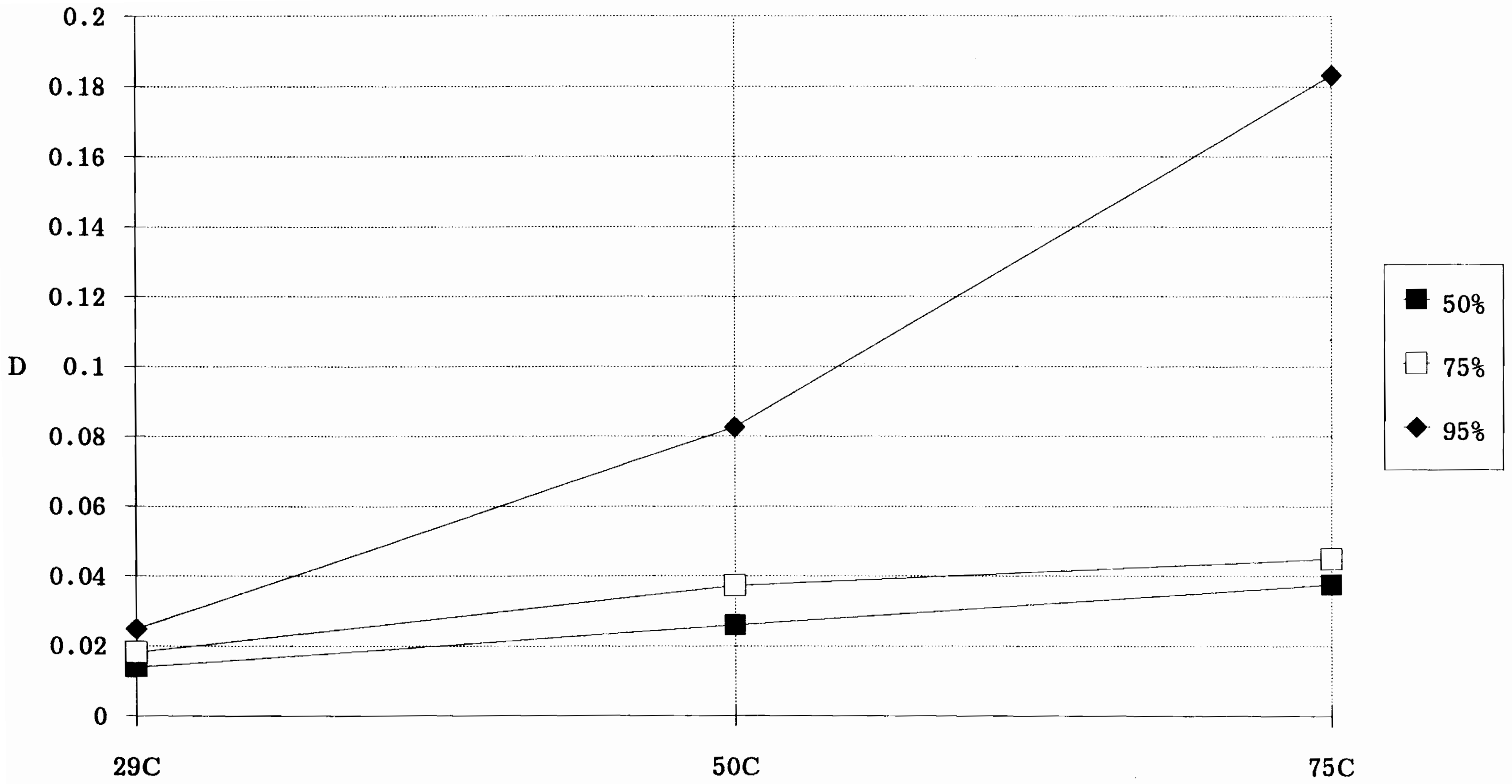


Figure 3.13 - Pyralux Dissipation Factor (d=7 mil, f=100kHz)

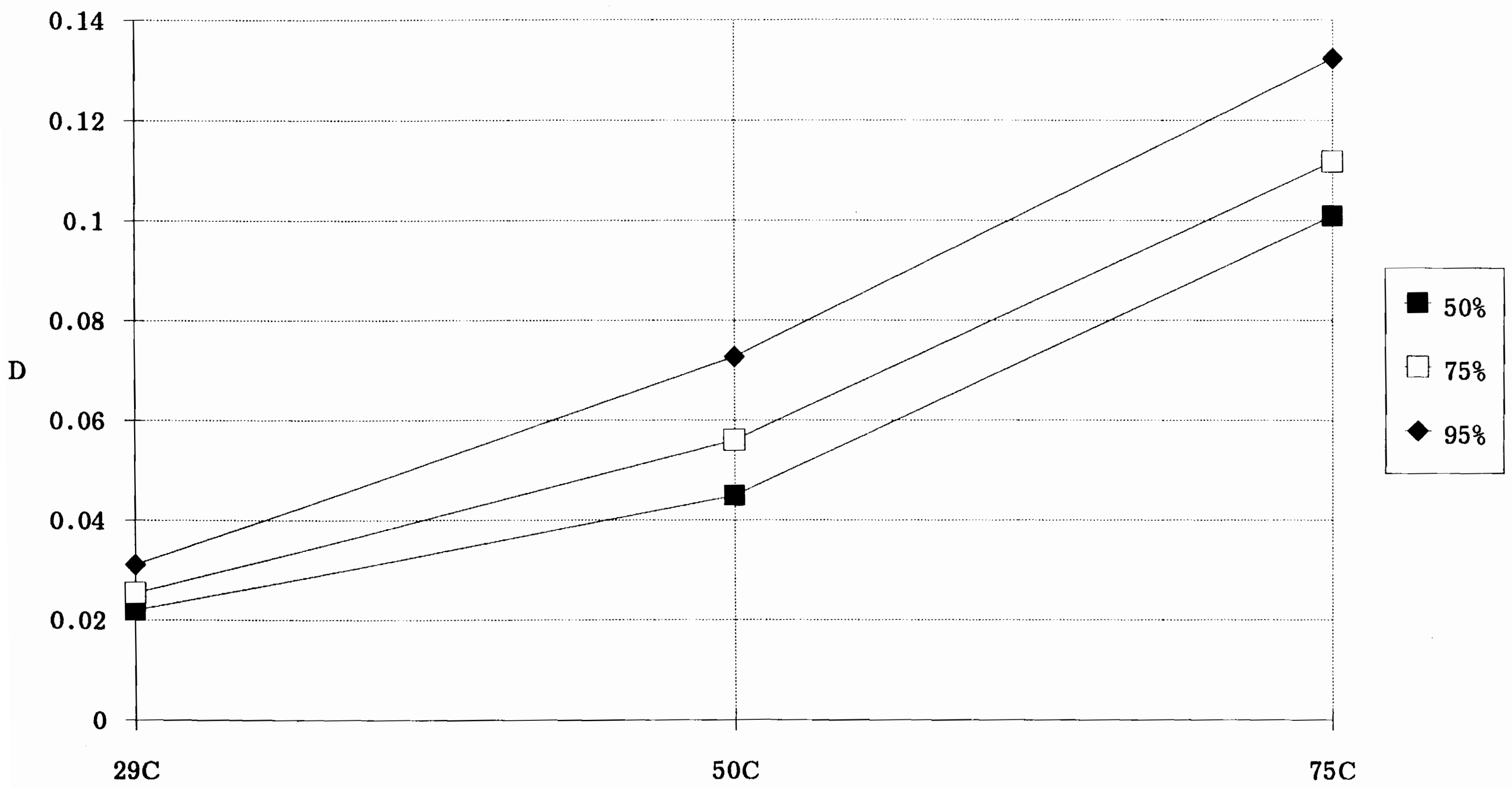


Figure 3.14 - Pyralux Dissipation Factor (d=3 mil, f=1MHz)

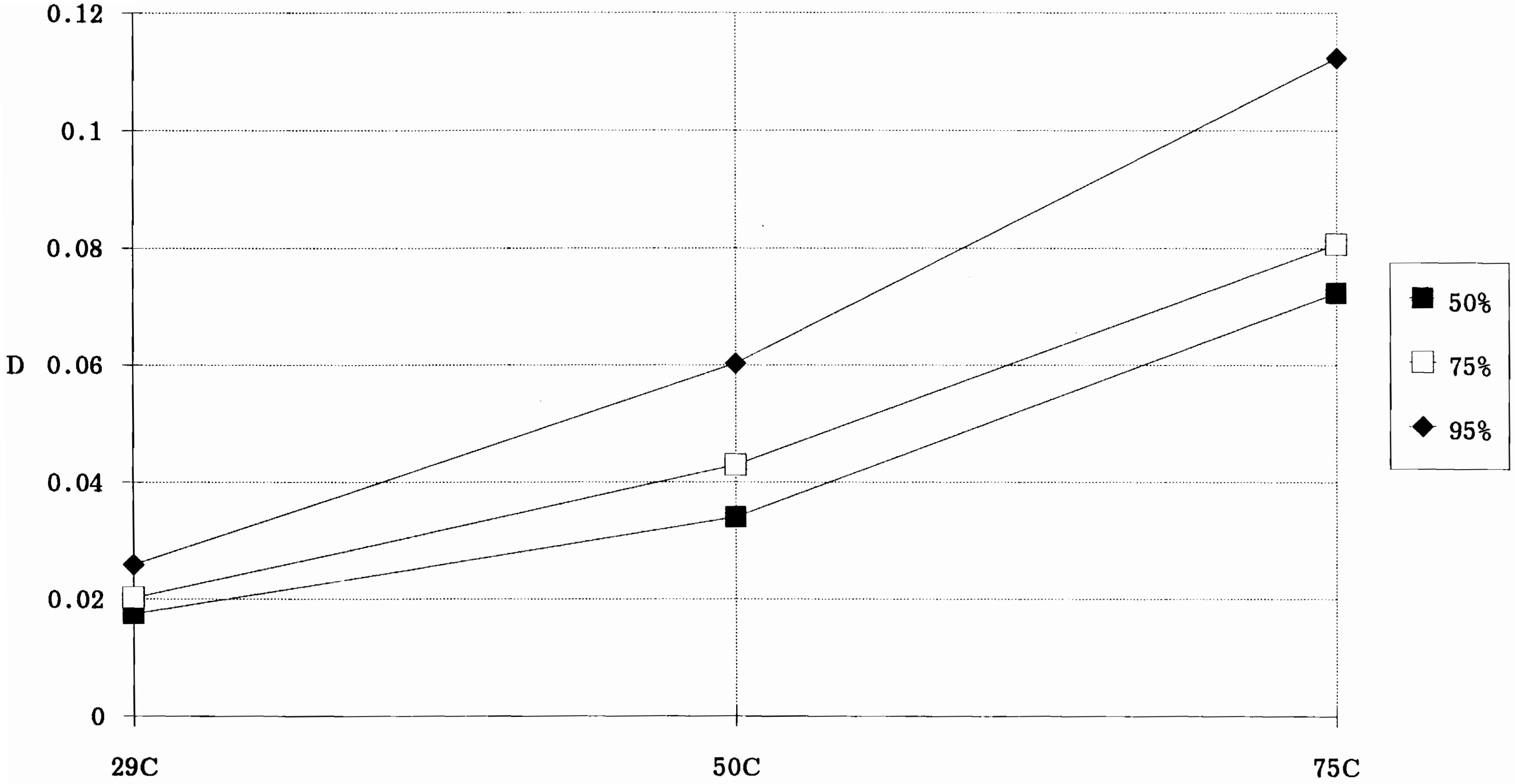


Figure 3.15 - Pyralux Dissipation Factor (d=4 mil, f=1MHz)

