S-349

NONLINEAR MICROWAVE DIELECTRIC MATERIALS

RAYTHEON

XEROX

Report No. 4
Contract No. DA 36-039SC-87369
Order No. 40536-PM-61-93-93
PR&C No. 60-ELE/R-5113

Final Progress Report

15 September 1960 - 14 September 1961

U.S. Army Signal Research and Development Laboratory

Fort Monmouth, New Jersey

RESEARCH DIVISION
RAYTHEON COMPANY
WALTHAM 54, MASSACHUSETTS

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Object
Conduct research and development investigations to develop nonlinear dielectric materials.

Prepared by
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R. O. Bell, B. diBenedetto
J. S. Waugh

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1. PURPOSE

Work under this contract was directed toward the investigation and development of nonlinear dielectric materials useful for application in the microwave region.

2. ABSTRACT

The loss tangent and the nonlinearity constant in polycrystalline mixtures of (Ba, Sr)TiO_3 have been studied. It was found that the nonlinearity constant and the part of the loss tangent which originates from fourth order anharmonic scattering stays essentially constant as a function of barium concentration, whereas the impurity contribution to the loss tangent varies appreciably. This indicates that barium acts as a lattice imperfection in strontium titanate. A microstrip phase shifter with $SrTiO_3$ as a voltage sensitive element has been built and tested.

3. PUBLICATIONS, LECTURES, AND CONFERENCES

3.1 Publications

G. Rupprecht, R. O. Bell, and B. D. Silverman, "Nonlinearity and Microwave Losses in Cubic Strontium Titanate," Phys. Rev. 123, 97 (1961).

3.2 Lectures

G. Rupprecht, Ferroelectricity in $SrTiC_3$, Solid State Seminar, University of California, Berkeley, California.

3.3 Conferences

J. Charlton of the Signal Corps and G. Rupprecht of Raytheon Company met to discuss the contract on September 25 at Waltham.

4. FACTUAL DATA

4.1 Introduction

The following brief summary of the ferroelectric properties of $SrTiO_3$ is intended to give enough background that the conclusions drawn in this report may be readily understood.

There is strong evidence that the ferroelectric behavior of $SrTiO_3$ arises from an expected instability of an optical mode at the Curie temperature T_C. From infrared measurements one can conclude that this soft mode may be identified with the normal mode which involves the vibration of the Sr sublattice against the sublattice of the TiO_3 octahedra. The absorption peaks in the near infrared have been attributed to two optically active normal vibrations of the TiO_3 octahedron. An analysis of this situation in the harmonic approximation leads to the conclusion that the presence of the TiO_3 octahedron plays an important part in explaining ferroelectric properties. In essence the TiO_3 sublattice provides a medium of large dielectric constant for the embedded Sr ions whose collective motion becomes unstable. Before this instability can set in, however, a phase transition at 112 °K occurs. The details of this transition are still under investigation. A great deal of information has been collected by a variety of independent experiments. The results indicate that this phase transition is due to a change of the oxygen configuration.

An important feature of our work schedule is to obtain a more basic understanding of the origin of the microwave losses. To date we have been successful in interpreting most of the microwave loss data in a qualitative fashion. Further results from a systematic experimental program now in progress should clarify many points which are now somewhat speculative. Our ideas concerning the loss mechanism are briefly reviewed.

It is assumed that the microwave radiation field couples into a long wavelength optical mode of the lattice. The electric field drives this soft mode in a forced vibration. The energy loss is believed to result from the scattering of microwave phonons out of this mode into other modes of the material. The scattering can result from the presence of imperfections or anharmonic interactions in the lattice. As the temperature is lowered the frequency of the long wavelength optical mode is reduced and can be driven more effectively by the microwave field. The increase in the loss tangent as the temperature is lowered is then due to this improved match between the lattice and microwave frequencies. The loss tangent of single crystal strontium titanate goes through a minimum at about 170°K and increases roughly linearly with temperature at higher temperatures. This can be explained if anharmonic interactions are held responsible for the scattering of phonons out of the long wavelength mode.

The experimental results on doped material also can be understood qualitatively with this assumption. The addition of barium to single crystalline strontium titanate, for instance, modifies the low temperature value of the loss tangent without affecting the high temperature linear component.* This strongly suggests that the high temperature contribution is intrinsic to the lattice and possibly related to anharmonic interactions. A different behavior is observed for the addition of gadolinium to strontium titanate, where the loss tangent is enhanced at all temperatures investigated. This is not surprising since a trivalent ion is being substituted for a divalent ion. A disturbance of the charge periodicity enables the microwave field to couple into modes with wavelength different from the wavelength of the radiation field in the material. On the other hand, the introduction of oxygen deficiencies into the lattice does not affect the loss tangent. We suspect that the vacancies are, each, charge compensated and therefore not very effective as microwave phonon scatterers. The reason may be that the ionic potential of the Sr ions in the vicinity of an oxygen vacancy show only insignificant modification when an oxygen ion is replaced by a "color center." This result seems to support the initial assumption that ferroelectricity in $SrTiO_3$ comes from the Sr ions in a highly polarizable surrounding of TiO_3 octahedra.

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*This has been shown for polycrystalline ceramic material in a previous report.

4.2 New Materials - Cadmium Niobate

Preliminary studies on cadmium niobate have been started. Cadmium niobate becomes ferroelectric below -90°C and has room temperature dielectric losses nearly equal to BST-50 at 21 kMc. The crystal structure is cubic pyrochlore with a lattice constant of a = 10.371 Å.

Both hot pressed and dry pressed ceramics and small single crystals have been prepared. The hot pressed samples are still too lossy to be measured, but the dry pressed specimens have room temperature dielectric constants (1 kc) of c.a. 1000 and losses c.a. 0.03. The microstructure reveals that the crystals in the dry pressed ceramics are almost always twinned.

Since CdO vaporized quite easily from the lattice, care must be taken to prevent this volatilization which lowers the Curie temperature, the dielectric constant, and the dielectric losses.

A Curie constant of 8.3×10^4 °K was calculated from a Curie-Weiss plot for dry pressed ceramics. Data on the hot pressed ceramics or the single crystals are not yet available.

Hysteresis data have been obtained on polycrystalline samples at temperatures from -180°C to 0°C. The saturation polarization just below the Curie point (-108°C) is 5.14μ coulombs/cm^2.

Single crystal platelets about 1 mm on a side and 0.5 mm thick have been grown from a KF flux.

Work will continue on hot pressing $Cd_2Nb_2O_7$ as well as growing larger single crystals. A slab line specimen is now being prepared for measuring of the nonlinearity at microwave frequencies.

4.3 Materials Improvement Studies

Work on perovskite-type materials has been continued on the solid solution system of barium titanate and strontium titanate. In this system the Curie temperature can be shifted at will over a wide range. It is the purpose of this investigation to find out how properties and losses in these mixtures compare with those of the strontium titanate single crystals. The work on polycrystalline material was continued because of its importance for practical applications.