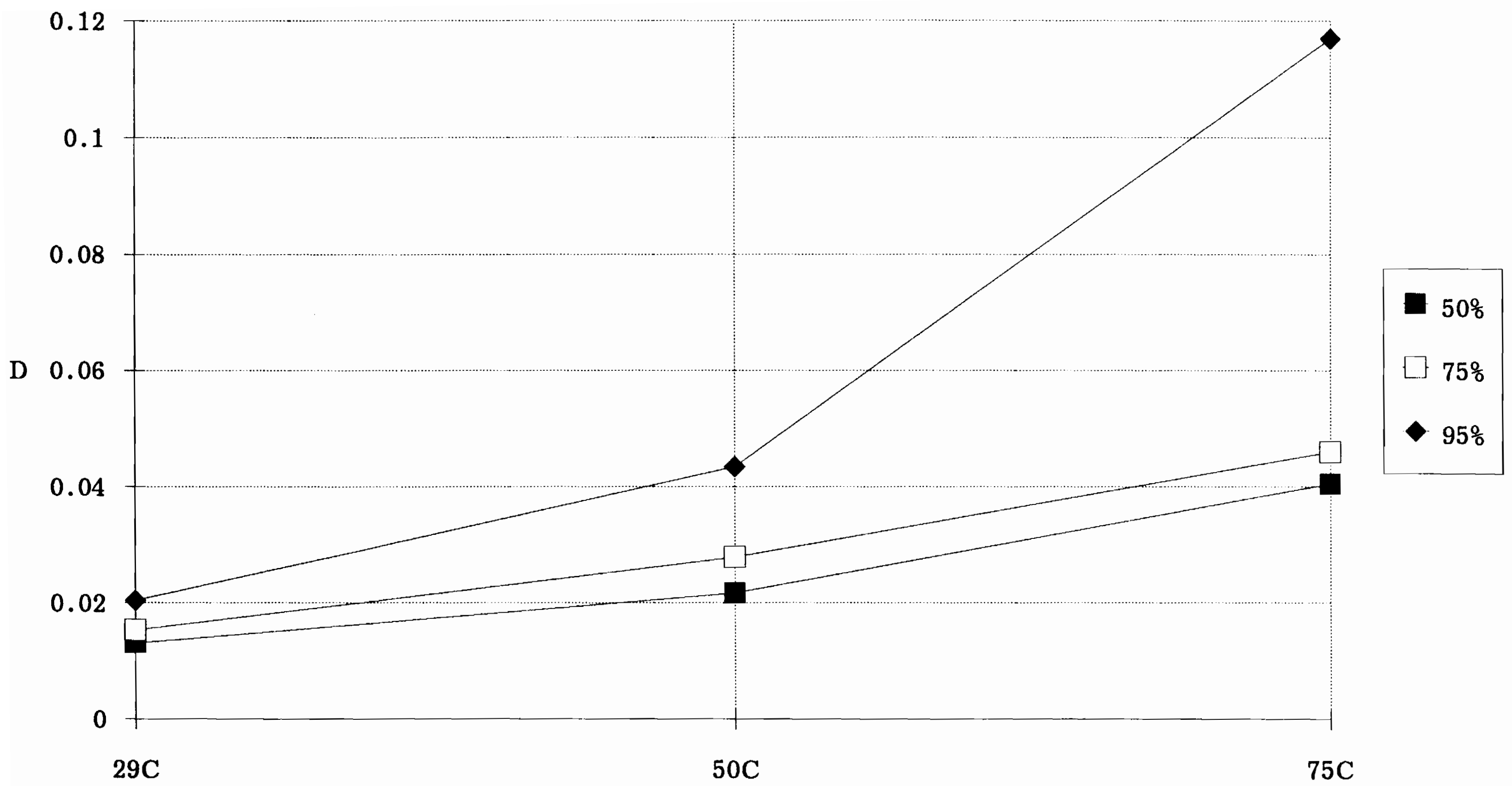


Figure 3.16 - Pyralux Dissipation Factor (d=7 mil, f=1MHz)

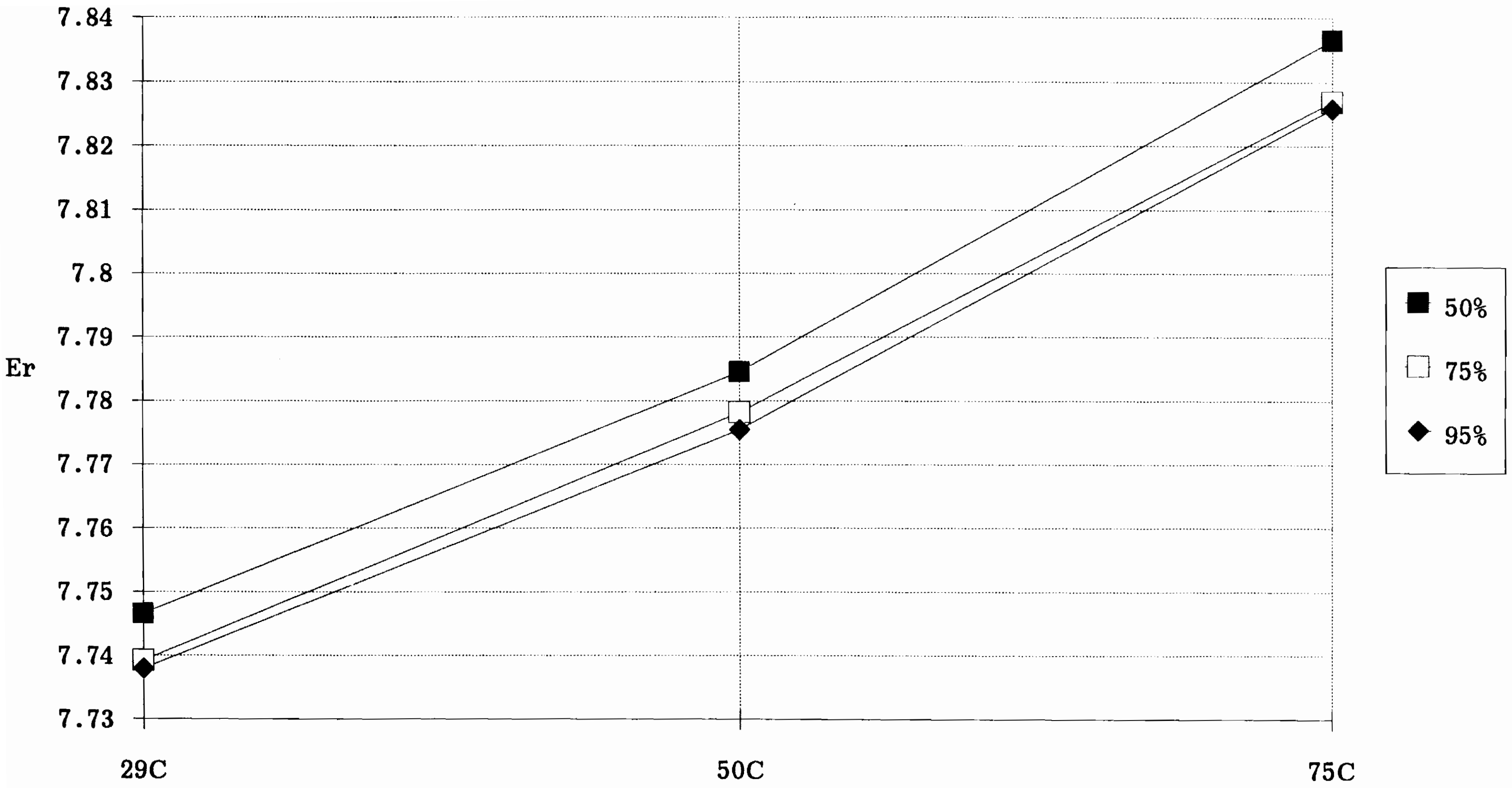


Figure 3.17 - Green Tape Dielectric Constant (d=3.7 mil, f=100kHz)

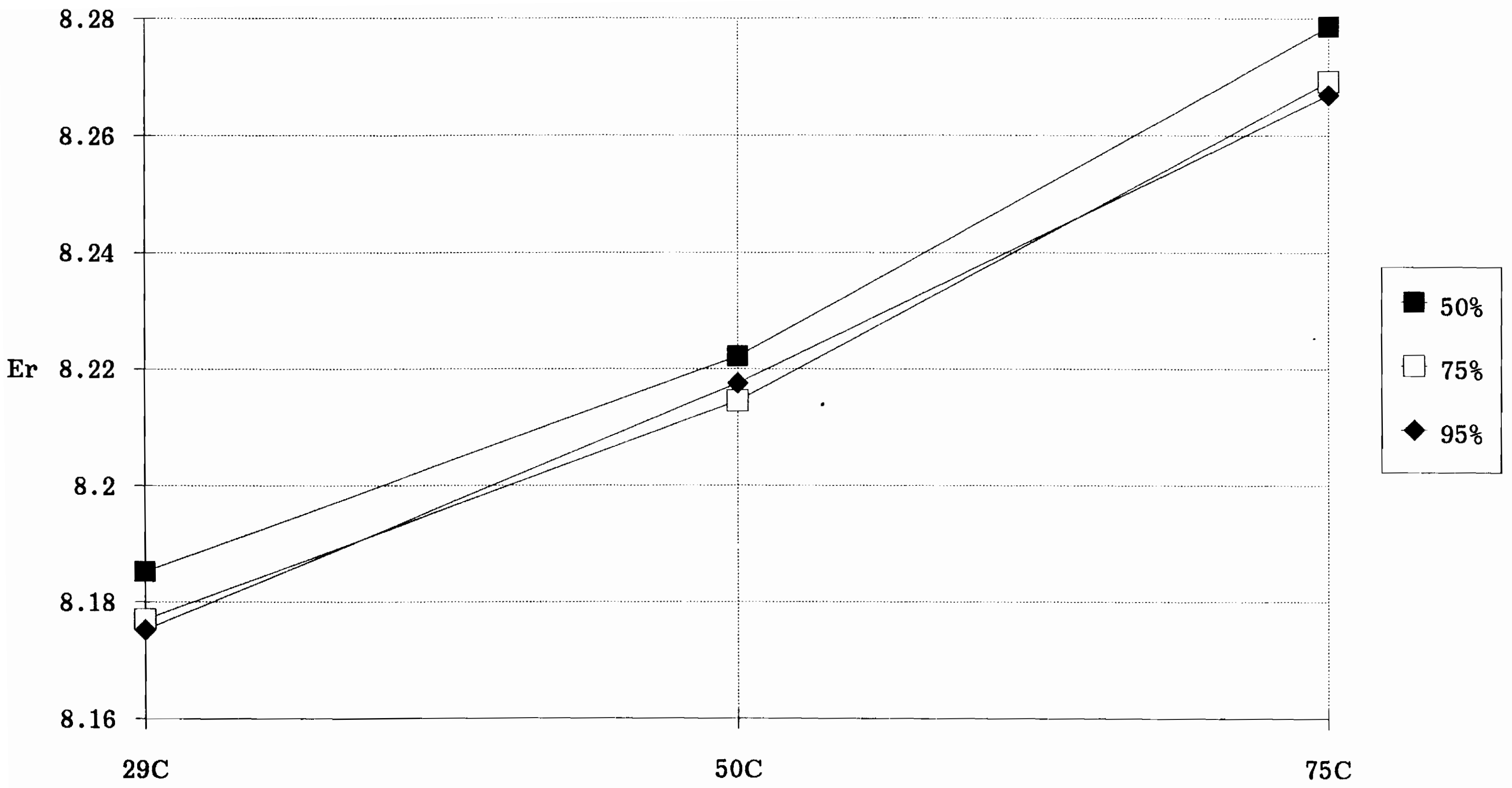


Figure 3.18 - Green Tape Dielectric Constant (d=7.4 mil, f=100kHz)

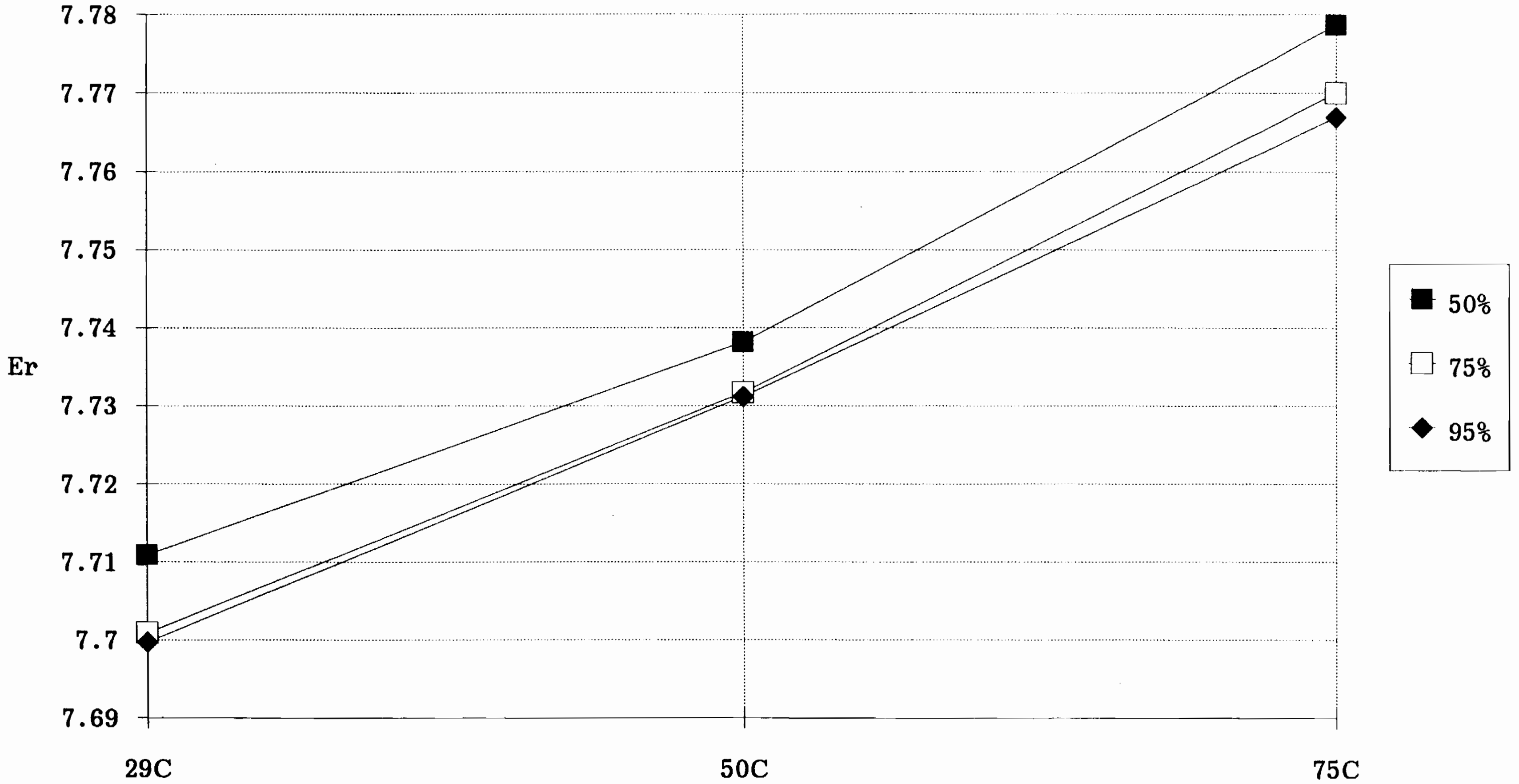


Figure 3.19 - Green Tape Dielectric Constant (d=3.7 mil, f=1MHz)

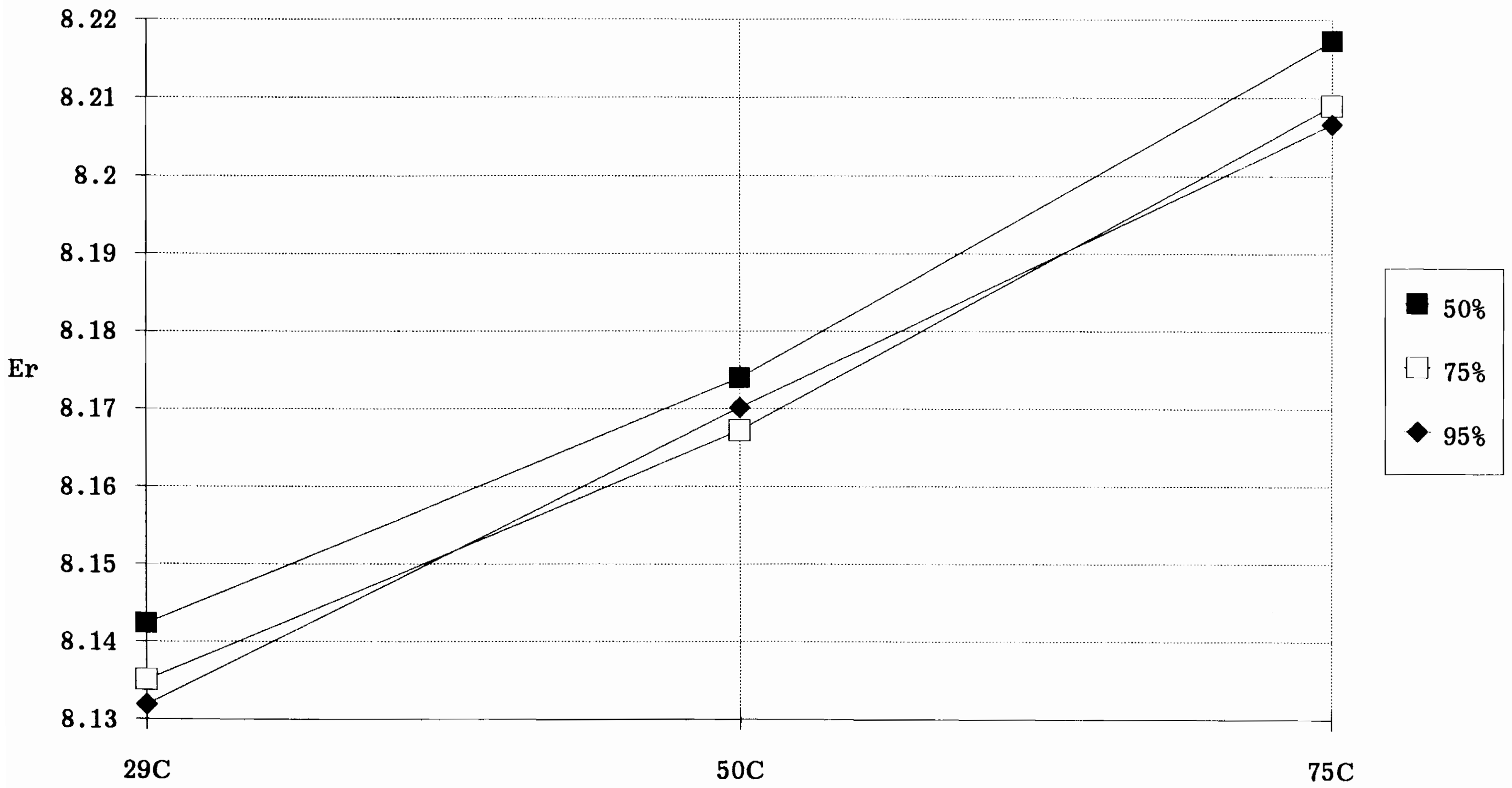


Figure 3.20 - Green Tape Dielectric Constant (d=7.4 mil, f=1MHz)

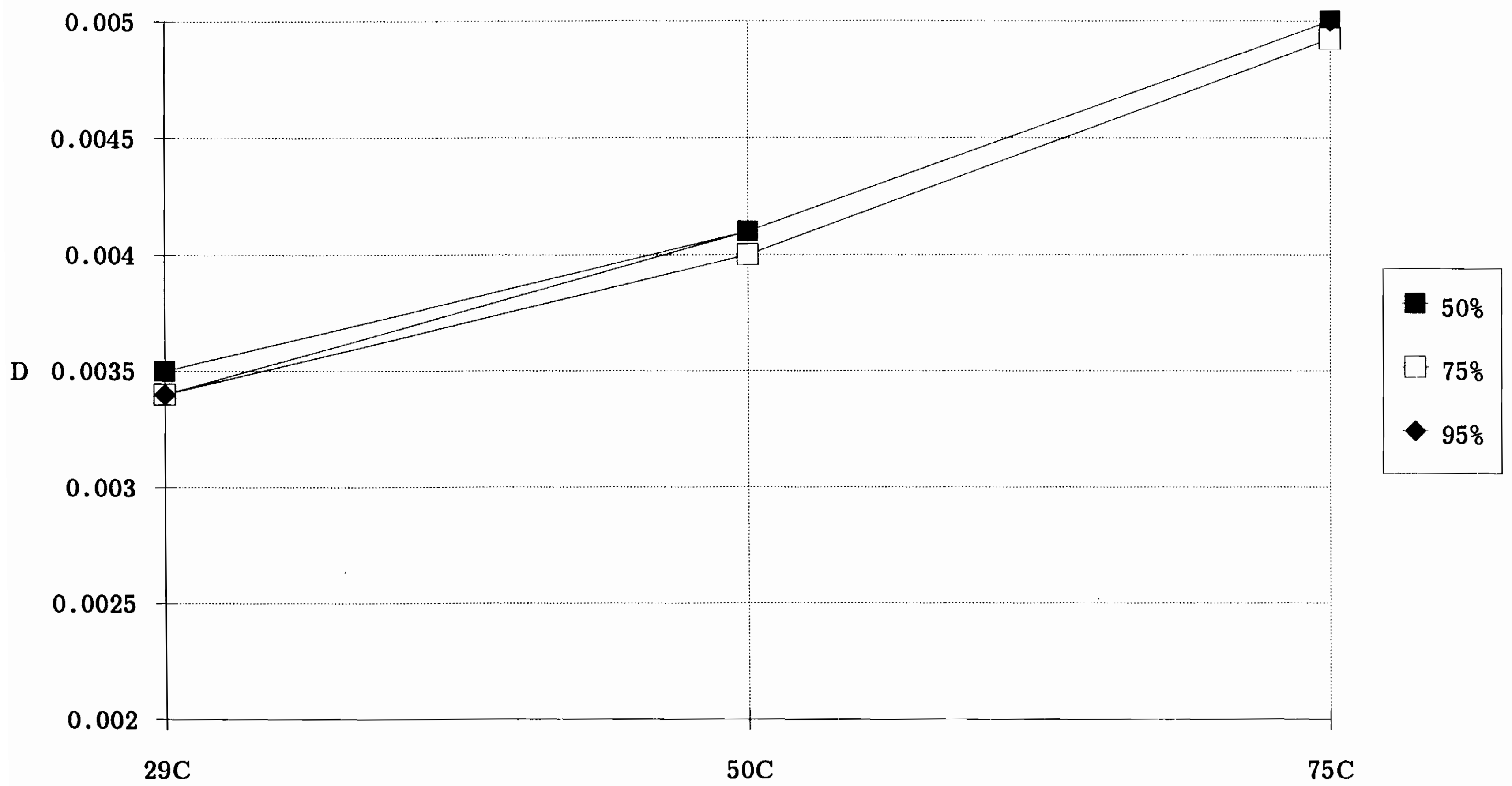


Figure 3.21 - Green Tape Dissipation Factor (d=3.7 mil, f=100kHz)

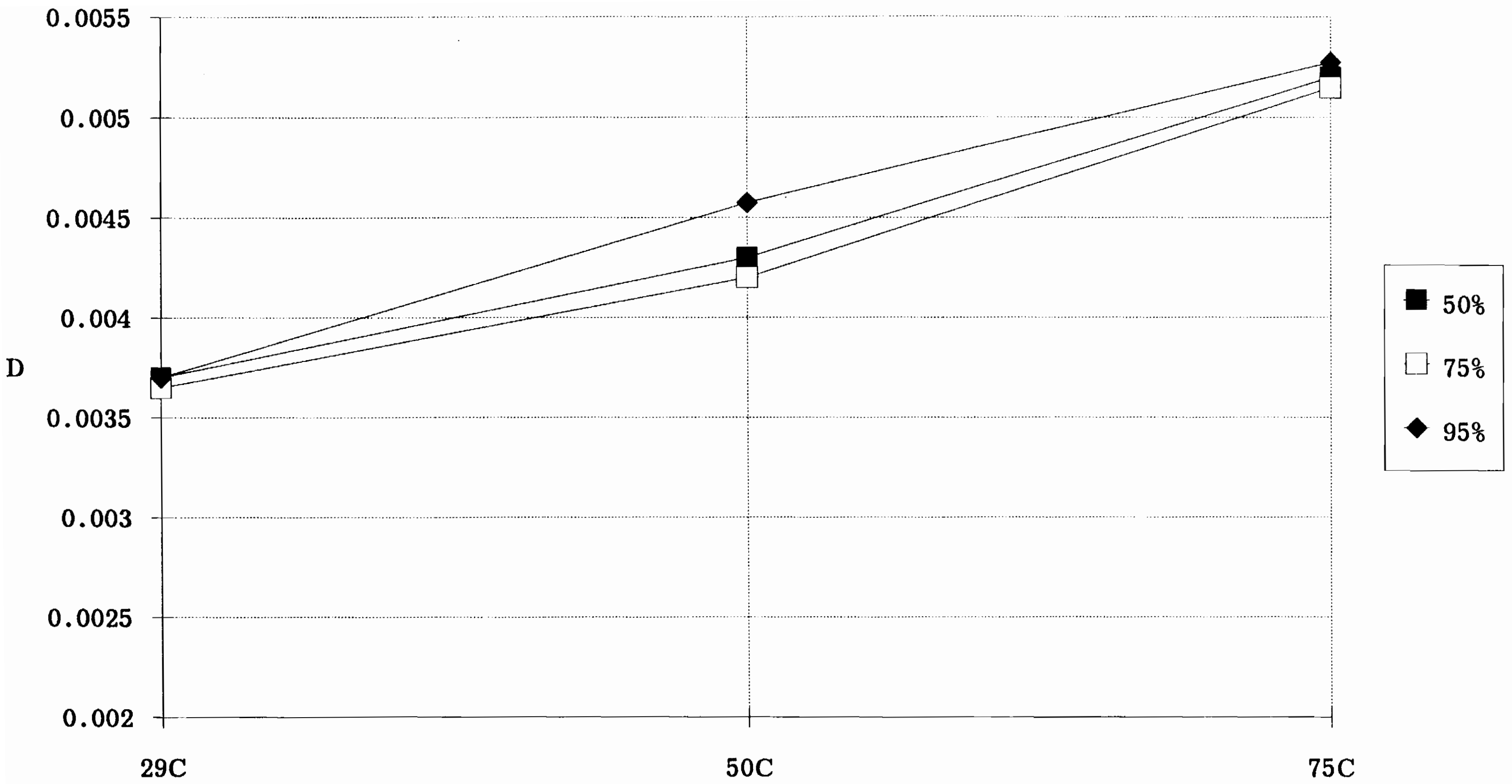


Figure 3.22 - Green Tape Dissipation Factor (d=7.4 mil, f=100kHz)