4.3.1. Single Crystal Growth of $(Ba_xSr_{1-x})TiO_3$

The flux method was chosen for the growth of these solid solution crystals with potassium fluoride as a flux.. At 1200°C, approximately 10 mole percent titanate would dissolve in the potassium fluoride flux.

The first crystals grown were the end members of the solid solutions, $SrTiO_3$ and $BaTiO_3$. The $SrTiO_3$ crystals were small and had a yellow-brown coloration in contrast to the colorless Verneuil grown crystals. The loss tangent was slightly higher in these crystals but no significant differences could be found between both types of crystals concerning their dielectric behaviour. The $BaTiO_3$ crystals appeared in the characteristic butterfly twins. The first solid solution crystals were then grown from a starting composition of $(Ba_{.5}Sr_{.5})TiO_3$. The crystals that resulted were agglomerates of several crystals. Small single crystalline areas could be separated from the agglomerate.

Two pieces of information were used for the identification of the crystal composition, the Curie temperature and lattice parameter. The maximum of the dielectric constant was measured and compared to results from known polycrystalline samples. Lattice parameters were measured with a GEXRDS x-ray diffractometer and compared to parameters from known samples.

Identification of the early solid solution crystals grown from the starting composition, $(Ba_{.5}Sr_{.5})TiO_3$, indicated that the crystals were agglomerates consisting of several small crystals of different compositions. One small single area was separated from the agglomerate and measured by itself. This area turned out to have a Curie temperature of -178°C corresponding to a crystal composition of $(Ba_{.16}Sr_{.84})TiO_3$.* This was 34 mole percent higher in strontium than the starting composition. It was then decided to grow crystals with a high concentration of barium. Starting compositions were: $(Ba_{.9}Sr_{.1})TiO_3$, $(Ba_{.8}Sr_{.2})TiO_3$, and $(Ba_{.7}Sr_{.3})TiO_3$. These crystals had compositions close to their starting composition. However, the maximum of the dielectric constant became less pronounced and wider with increasing strontium concentration.** This indicated that the agglomerates were possibly of several compositions.

Since the last report crystals have been grown from starting compositions $(Ba_{.6}Sr_{.4})TiO_3$ and $(Ba_{.5}Sr_{.5})TiO_3$. These crystals were also agglomerates. This was borne out by the measurement of the dielectric constant as a function of temperature exhibiting several independent peaks at temperatures from -180°C on up to the Curie temperature of pure $BaTiO_3$. Different crystals from the same batch show different peaks and different ranges of peaks.

The formation of agglomerates was observed for all those crystals runs where crystallization took placed in the whole temperature range from 1200°C to 800°C. It is evident that the barium-strontium molar ratio of the precipitating solid solution was changing during crystallization. A preliminary investigation indicated that the first crystallization had a high concentration of $SrTiO_3$ and that the barium-strontium ratios changed in favor of $BaTiO_3$ as crystallization continued to lower temperatures. The rounded

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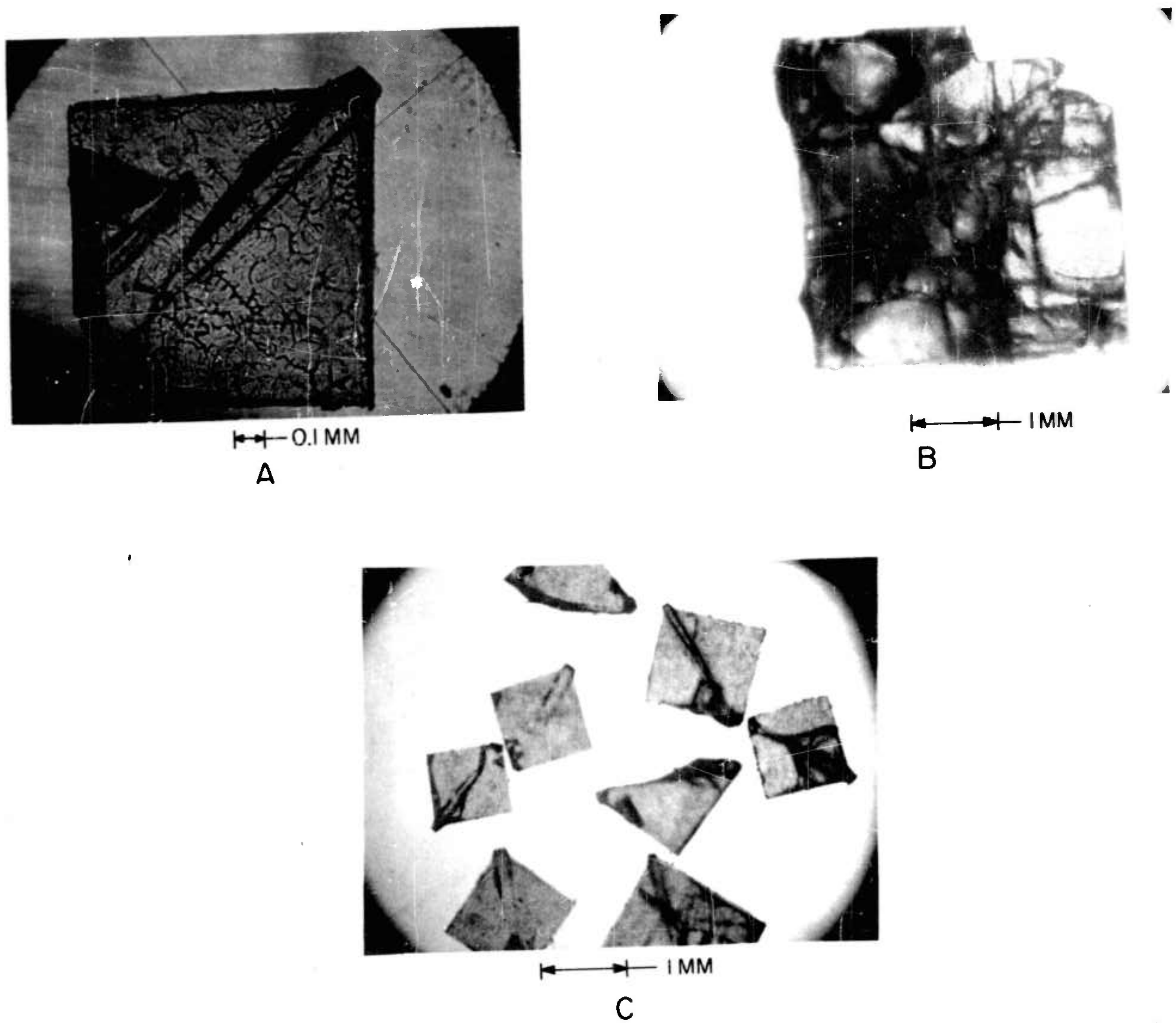
*See Fig. 1, Report No. 2 (DA 36-039 SC-87369).

**See Fig. 5, Report No. 3 (DA 36-039 SC-87369).

edges on the crystallites in the interior of the agglomerates showed that there was some redissolving of the original crystals as others appeared.

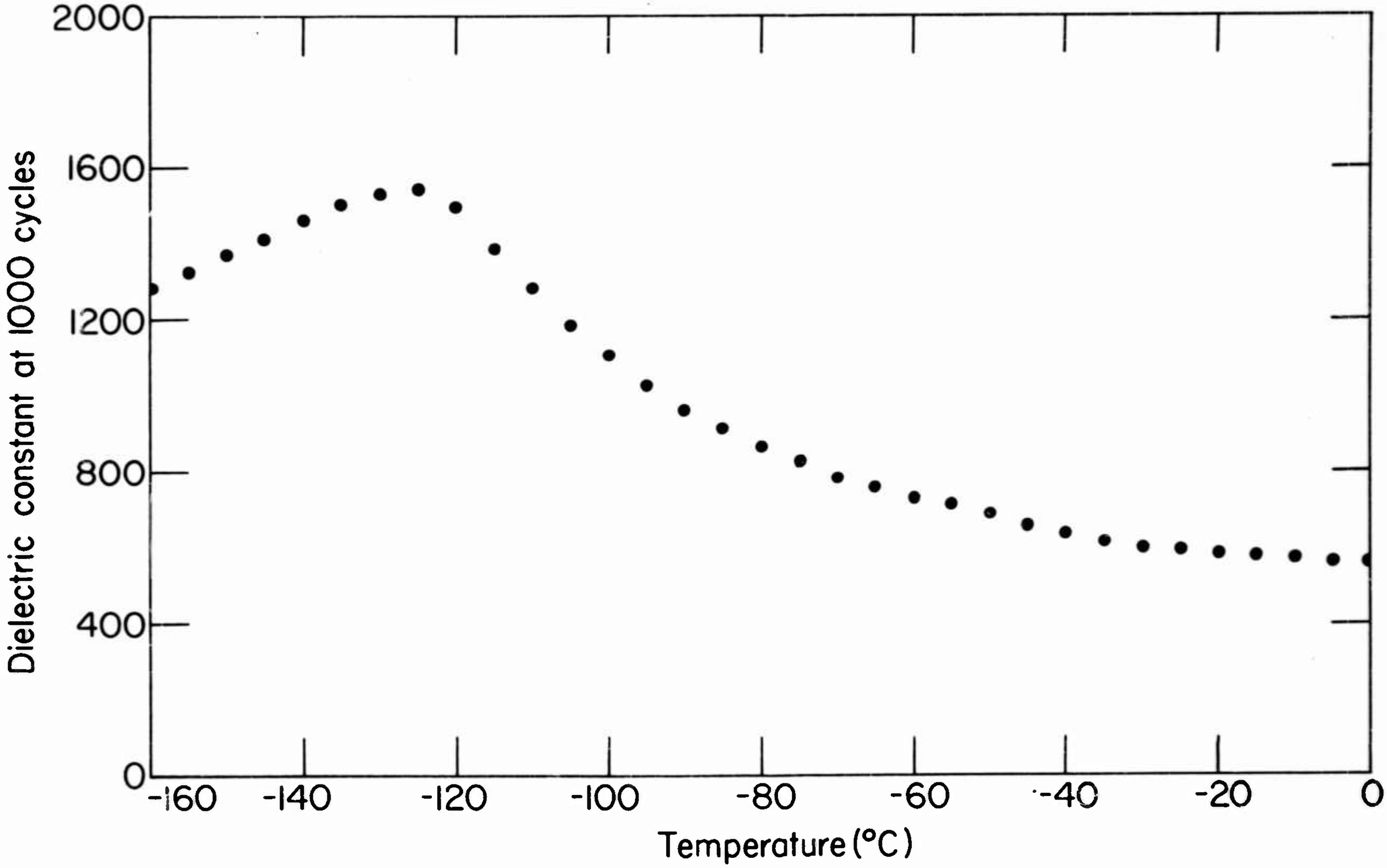
It, therefore, became necessary to control the range in which a desired composition will crystallize. A small portion of the crystallization range was selected, where the crystallizing solid solution was expected to vary the least in composition. This was felt to be the case at the beginning of the crystallization process, where, in our experience, the larger crystallites in the interior of the agglomerate had been produced. Now, the experimental procedure was the same as in previous runs with the exception that the crucible was quenched from an appropriate temperature to prevent redissolving of already formed crystals or co-precipitation of a different barium-strontium ratio. Quenching a starting composition $(Ba_{.5}Sr_{.5})TiO_3$ from approximately 1050°C resulted in single crystals (Fig. 1A and 1C). A comparison with the results of the previous method is shown in Fig. 1B. Aside from quenching, the experimental conditions were the same in both runs. The single crystals obtained by quenching had a Curie temperature of -135°C (Fig. 2) which can be related to a crystal composition of $(Ba_{.25}Sr_{.75})TiO_3$. Another run was then made under identical conditions with a starting concentration $(Ba_{.6}Sr_{.4})TiO_3$. The resulting crystals were single with a Curie temperature of -95°C (Fig. 3) indicating a crystal composition of $(Ba_{.4}Sr_{.6})TiO_3$.

Annealing of these crystals was started for several purposes. It was expected that at high enough temperatures, for long periods of time, some of the impurities such as K^+ and F^- might volatilize. If cooled sufficiently slowly, a release of the strains caused by the quenching may also be expected. Further, at sufficiently high temperature and extended period of time the diffusion of the Ba^{++} and Sr^{++} ions might reduce compositional gradients. Preliminary measurements of the dielectric constant on $(Ba_{.25}Sr_{.75})TiO_3$ crystals, which were annealed at 1000°C, 1200°C, and 1400°C for 10 hours showed no noticeable changes.