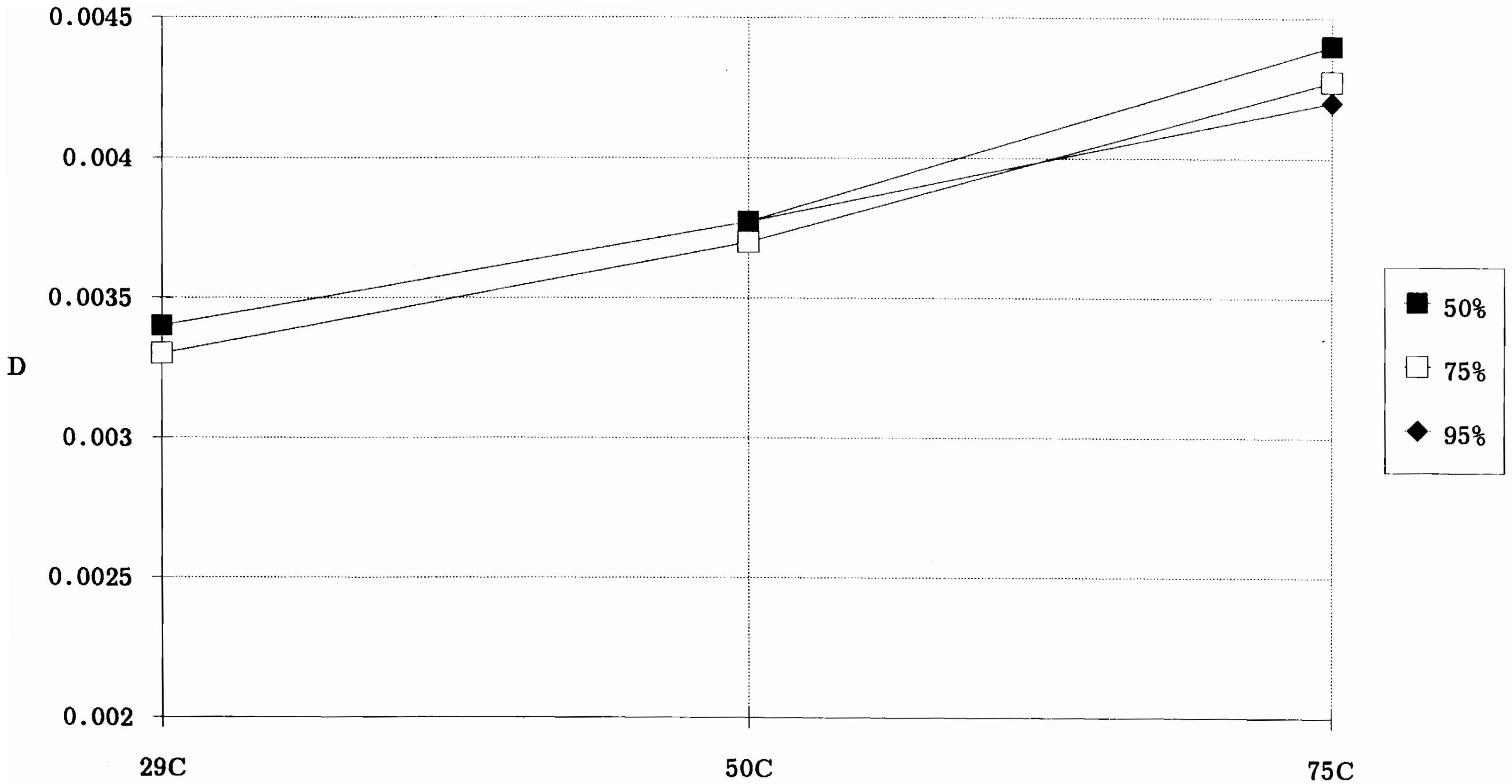


Figure 3.23 - Green Tape Dissipation Factor (d=3.7 mil, f=1MHz)

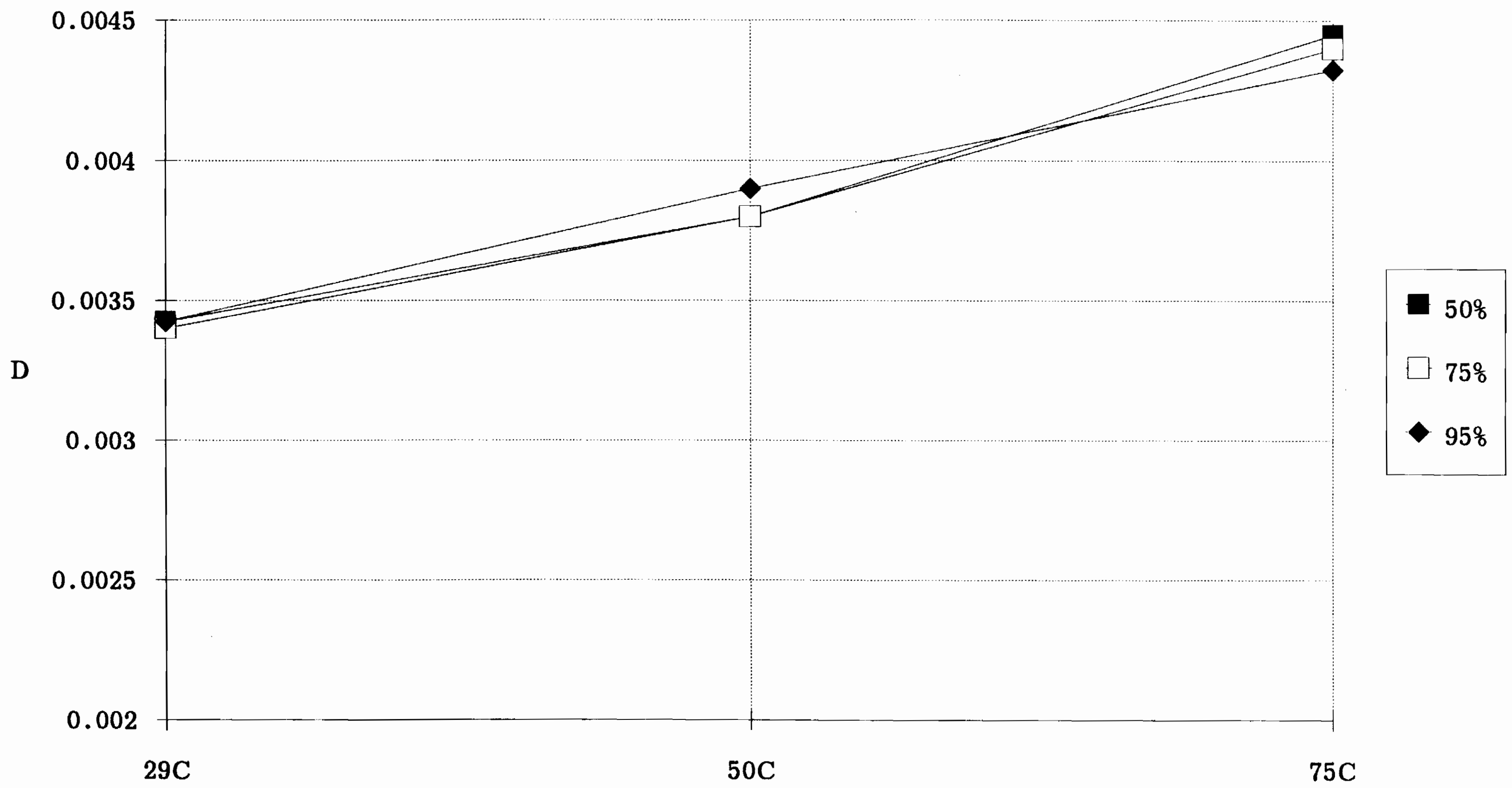


Figure 3.24 - Green Tape Dissipation Factor (d=7.4 mil, f=1MHz)

CHAPTER 4

AMBIENT TESTING AT HIGH FREQUENCIES

4.1 INTRODUCTION

Low-loss dielectric materials used in multilayer technologies such as Pyralux® and Green Tape^{TM} have rapidly gained popularity in microwave circuit applications. The high frequency characterization of these materials, therefore, is becoming increasingly important. Due to the acute sensitivity of high frequency measurements, it is very difficult to repeat the environmental tests that were performed at low frequencies (Chapter 3). The custom-made set-up used in those experiments simply does not provide the precise interface between sample and measurement device needed to perform accurate and repeatable measurements at high frequencies. Thus, the purpose of this chapter is to study the complex dielectric constant of Pyralux® and Green Tape^{TM}, at ambient conditions (approximately 29°C temperature, 50% relative humidity), over a range of high frequencies.

4.2 BACKGROUND

A significant amount of work has already been dedicated to the microwave characterization of thick film materials [6] and, more recently, of multilayer materials [3], [4], [7] at ambient conditions. These studies have utilized a number of test structures and techniques. Specifically, dielectric filled cavities, line resonators, ring resonators, and T-resonators have been evaluated using both time-domain and frequency-domain techniques. In addition, a high frequency characterization of aluminum nitride has been made over a wide temperature range [1]. This experiment involved an elaborate experimental set-up using free-wave transmission and reflection techniques. However, in order to make measurements of the sample inside the oven, the side doors of the oven had to be temporarily removed. This action further illustrates the difficulty of performing accurate, repeatable measurements without disturbing the set environmental condition. As explained in Chapter 3, ceramic materials appear to be affected less by this disturbance than polymer materials. Although both Pyralux® and Green Tape^{TM}

have been investigated in the above studies, very little data is available for the characterization of both materials using the same structure. Thus, the focus of this chapter is the evaluation of both Pyralux® and Green Tape^{TM} using a single structure, followed by a comparison of these results with those obtained from other structures and techniques.

4.3 PROCEDURES

The structure selected for this study is the T-resonator microstrip. The T-resonator is designed to resonate at integer multiples of its quarter-wavelength frequency. An estimate of the effective dielectric constant of the material under test is then used to determine the appropriate stub length for a desired primary resonant frequency (Equation 4.1) [7]:

$$L_{stub} = \frac{c}{4f\sqrt{E'_{re}}}, \qquad (4.1)$$

where L_{stub} is the stub length, c is the speed of light in vacuum, f is the primary resonant frequency, and E'_{re} is the estimated effective dielectric constant. The measured resonant frequencies of the structure will therefore provide information on the effective dielectric constant. Corrections for end and junction effects are then performed to determine the actual material effective dielectric constant value (E_{re}) [7]. Finally, corrections for dispersion effects are performed to yield a value for the material dielectric constant (E_r) [7]. These values, in addition to the quality factor (Q) of the material, are then used to calculate the material loss tangent (Equation 4.2) [7]:

$$\text{Loss Tangent} = \frac{E_{re}(E_r-1)}{Q_d E_r (E_{re}-1)}, \qquad (4.2)$$

where Q_d is the quality factor due to dielectric losses, and E_{re} and E_r are defined as above. As mentioned in Chapter 2, this measurement procedure invokes several approximations so that only one test structure is necessary. A comparison of these results with those obtained from the line and ring resonators (which do not use these approximations, and thus require multiple test structures [9]) will therefore demonstrate the accuracy of these approximations.

During the summer of 1991, the researcher had the opportunity to work at the DuPont Electronics facility in Research Triangle Park, North Carolina (RTP). At the DuPont facility, he worked on a number of projects, including a worldwide Correlation Testing Program which involved the high frequency characterization of Green Tape^{TM} using the T-resonator microstrip structure. Upon returning to Virginia Tech, he designed, constructed, and tested Pyralux® T-resonator microstrips to provide complete results for this current study.

The processing of these materials is the same as for those used in the low frequency tests of Chapter 3. Although the Green Tape^{TM} samples were made at the DuPont Experimental Station in Wilmington, Delaware, extensive physical measurements were performed at the RTP facility before conducting any experimentation. The stub length of the T-resonator microstrip was designed to be 2", to provide a primary resonance of approximately 660 MHz. A 32.5 mil-wide gold (DuPont 5715) microstrip was screen printed on a 29.6 mil (8 layers of 851-AT) dielectric thickness to create a characteristic impedance of approximately 50Ω. The external ground was formed by screen printing the entire 3" x 3" bottom surface area with gold. The Pyralux® samples were designed [7], [8] with a stub length of 2.8" to provide a primary resonance at approximately 660 MHz. A 25 mil wide microstrip was etched on top of a 10 mil thick dielectric layer (6 mil Kapton®, 4 mil adhesive) to create a characteristic impedance of approximately 50Ω. The external ground plane consisted of an entire 3" x 3" area of 1 oz. copper. Figure 4.1 illustrates the T-resonator microstrip structure, as well as typical line and ring resonator structures.

4.4 MEASUREMENTS

Measurement of 15 Green Tape^{TM} samples was performed using an HP 8753A Network Analyzer at the DuPont RTP facility, while an HP 8510B Network Analyzer was used to measure two Pyralux® samples at Virginia Tech. The transmission response of the T-resonator provides information regarding the resonant frequency and quality factor (Q) at each resonant "null". The resonant frequency data is used to determine the material dielectric constant, while the Q data is used to determine the material loss tangent [7]. A computer program, written at the DuPont Experimental Station, was used to calculate the above data for the Green Tape^{TM}

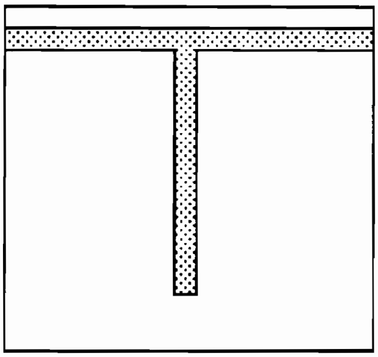
samples. In order to maintain consistency in the measurements, the same computer program was used in the Pyralux® measurements. At both experimental locations, repeatable connections between the samples and the Network Analyzer were obtained through the use of Cascade Microtech Launchers.

4.5 RESULTS

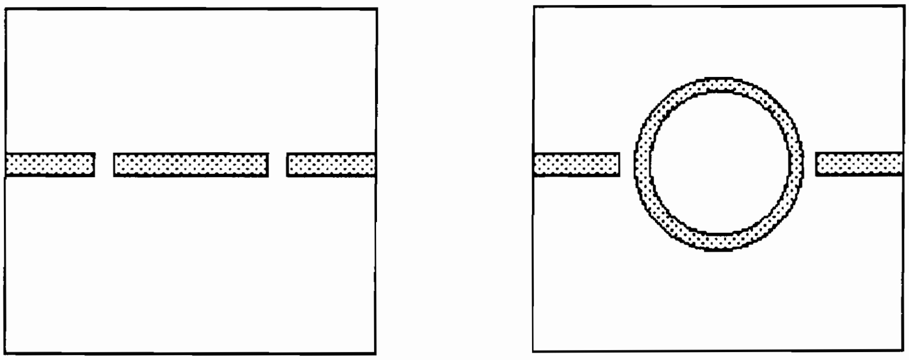
Due to the frequency limitations of the HP 8753A, data for the Green Tape^{TM} T-resonators have been obtained for only the first two resonances (up to approximately 2 GHz). Averaging over the 15 samples, the first two resonant frequencies are 0.656 GHz and 1.964 GHz. The respective dielectric constants and loss tangents at these frequencies are 7.211, 0.014 and 7.212, 0.008. Despite the short frequency range, these dielectric constant results are within 2% of those stated in literature [7]. The loss tangent results appear to be slightly higher than those in the above reference, yet still within the expected range. On the other hand, data for the first six resonant frequencies have been obtained for the Pyralux® samples. Figure 4.2 illustrates the excellent agreement between three different resonator structures (line and ring striplines, and T microstriplines) in the measurement of the dielectric constant over a large frequency range. In addition, the loss tangent data is found to be on the order of 0.01 ± 0.001 for all three structures over the same frequency range [9].

4.6 CONCLUSIONS

This chapter has addressed the high frequency characterization of Pyralux® and Green Tape^{TM} materials under ambient conditions. The primary objective of this work is to measure both materials using the same test structure and technique. This requirement removes any concern about possible anomalies in the data, resulting from the use of different structure types and measurement techniques. Thus, an analysis and comparison of the materials, alone, may be performed. Finally, this data also compares very well with results obtained from earlier studies involving different resonator types and measurement procedures.



T-Resonator [7]



Line Resonator [9]

Ring Resonator [9]

Figure 4.1 - Resonator Structures

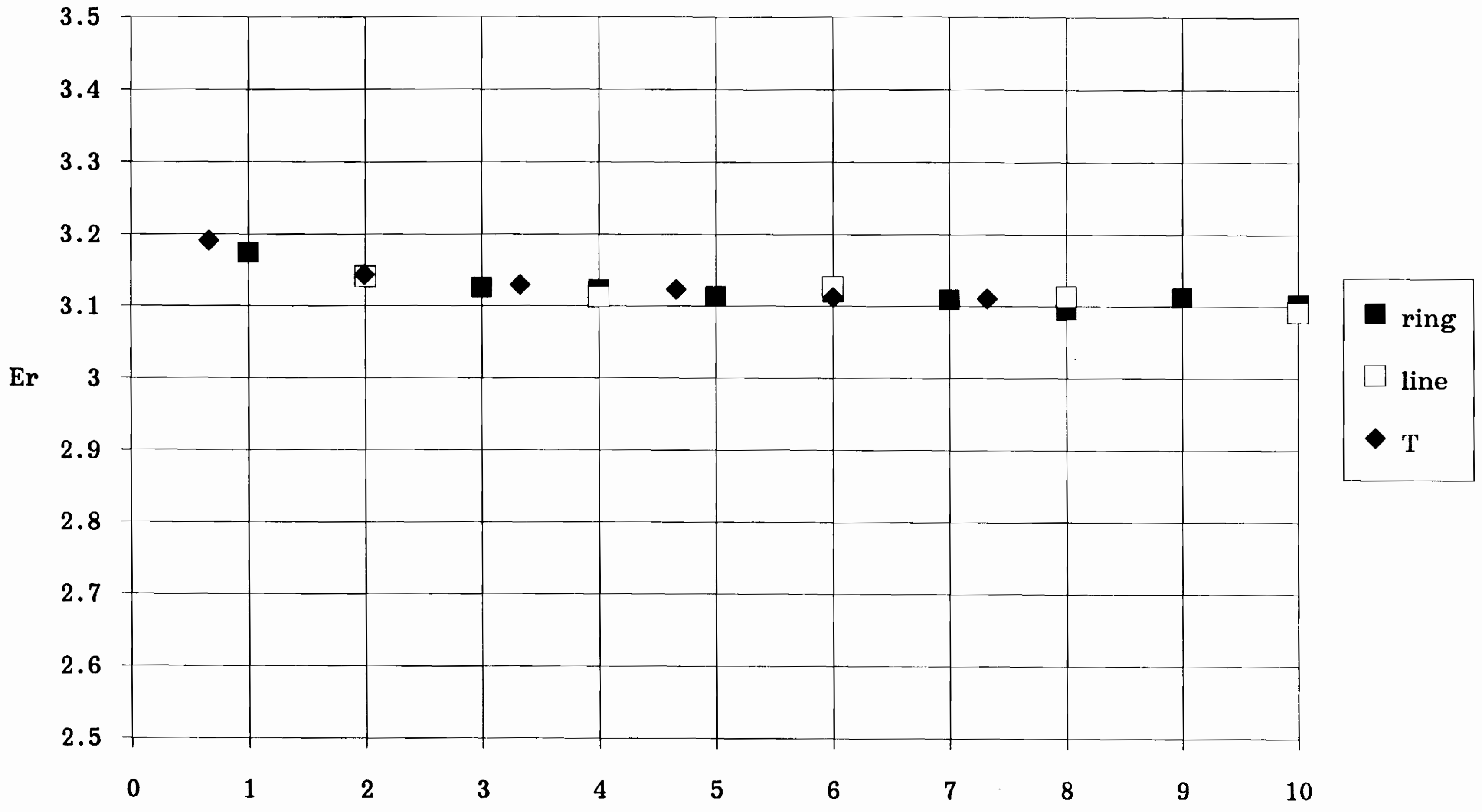


Figure 4.2 - Pyralux Dielectric Constant vs High Frequency (GHz)
(Ambient Conditions: Temperature 29C, Relative Humidity 50%)

CHAPTER 5

LIFETIME TESTING AT LOW FREQUENCIES

5.1 INTRODUCTION

The previous chapters have studied the characterization of Pyralux® and Green Tape^{TM} materials at low and high frequencies, with and without imposed environmental conditions. Since these measurements are performed during a relatively short time frame (minutes or hours), they may be characterized as short-term data. One characteristic of this type of data is the elimination of time as a contributing factor. Some materials, however, experience property transformations simply due to gradual aging. These aging effects, therefore, may only be detected by performing long-term measurements, involving a time frame of weeks or months. Thus, the purpose of this chapter is to determine the effects of aging on the complex dielectric constant of Pyralux® and Green Tape^{TM} materials at low frequencies.

5.2 BACKGROUND

In theory, the study of aging effects involves long-term measurement procedures. The general practice, however, is to simulate the natural aging process with forced aging, in order to shorten the measurement time frame. One example of a forced aging technique is thermal cycling. Thermal cycling is a short-term measurement procedure in which the material is gradually heated to a peak temperature, held at that peak temperature for a short duration of time, and then gradually cooled back to ambient temperature. This chapter will investigate and compare the effects of both long-term (natural) aging, as well as short-term (forced) aging on the complex dielectric constant of Pyralux® and Green Tape^{TM} materials at low frequencies.

5.3 PROCEDURES

Since these experiments will be performed at low frequencies (100 kHz and 1 MHz), additional capacitor laminates, almost identical in structure to those used for the environmental testing (Chapter 3), are designed and constructed with the Pyralux® and Green Tape^{TM} 851-AT systems. The Pyralux® laminate has a dielectric layer consisting of a 1 mil Kapton® layer, sandwiched between two adhesive layers. The Green Tape^{TM} laminate has a dielectric layer consisting of a single layer of 851-AT. The only difference between the dimensions of these laminates and those of the environmental test laminates involves the size of the Green Tape^{TM} electrodes and vias. Since these samples will be probed manually, the shrinkage of the Green Tape^{TM} does not need to be considered, unlike the case for the environmental testing. Thus, the upper electrodes of the Green Tape^{TM} laminate are 200 mil x 200 mil pre-fired, and 177 mil x 177 mil post-fired. The bottom electrodes are 300 mil x 700 mil pre-fired, and 268 mil x 618 mil post-fired. Finally, the diameter of the vias is 11.5 mil pre-fired and 10.5 mil post-fired. Once again, each laminate contains an array of four capacitors to provide repeatability.

5.4 MEASUREMENTS

After construction, the capacitor laminates are enclosed in a desiccant chamber, under vacuum conditions. Over a span of several months, in two week intervals, the laminates are removed from the chamber, tested, and immediately returned to the chamber. The HP 4192A impedance analyzer is once again used to perform measurements of the capacitance value and dissipation factor at 100 kHz and 1 MHz in an ambient environment (approximately 29°C temperature, 50% relative humidity). All the results, including a least-squares linear fit to the data, are summarized in Figures 5.1-5.8.

As a comparison to these results, a Pyralux® laminate and a Green Tape^{TM} laminate, both identical in structure to those used in the long-term aging experiment, are thermally cycled in order to force a rapid aging of the materials. The laminates are placed in a Fisher Isotemp® programmable ashing furnace (model 497), and exposed to a cycle consisting of a 175°C/30 minute peak, with heating and cooling rates of 10°C/minute. After cycling, the HP 4192A impedance analyzer is used to measure the capacitance values and dissipation factors, at the same low frequencies, and in the same ambient environment. These results are noted in the upper right hand corner of Figures 5.1-5.8, for ease of comparison.

5.5 RESULTS

The data obtained from the long-term aging and thermal cycling experiments demonstrate some interesting trends. The dielectric constant and dissipation factor of the Pyralux® material are increasing over time. Conversely, both the dielectric constant and the dissipation factor of the Green Tape^{TM} material are decreasing over time, but at a much slower rate than that for Pyralux®. Note, however, that the results do not appear to be very sensitive to the operating frequency. Support for these conclusions comes from the results of the thermal cycling experiment. After cycling, the dielectric constant was observed to be significantly higher in value for the Pyralux® sample, and significantly lower in value for the Green Tape^{TM} sample. In addition, the dissipation value of the Pyralux® sample was notably higher in value after cycling, whereas the dissipation value of the Green Tape^{TM} sample appeared only slightly lower in value after cycling.