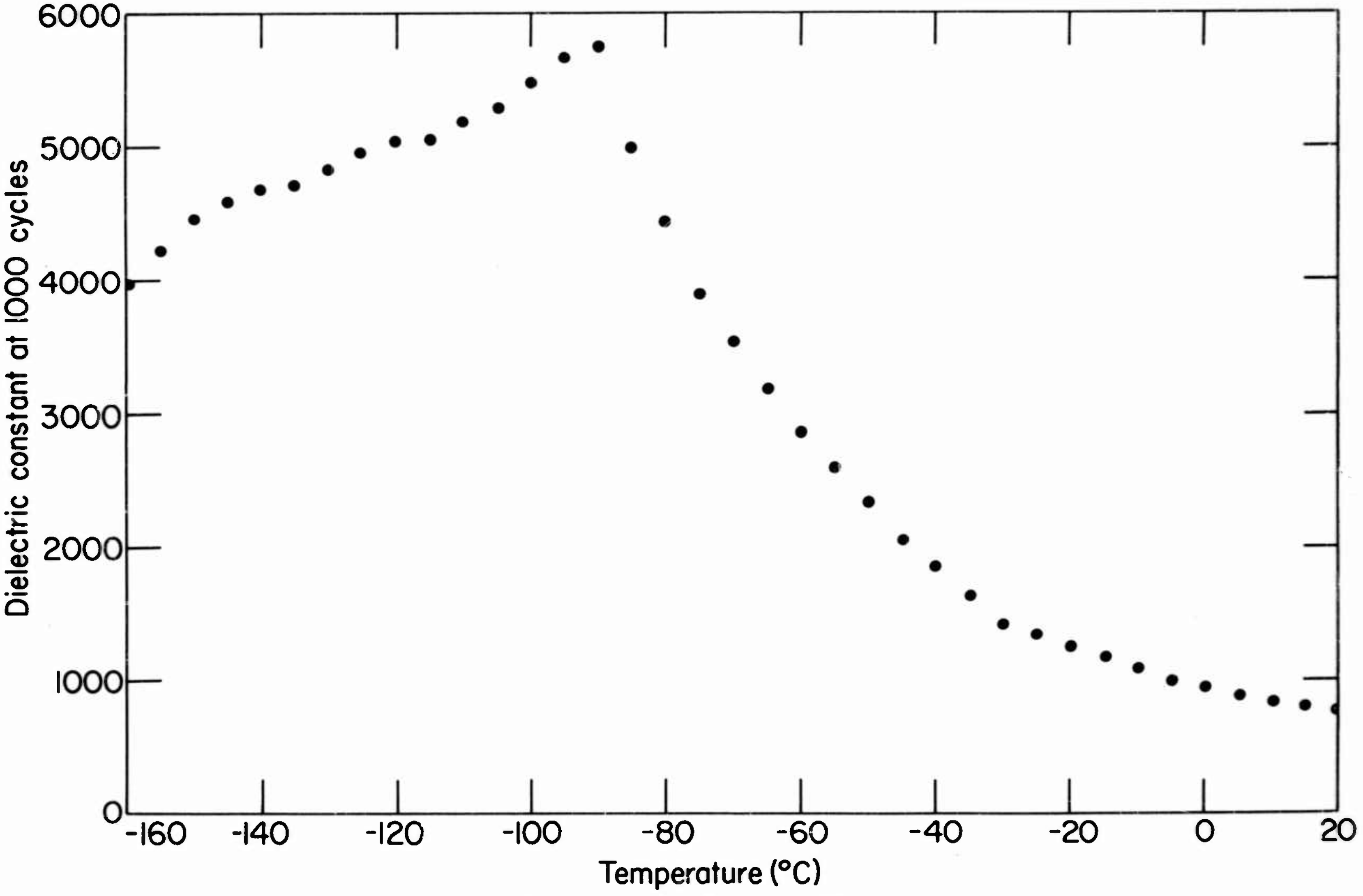
CRYSTALS GROWN FROM A STARTING COMPOSITION OF $(B_{4.5}Sr_{0.5})$ T:O_3 = A & C ARE SINGLE CRYSTALS FROM QUENCH RUNS B AGGLOMERATE FROM RUN OVER TOTAL CRYSTALIZING RANGE

FIGURE 1



BST-25 SINGLE X-TAL

FIGURE 2



BST-40 SINGLE X-TAL

FIGURE 3

4.3.2. Polycrystalline $(Ba_xSr_{1-x})TiO_3$

The hot pressing was concentrated on obtaining a $(Ba_{.5}Sr_{.5})TiO_3$ polycrystalline ceramic with nearly theoretical density. In order to achieve this goal the starting material must be uniform and small in particle size.

During this report period the development of proper starting materials was pursued. Two adequate preparation techniques have been found. Barium and strontium titanates were weighed out in molar ratios, mixed thoroughly by milling, and were then calcined to obtain a complete solid solution. This calcined material was then ground up by milling and the small particles were separated out by a sedimentation procedure. Powders of colloidal particle size were obtained by this method. The second technique consists of the chemical precipitation of $(Ba_{.5}Sr_{.5})TiO_3(C_2O_4)_2 \cdot 2H_2O$ (barium-strontium titanate oxalate tetrahydrate) which was calcined at 900°C to produce $(Ba_{.5}Sr_{.5})TiO_3$ in small particle size.

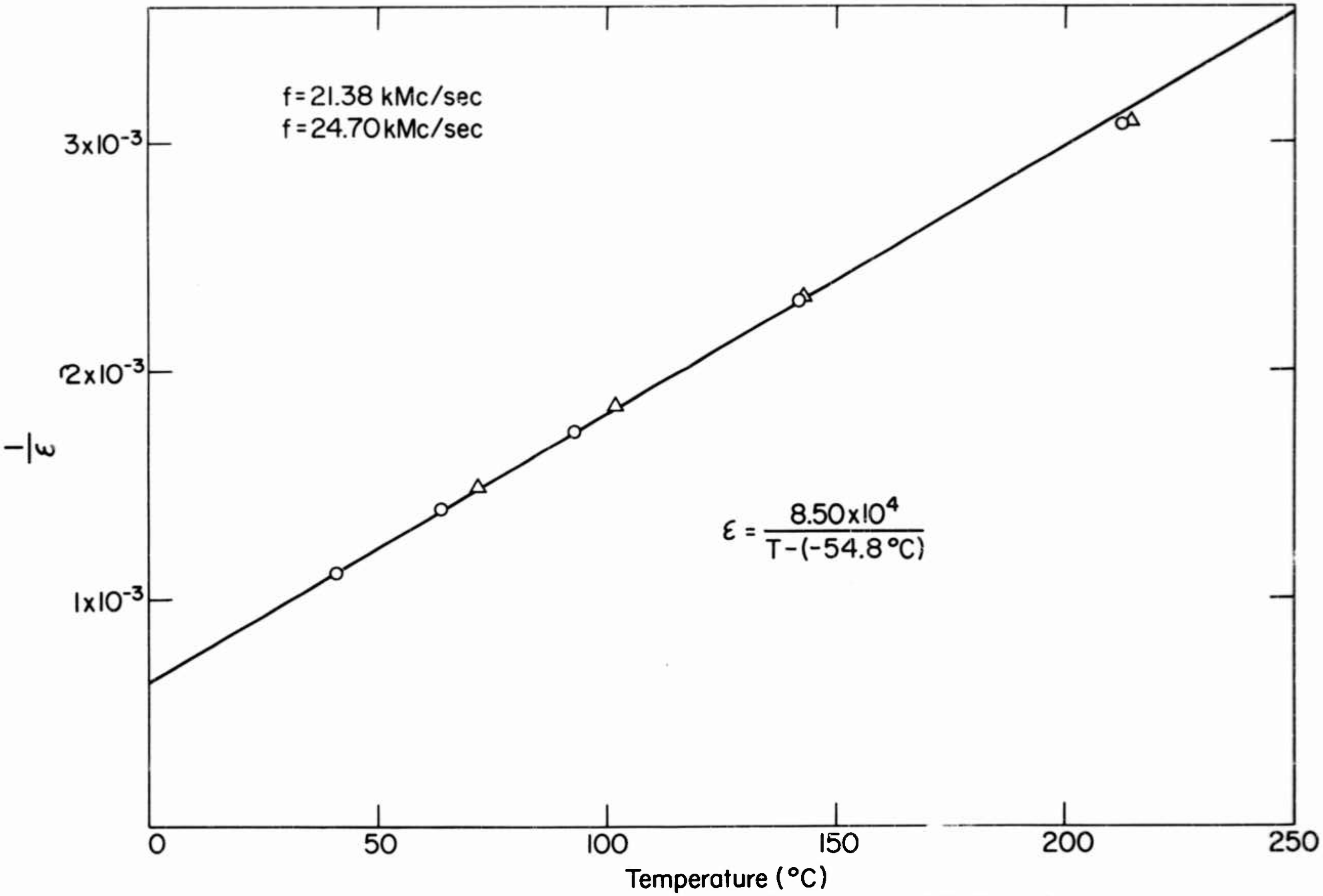
4.4 Dielectric Measurements

The dielectric constant of BST-50 hot press run number 7 was measured at K-band by observing dielectric resonances in a sphere of diameter 2.34 mm. Figure 4 shows the results of these measurements. The dielectric losses were also measured for this BST-50 sample and are in substantial agreement with previously reported data.

An investigation of the loss tangent of $Cd_2Nb_2O_7$ has been made at 22 kMc. Figure 5 shows $\tan \delta_o$ versus the temperature. The loss tangent at 22 kMc/sec is approximately

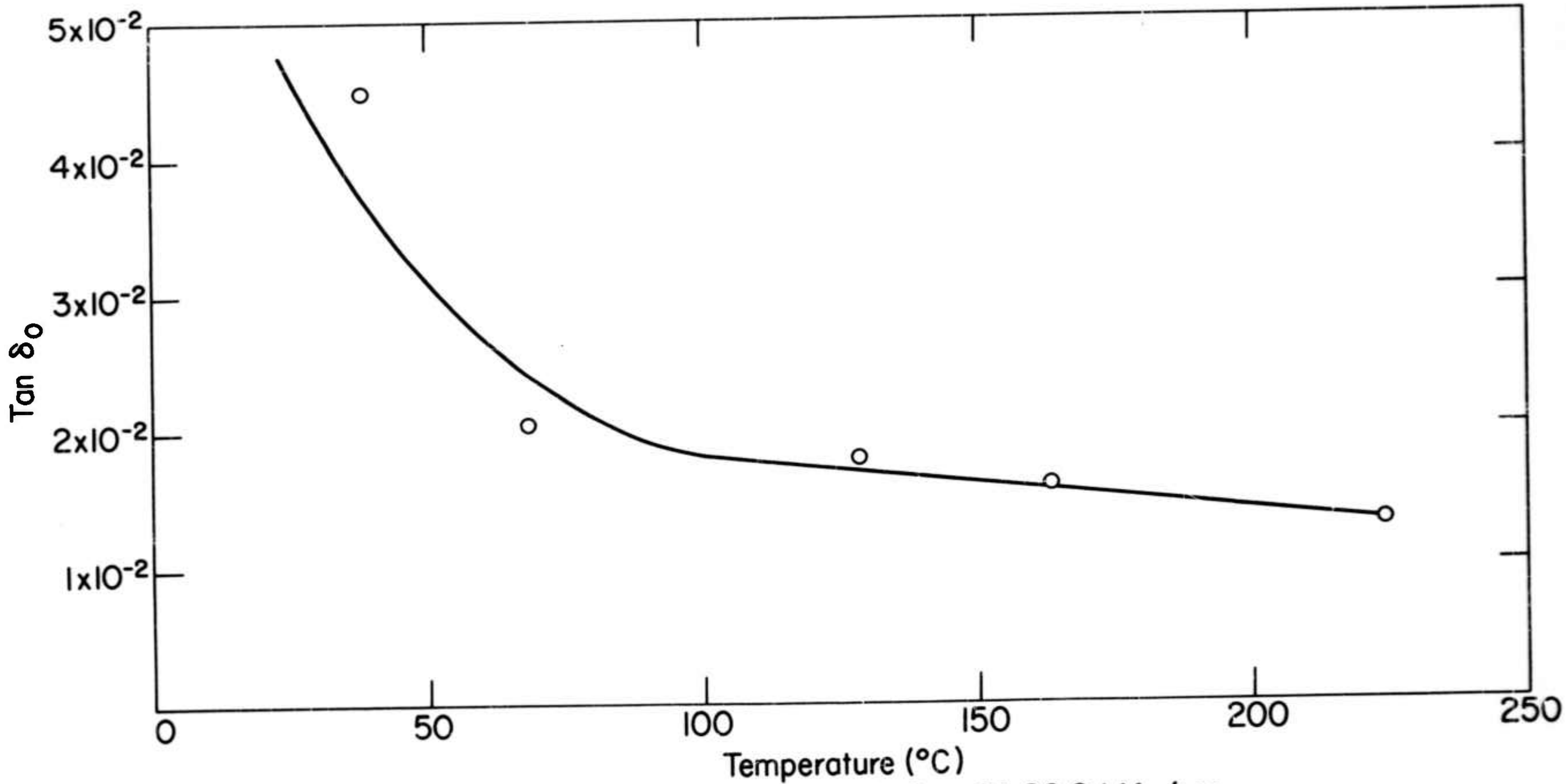
$$\tan \delta_o = \frac{\alpha}{T - T_c}$$

where $\alpha = 4.4$°K. This α is 1.8 times larger than the value for BST-50 at the same frequency. No attempt has yet been made to reduce this value by



CURIE - WEISS PLOT FOR BST - 50 H.P. RUN #7 FOR A 2.348 mm SPHERE

FIGURE 4



LOSS TANGENT OF $Cd_2Nb_2O_7$ AT 20.6 kMc/sec

FIGURE 5

annealing. A slab line of $Cd_2Nb_2O_7$ has been constructed to measure the microwave dielectric constant and nonlinearities, but results are not yet available.