5.6 CONCLUSIONS

This chapter addressed the effects of aging on the complex dielectric constant of Pyralux® and Green Tape^{TM} materials. Both long-term and short-term aging experiments were conducted, and the results of one supported the results of the other. The gradual increase in the Pyralux® values, and the slower decrease in the Green Tape^{TM} values over time is reminiscent of the trends related to an increasing humidity environment, discovered in Chapter 3. One may therefore deduce that the samples are gradually absorbing and retaining moisture over time, even though they are removed from the vacuum environment for only a short time. Since the desiccant chamber maintains a humidity of less than 5%, it is not surprising that the dielectric constant values in the aging experiment are relatively lower for Pyralux®, and relatively higher for Green Tape^{TM}, than the values observed during the environmental testing (humidity $\geq 50\%$). Similar relationships may also be observed in the dissipation factors of the two materials.

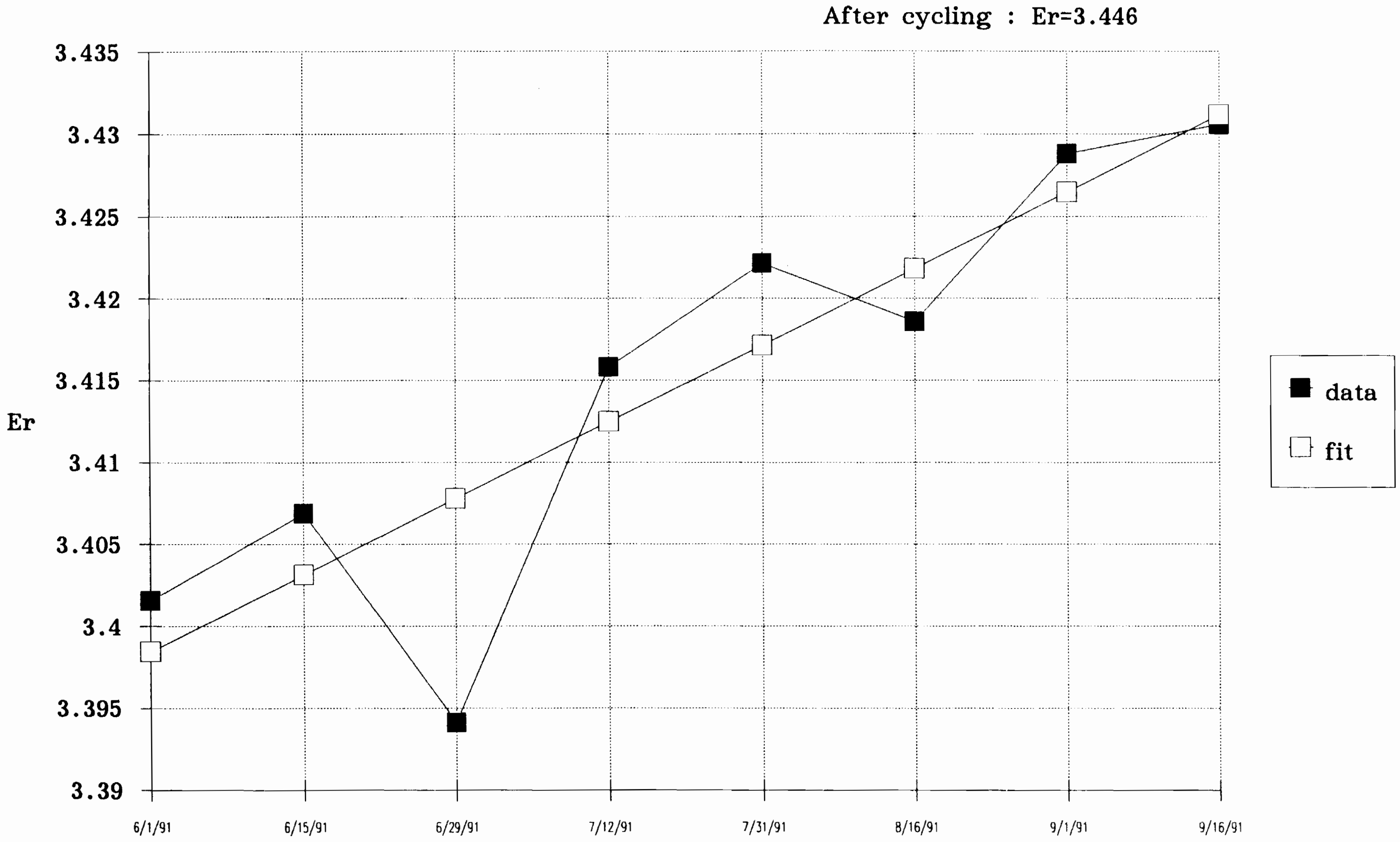


Figure 5.1 - Pyralux Dielectric Constant vs Time (f=100kHz)

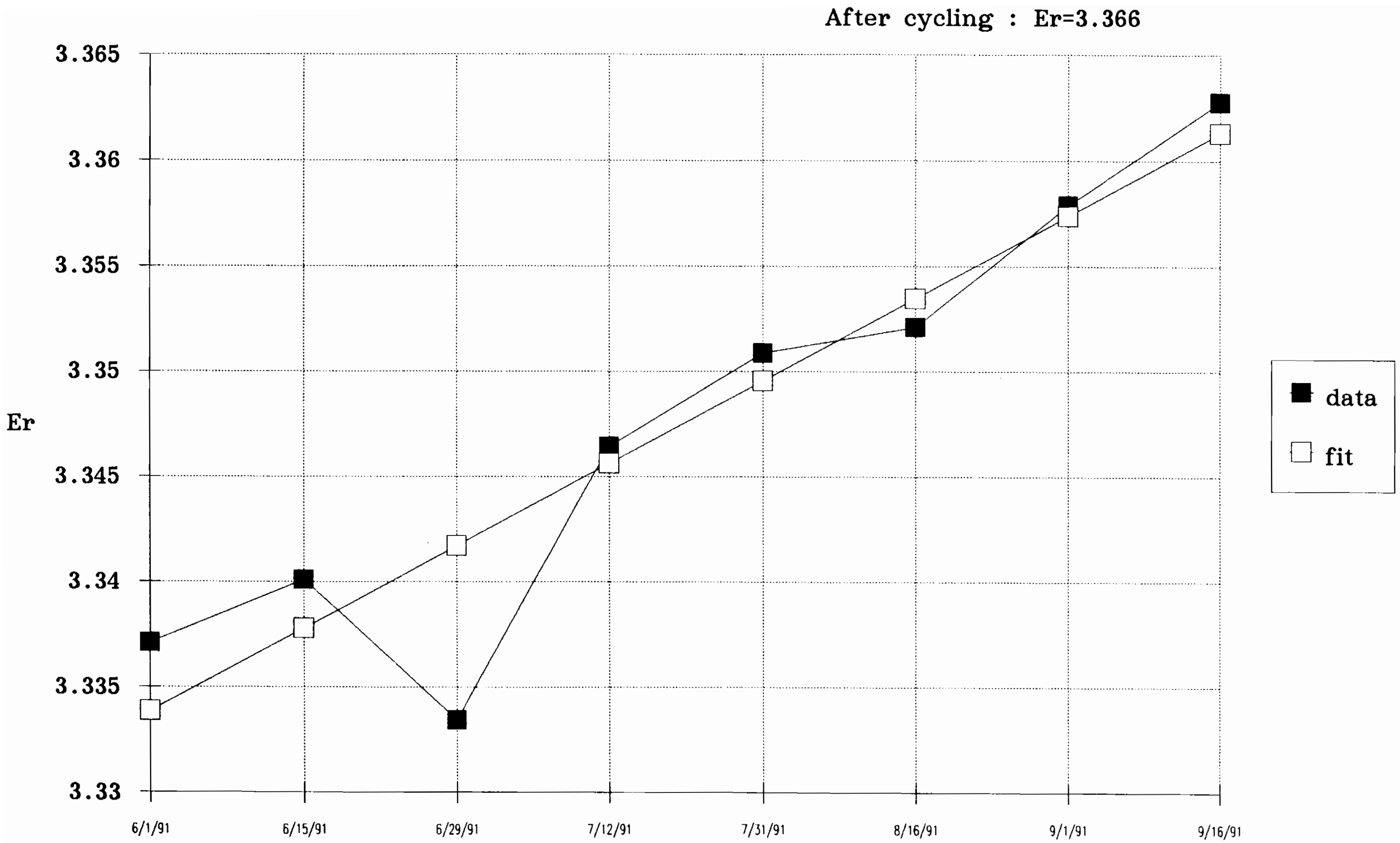
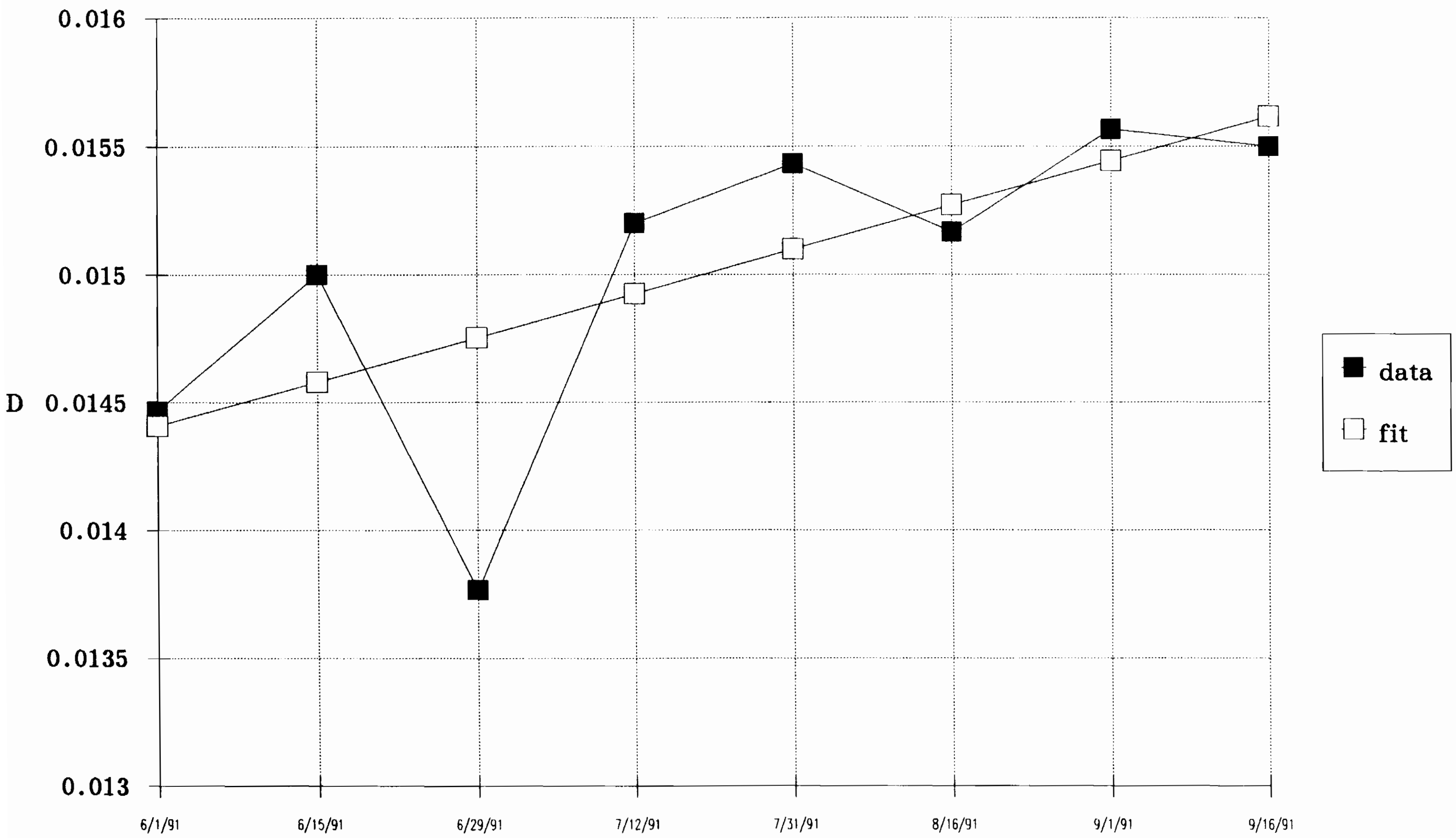


Figure 5.2 - Pyralux Dielectric Constant vs Time (f=1MHz)



After cycling : D=0.0175

Figure 5.3 - Pyralux Dissipation Factor vs Time (f=100kHz)