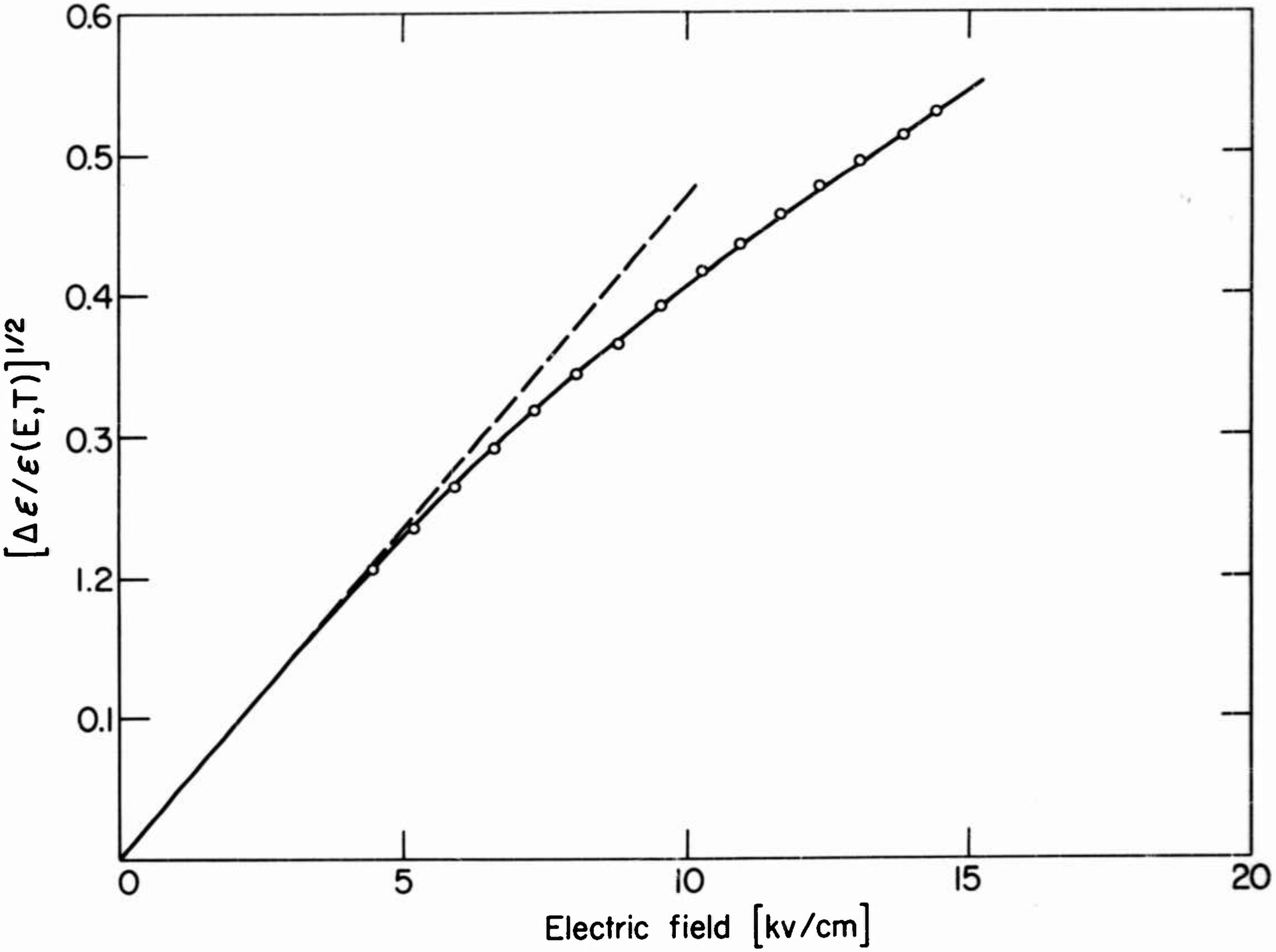
The nonlinear behavior of the dielectric constant of BST-50 H. P. below its Curie temperature has been observed at 1000 cps (Fig. 6). The nonlinearity constant for low fields is roughly the same as was obtained above the Curie temperature and at microwave frequencies. At higher fields the rate of change of the dielectric constant decreases.

In order to investigate the effect of composition on the nonlinearity constant <A>, a slab line of composition BST-20 was constructed. Figure 7 shows the results of measurements made at 5.00 kMc. Evaluation of the data yields $\langle A \rangle = 2.8 \times 10^{-18}$ (m/volt)^2 °K as compared to 1×10^{-18} (m/volt)^2 °K for BST-50 and single crystalline $SrTiO_3$. Since the sample was rather short, some difficulty was encountered with the identification of the modes of the slab line cavity. This result will be checked with a larger slab line.

The nonlinearity constant, A_{110}, was measured for a slab line of single crystalline $SrTiO_3$ doped with 0.1% gadolinium oriented in such a way that the electric field was applied along the [110] axis with the result $\langle A \rangle = 0.96 \times 10^{-18}$ °K (m/v)^2. This agrees with previous measurements on undoped $SrTiO_3$. The loss tangent at 21 kMc/sec of the gadolinium-doped sample was roughly 50% greater than undoped $SrTiO_3$ at the same frequency. An attempt to measure the losses on a sample of $SrTiO_3$ doped with 0.4% gadolinium was unsuccessful.