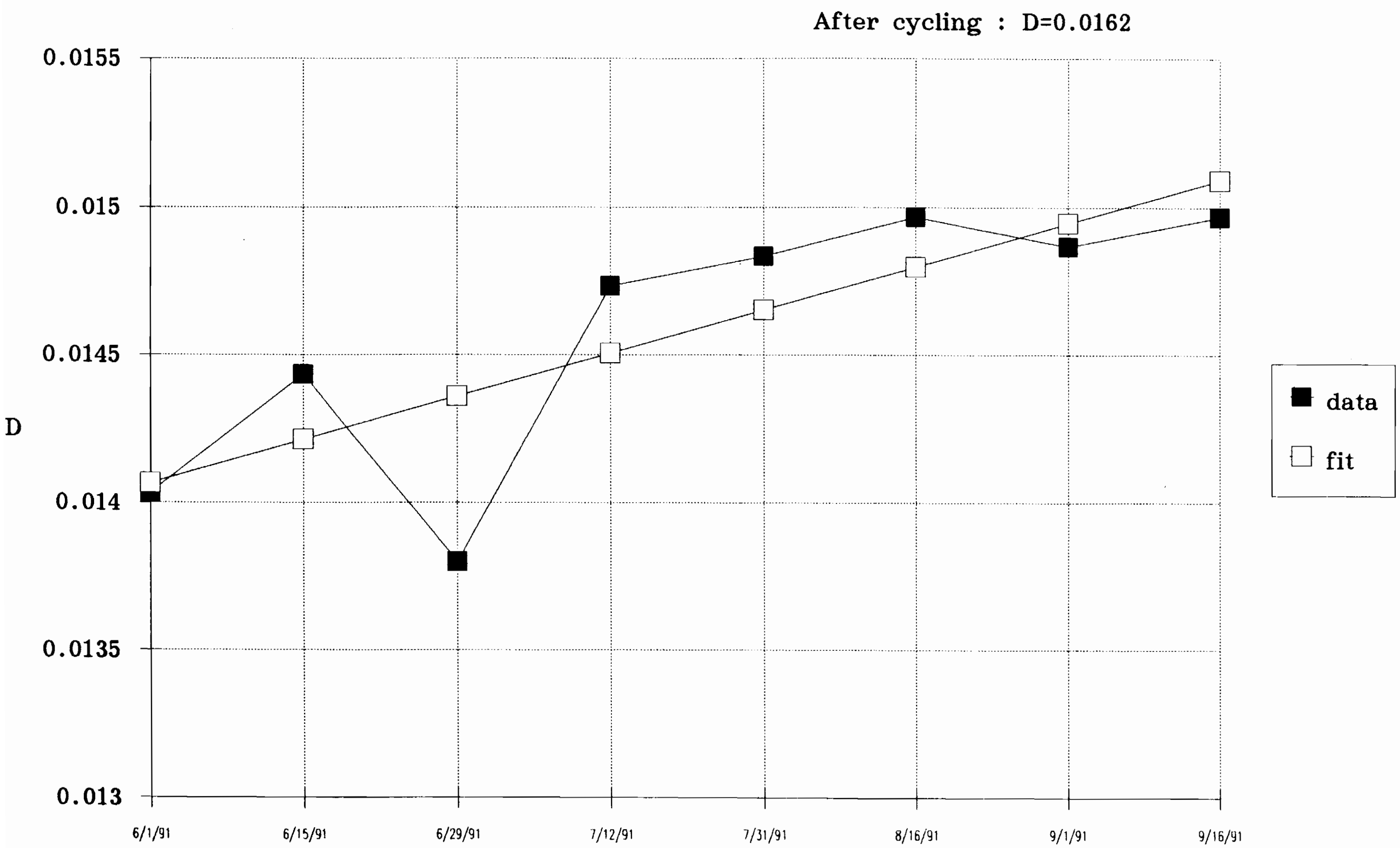

After cycling : D=0.0162

Figure 5.4 - Pyralux Dissipation Factor vs Time (f=1MHz)

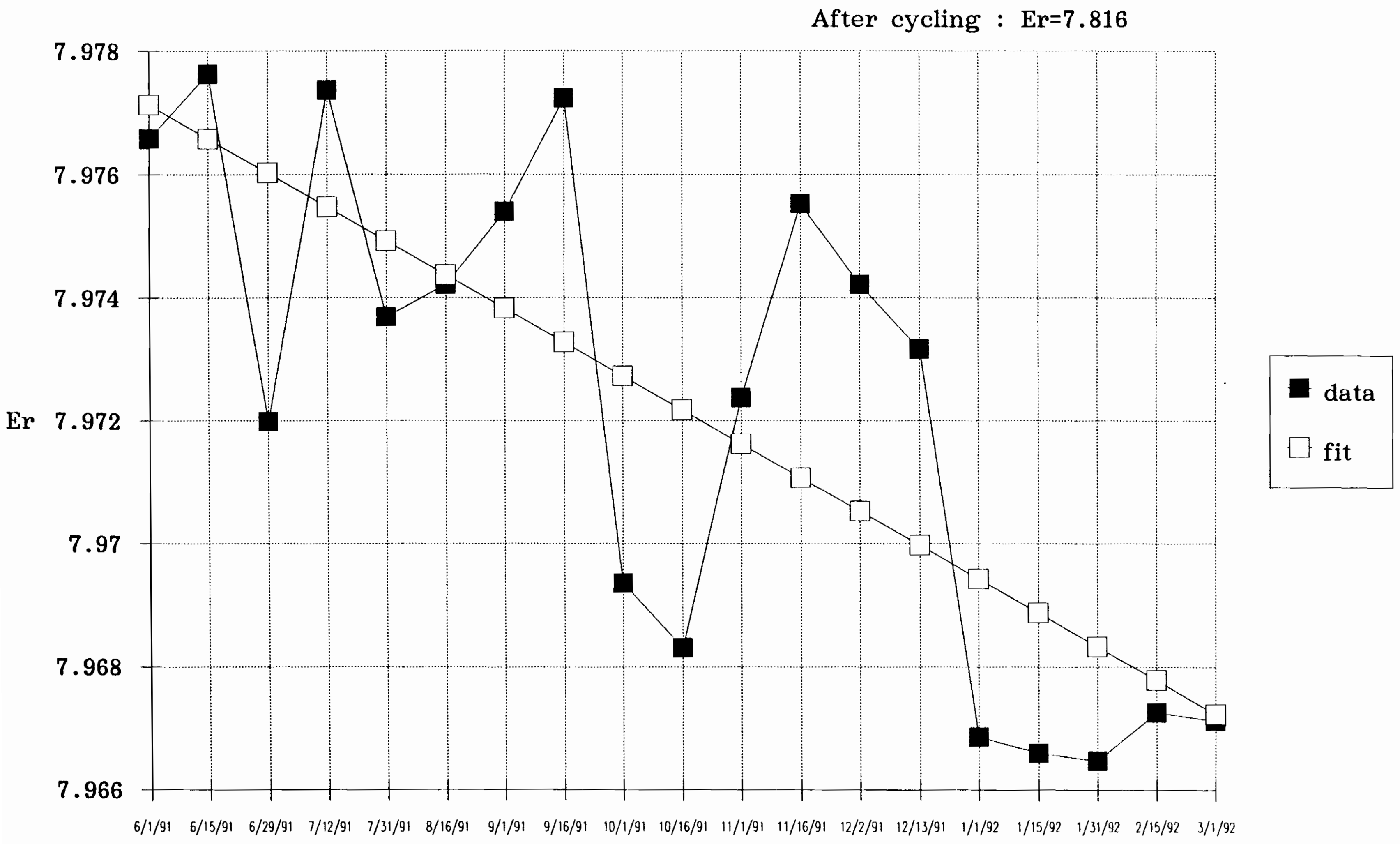


Figure 5.5 - Green Tape Dielectric Constant vs Time (f=100kHz)

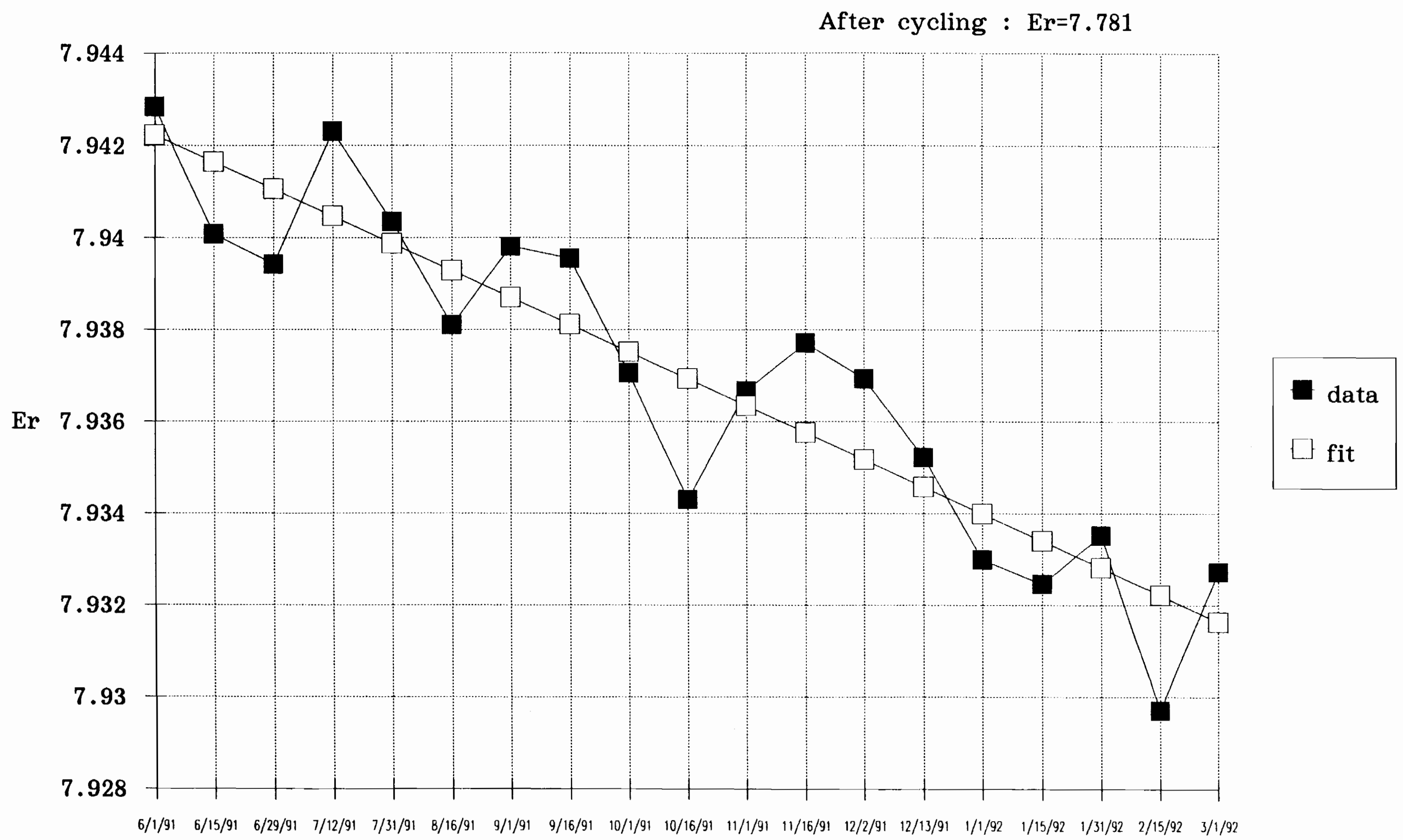


Figure 5.6 - Green Tape Dielectric Constant vs Time (f=1MHz)