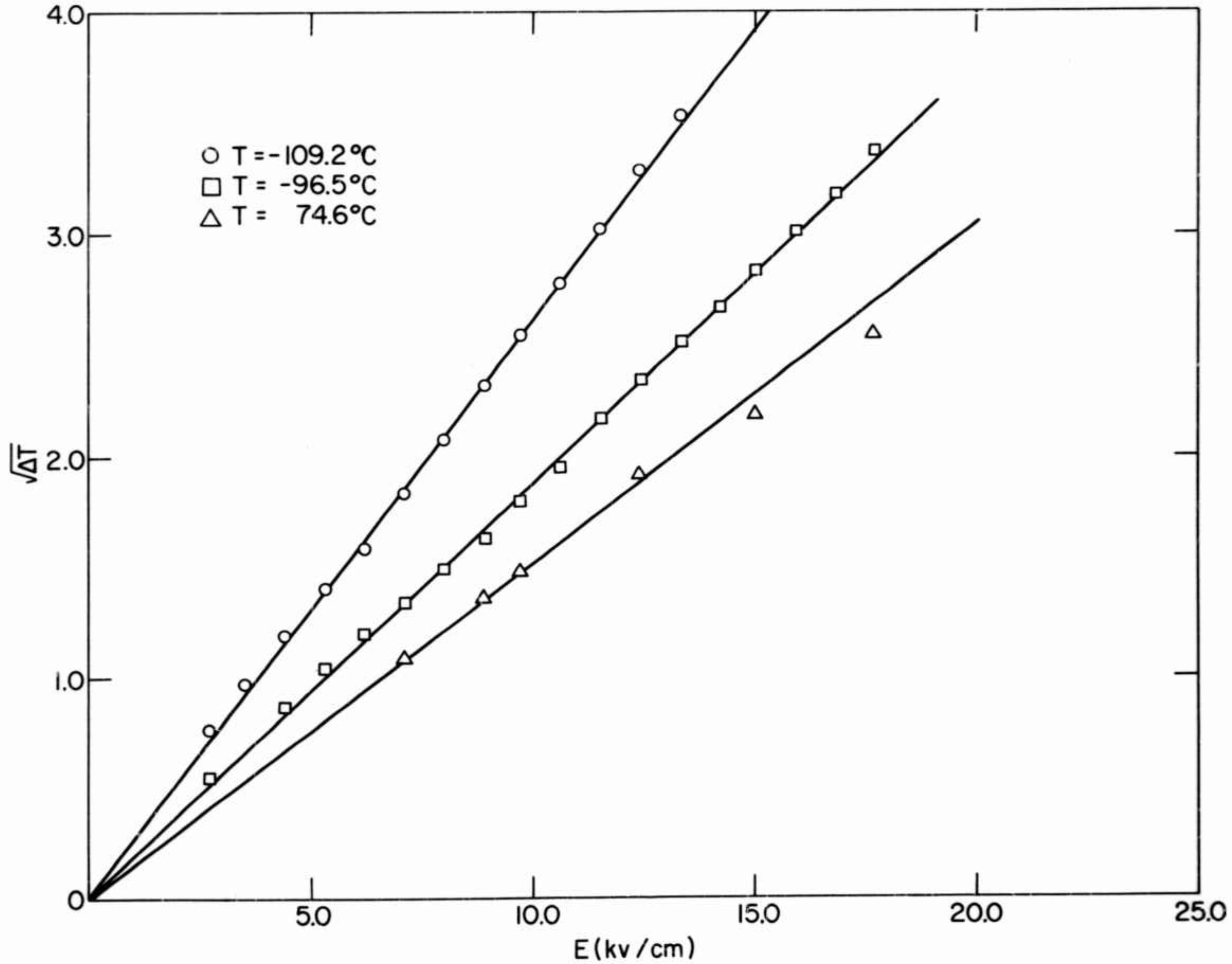
4.5 Phase Shifter

Single crystalline $SrTiO_3$ was used as the voltage sensitive element in a microstrip phase shifter. A top view is given in Fig. 8. Two transitions, coaxial-to-microstrip line serve as input and output terminals, and launch the microwaves into a microstrip with a height of 0.032". The impedance match of the $SrTiO_3$ crystal (0.976" length) was achieved by a TiO_2 transformer



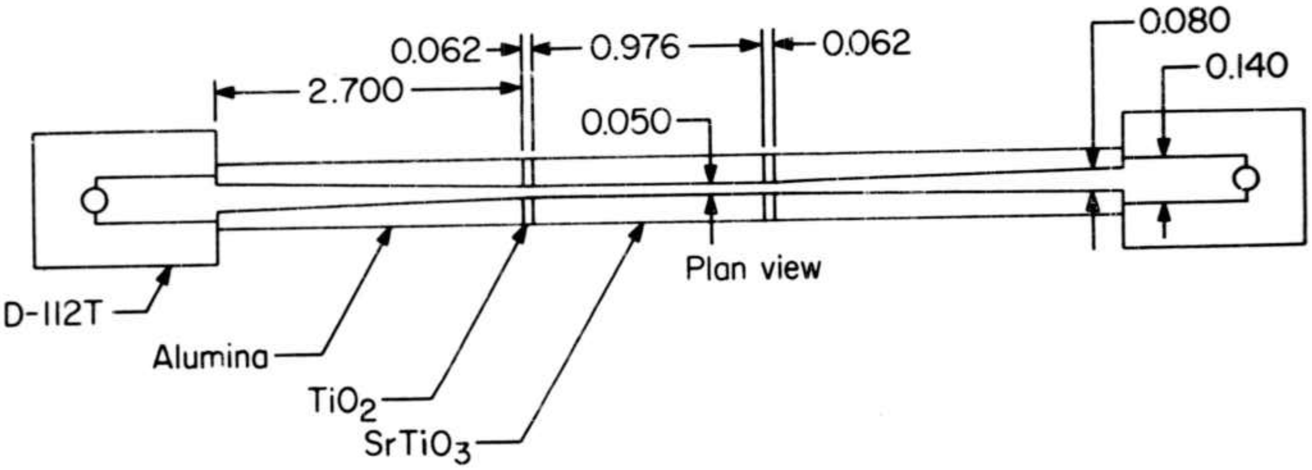
THE CHANGE IN THE DIELECTRIC CONSTANT AS A FUNCTION OF D.C. ELECTRIC FIELD AT -71 °C

FIGURE 6



$\sqrt{\Delta T}$ VERSUS APPLIED ELECTRIC FIELD FOR BST-20 AT 5.00 kMc/sec

FIGURE 7



TOP VIEW OF MICROSTRIP $SrTiO_3$ PHASE SHIFTER

FIGURE 8

between the $SrTiO_3$ crystal and the alumina stripline. The phase shifter was cooled with liquid nitrogen. The temperature of the voltage sensitive $SrTiO_3$ was probably close to 88°K. Figure 9 shows the phase shift ϕ in degrees as a function of applied voltage V for a frequency of 4.46 kMc. The insertion loss was 4.5 db. The standing wave ratio as a function of applied voltage is shown in Fig. 10.

5. CONCLUSIONS

The investigation of the microwave losses in single crystalline strontium titanate showed the existence of ferroelectric material with low microwave losses and gave an insight into the loss mechanism. In this connection the studies on polycrystalline strontium titanate became quite important. Annealing studies supported the basic assumption that the microwave losses are composed of two essential contributions: lattice imperfections and fourth order anharmonic scattering.

The microwave losses were measured on one and the same sample after subsequent annealings at increasing temperatures. The marked reduction of the loss tangent was attributed to the increase in grain size during this treatment (and therefore decreasing grain boundary scattering) and an expected perfection of the crystal lattice within the grains. These two effects, however, could not be separated in this experiment. Annealing studies of the polycrystalline hot pressed barium strontium titanate, however, gave additional information. It was observed that the grain growth in these mixed crystals during the annealing process was about the same as in hot pressed strontium titanate and it was therefore expected that the grain boundary scattering would be approximately the same in both cases. However, only an insignificant decrease of the loss tangent was obtained with subsequent annealing. This led to the conclusion that in the case of mixed crystals the barium ions act as impurities in the strontium titanate lattice, caused by impurity scattering. As expected under these circumstances the losses due to lattice imperfections increased with increasing barium concentration. The losses due to fourth order anharmonic scattering, on the other hand, remained unchanged. This is in agreement with