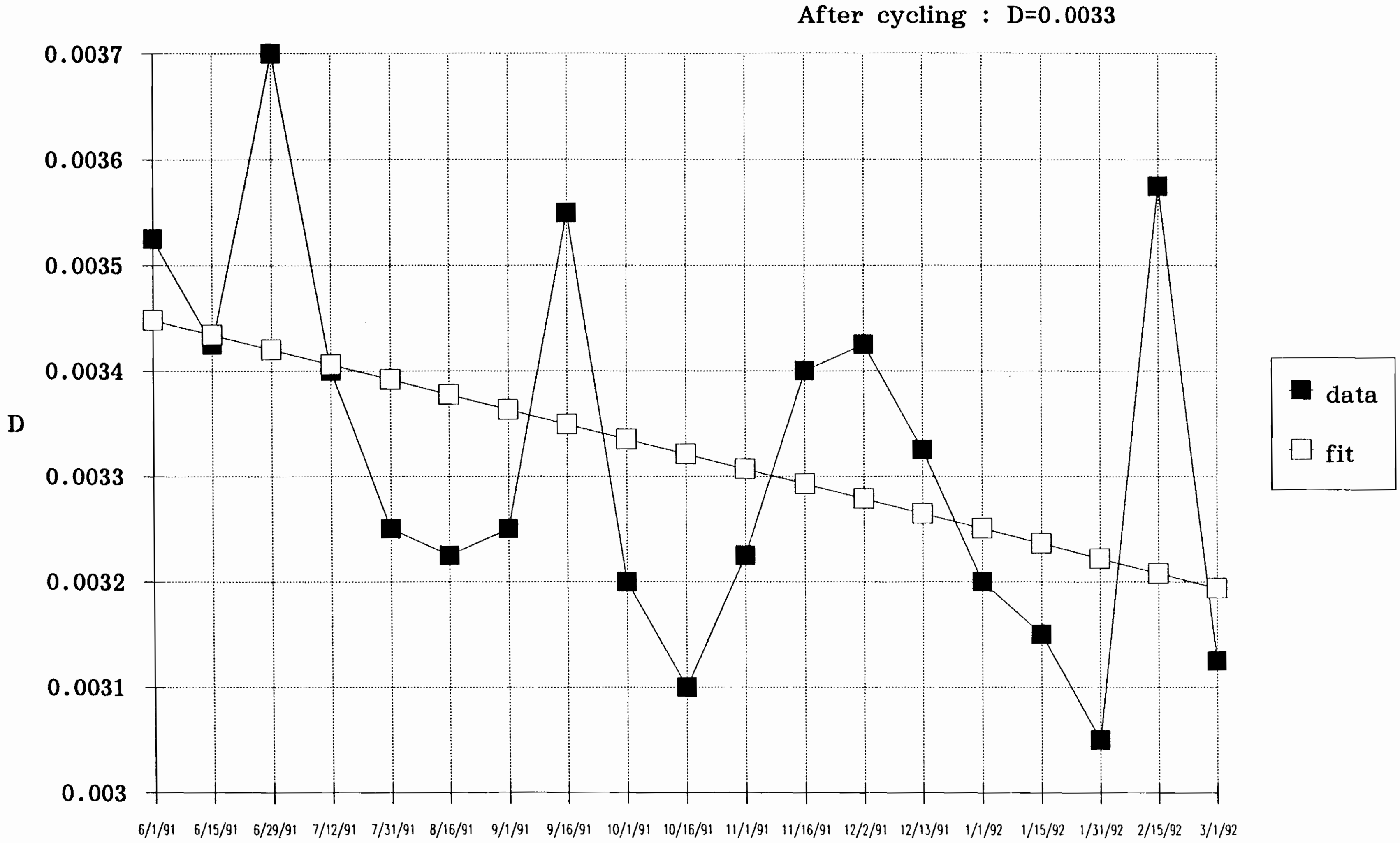
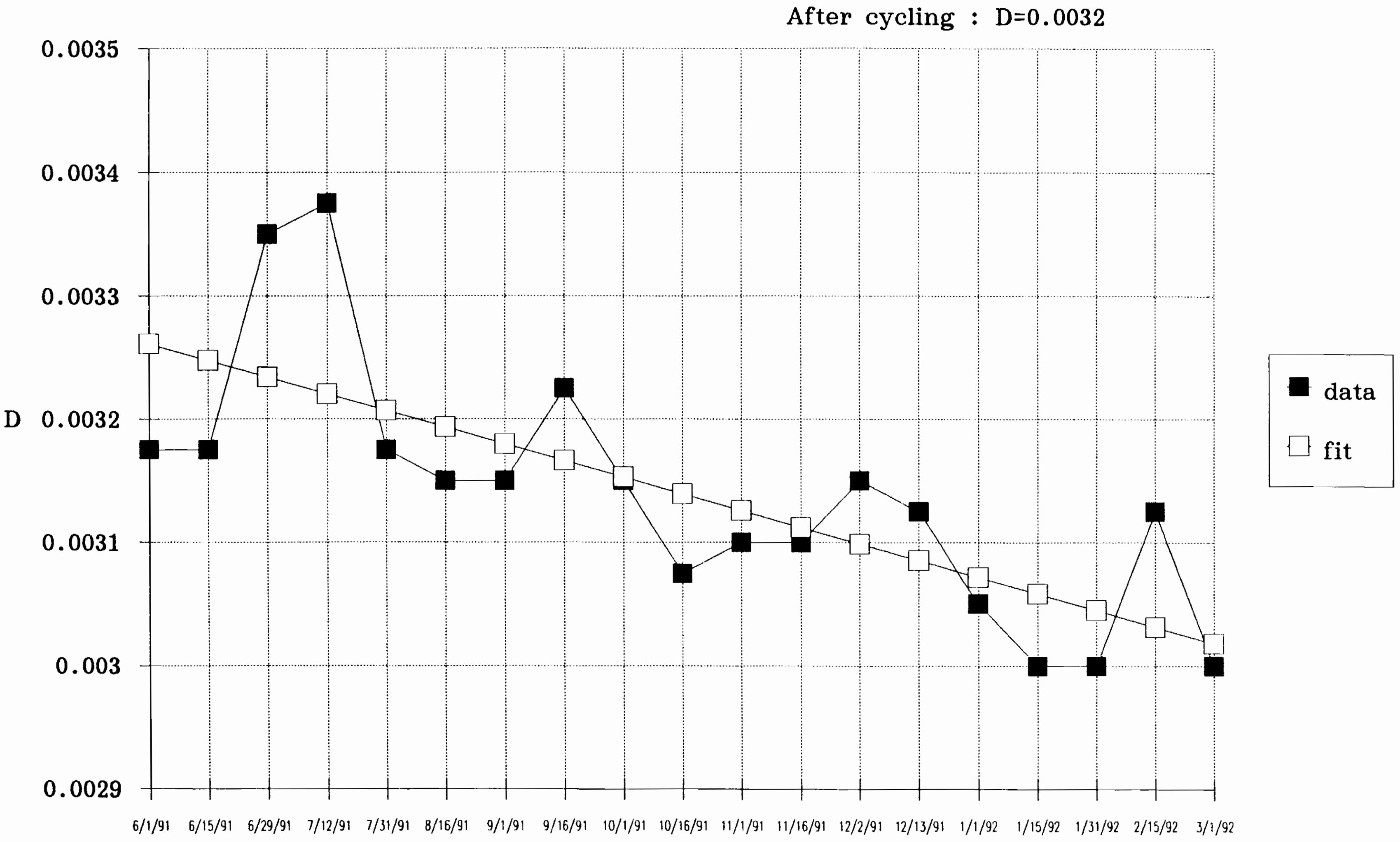


Figure 5.7 - Green Tape Dissipation Factor vs Time (f=100kHz)



After cycling : D=0.0032

Figure 5.8 - Green Tape Dissipation Factor vs Time (f=1MHz)

CHAPTER 6

CONCLUSIONS

This research involved the wideband characterization of the complex dielectric constant of low-loss dielectric materials used in multilayer structures. In particular, two DuPont multilayer materials - Pyralux® and Green Tape^{TM}, are considered in the research work. Specifically, the study was composed of three primary tasks. First, an extensive array of environmental tests were performed to determine the effects of operating temperature and relative humidity on the complex dielectric constant of these materials at low frequencies. Some very important trends were discovered, relating material type and thickness to the operating temperature, relative humidity, and frequency. This information will be useful in predicting the behavior of multilayer circuits in various environments. Secondly, measurements of the complex dielectric constant of Pyralux® and Green Tape^{TM} were made at high frequencies, under ambient conditions. These results compare very well with the results of earlier experimentation involving different test structures and measurement techniques. This information is growing increasingly important in microwave circuit applications. Finally, two types of lifetime tests were performed in order to determine the effects of aging on the complex dielectric constant of Pyralux® and Green Tape^{TM}. A natural aging of the materials has provided information on how this electrical parameter changes over time, independent of marked environmental variations. A forced aging of the materials, by thermal cycling, provided support for the conclusions drawn from the long-term aging data. These results are important in order to understand how the electrical characteristics of these materials change during their lifetime.

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APPENDIX A

Pyralux® Processing

1. Design structure layers with CAD program.
2. Plot patterns on rubylith.
3. Photoreduce patterns.
4. Blank necessary parts for etching.
5. Etch using the spray etcher.
6. Turn on drying oven, and set the temperature to 100° C.
7. Blank the remaining, non-etched parts of the structure.
8. Punch out any window regions in the appropriate layers, using the laminating die pins for accurate alignment.
9. Wearing gloves at all times (to avoid finger oil contamination), clean all parts with a lint-free (chamois) cloth. Use acetone, if necessary, to clean the etched parts.
10. Dry all cleaned parts in the oven for one hour. (Thinner sheets should be weighted down to prevent them from blowing around in the oven.)
11. Turn on the press equipment, and set the platen temperature to 350° F.
12. Blank out soft-pad system (*) if necessary:

laminating die (top)
* 200 angstrom FEP film
* bond paper
* clear vinyl sheet
* bond paper
* 200 angstrom FEP film
structure to be laminated (with window areas toward the soft-pad layers (*))
laminating die (bottom)

NOTE: The same soft-pad system (*) may be repeated on the bottom side of the structure if there are window regions on that side as well.

13. Remove parts from oven and store under laminar flow hood until cooled to room temperature.
14. Stack the parts in the appropriate order in the laminating die at room temperature.
15. Place die in heated press and add 335 psi of pressure.
16. Maintain both heat and pressure for one hour.
17. Turn off the heat, but maintain exerted pressure in order to prevent warping of the sample during the cooling process.
18. Gradually increase the flow of cooling water through the platens.
19. As the platen temperature approaches room temperature, slowly release the pressure.
20. Remove the die from the press.
21. Remove laminated sample from the die, and examine.

APPENDIX B

Green Tape^{TM} Processing

1. Design structure layers with CAD program (remember shrinkage factor).
2. Plot patterns on rubylith.
3. Photoreduce patterns.
4. Create the necessary screens for printing.
5. Wearing gloves at all times (to avoid finger oil contamination), blank out the required number of layers.
6. Form vias in the appropriate layers using either the programmable drill or the laser unit.
7. Turn on the drying oven, and set the temperature to 120° C.
8. Turn on press equipment, set the platen temperature to 70° C, and store the laminating die between the platens.
9. Turn on the belt furnace, and set the zone temperatures and belt speed as follows:

zone 1: 700° C
zone 2: 860° C
zone 3: 860° C
zone 4: 855° C
belt speed: 1.87 in./min.

10. Prepare the printing equipment for Green Tape^{TM} printing (i.e. use the porous stone stage)
11. Using the correct screen, fill the vias with the via-fill paste. (Remember to use lens paper under the tape layer.) Or, using the Mylar backing of the Green Tape^{TM} as the stencil, fill the vias by hand with a squeegee.
12. Dry the filled layers in the oven for 5 minutes.
13. Remove the filled layers from the oven and examine by microscope.
14. Discard any layers with improperly filled vias, and redo them.
15. After all vias have been filled properly, replace the via-fill screen with the first conductor screen, and change to the correct conductor paste. (Keep the lens paper in place to prevent the vacuum from pulling out the dried vias.)
16. After all layers have been printed with the appropriate conductor pattern, dry them in the oven for 5 minutes.
17. Remove the printed layers from the oven, and examine.
18. Correct or discard and redo any defective layers.
19. After all layers are inspected, remove the laminating die from the press, and correctly stack the layers in the die.
20. Place the die back into the press, and add 3000 psi of pressure.
21. Maintain both heat and pressure for 10 minutes.
22. Release the pressure (slowly, to prevent warping of the sample), and remove die from the press.
23. Remove laminated sample from the die.
24. Carefully cut away the registration holes, using a hot blade or laser, if necessary.
25. Clean an alumina substrate, or setter, (of larger size than the trimmed laminate) with acetone and compressed air.
26. Place laminated sample on the cleaned setter, and place setter in the ashing furnace.
27. Set the ashing furnace profile as follows:

ramp 1: 8° C/min.
temp 1: 100° C
hold 1: 0 min.
ramp 2: 8° C/min.

APPENDIX B (cont.)

temp 2: 250° C
hold 2: 0 min.
ramp 3: 8° C/min.
temp 3: 350° C
hold 3: 60 min.
ramp 4: 10° C/min.
temp 4: 250° C
hold 4: 0 min.
ramp 5: 10° C/min.
temp 5: 100° C
hold 5: 0 min.
ramp 6: 10° C/min.
temp 6: 30° C
hold 6: 0 min.
fan: ON

28. After cycle is complete, carefully remove sample (with setter) from the ashing furnace, and place it on the belt furnace.
29. After firing, remove setter and sample from belt, and proceed with any post-fired processing.

VITA

J. Shawn Addington received a Bachelor's Degree in Electrical Engineering from the Bradley Department of Electrical Engineering at Virginia Tech in May 1990. In the summer of 1990, he began working toward a Master's Degree in Electrical Engineering at the same University. As a Master's student, he has worked as both a Graduate Research Assistant, as well as a Graduate Teaching Assistant. As a DuPont Fellow, he also had the opportunity to work one summer (1991) at the DuPont Electronics Facility in Research Triangle Park, North Carolina, performing wideband electrical characterizations of dielectric materials. In March 1992, he received the Harry Lynde Bradley Fellowship from the Bradley Department of Electrical Engineering at Virginia Tech. In May 1992, he will receive a Master's Degree in Electrical Engineering from Virginia Tech, and plans to continue his studies toward a Doctoral Degree in Electrical Engineering at the same University.

J. Shawn Addington