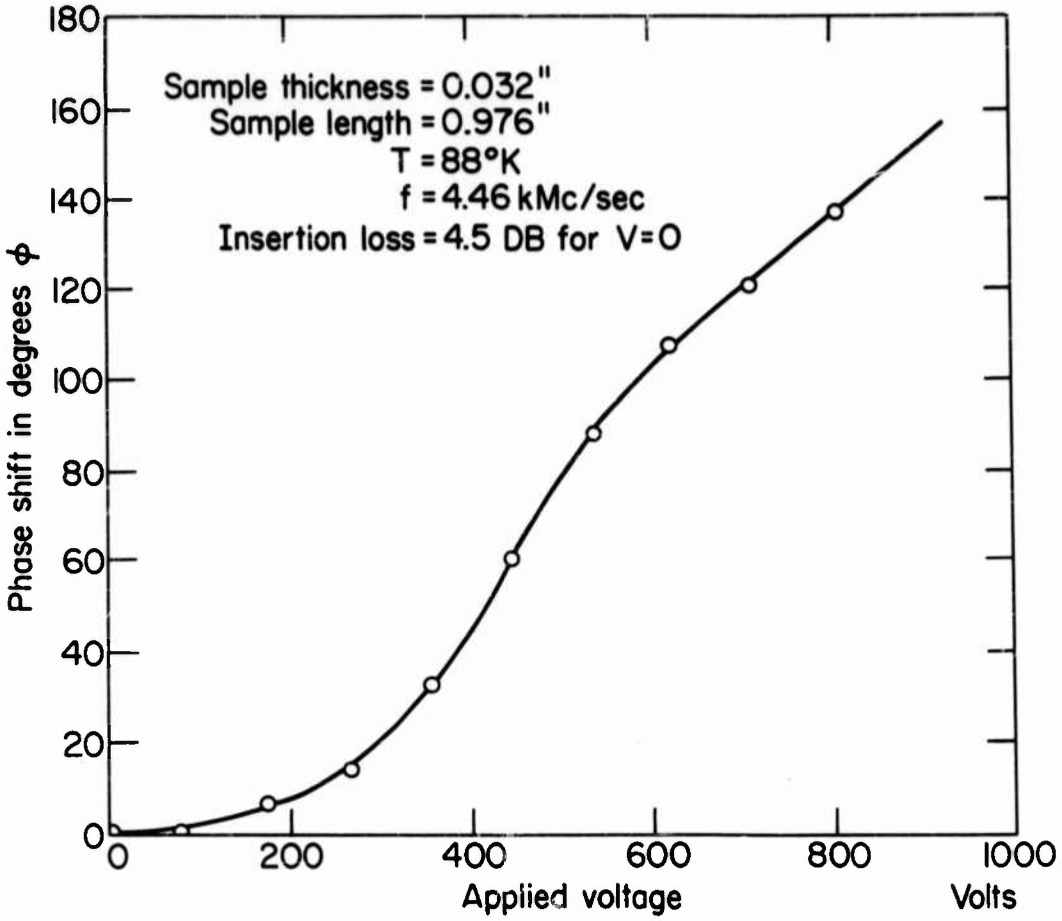
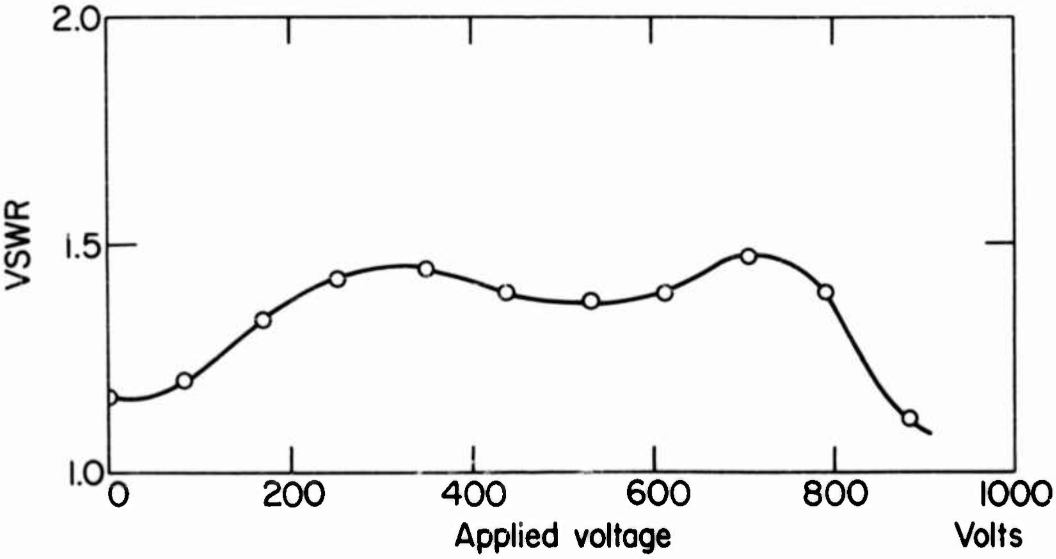


FIGURE 9



PHASE SHIFT AND STANDING WAVE RATIO OF MICROSTRIP AS A FUNCTION OF APPLIED VOLTAGE

FIGURE 10

a study of the nonlinearity constant <A> as a function of the barium concentration. Although <A> varies somewhat from sample to sample, it can be stated safely that the nonlinearity constant does not depend strongly upon the barium concentration. Since <A> is directly connected with the fourth order anharmonic terms in the potential of the strontium ions it appears reasonable that the losses from this source do not change with varying barium concentration. This result strongly supports the initial interpretation of the microwave loss mechanism.

The results quoted above are actually very disappointing. It was hoped that barium strontium titanate mixtures would exhibit similar low losses as was observed in strontium titanate. This negative result was accepted very reluctantly and not before single crystals of barium strontium titanate were available. These crystals, however, were grown by the flux method and therefore contain probably a relatively large amount of impurities. The uniformity is also by no means perfect. It is not until recently that reasonably uniform single crystals of barium strontium titanate have been obtained. These crystals, however, are not yet evaluated to be included in this report. Another piece of information, however, though not entirely convincing, is available. The loss tangent in flux-grown strontium titanate is only about 50 percent larger than the loss tangent in the flame fusion grown crystals. This indicates that the impurities which enter the crystal during its growth from the flux do not influence the loss tangent severely. The same situation might be expected to hold for mixed crystals. Therefore, all the evidence is pointing toward the result that barium acts as an impurity in strontium titanate. The final answer to this question, however, should depend on the outcome of some additional experiments which will exhaust rather completely the possibilities of obtaining low loss mixed (Ba, Sr)TiO_3 as a nonlinear dielectric material.

6. IDENTIFICATION OF PERSONNEL

Georg Rupprecht	- 80	man	hours
Bernard diBenedetto	- 104	"	"
Paul C. Murray	- 130	"	"

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2. Phase shifters

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III. Contract DA 36-039 sc-87369

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