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# Investigation of Aging Effects From High Voltage Profiles in Ceramic Phase Shifter Materials

S. Stowell, L.C. Sengupta, E. Ngo, M.E. O'Day,  
and R. Lancto

ARL-TR-653

December 1994

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1. AGENCY USE ONLY (Leave blank)		2. REPORT DATE December 1994	3. REPORT TYPE AND DATES COVERED	
4. TITLE AND SUBTITLE Investigation of Aging Effects from High Voltage Profiles in Ceramic Phase Shifter Materials			5. FUNDING NUMBERS	
6. AUTHOR(S) S. Stowell, L.C. Sengupta, E. Ngo, M.E. O'Day and R. Lancto				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Army Research Laboratory Watertown, MA 02172-0001 AMSRL-MA-CA			8. PERFORMING ORGANIZATION REPORT NUMBER  ARL-TR-653	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)			10. SPONSORING/MONITORING AGENCY REPORT NUMBER	
11. SUPPLEMENTARY NOTES				
12a. DISTRIBUTION/AVAILABILITY STATEMENT  Approved for public release; distribution unlimited.			12b. DISTRIBUTION CODE	
13. ABSTRACT (Maximum 200 words)  Ceramic composite materials comprised of $Ba_{0.60}Sr_{0.40}TiO_3$ and Alumina and other non-ferroelectric oxides have been fabricated for use in phased array antennas. These composites have shown superior electronic properties and have been incorporated into several antenna designs. However, minimal testing has been accomplished to determine the electrical aging and fatigue caused by the large electric fields required to operate these devices. In this paper, a systematic study of the effect of the application of large fields will be presented. The electronic properties of the BSTO-Oxide composites (pre-voltage application) will be presented and include the dielectric constant, loss tangent, and tunability (change in dielectric constant with applied field). The study of the change in the electronic properties after large voltages applications include results on the electronic aging and fatigue of the material and appropriate conditioning of the material for application in phased array antenna systems.				
14. SUBJECT TERMS  BSTO, Aging Ceramic Phase Shifters			15. NUMBER OF PAGES 11	
			16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT Unclassified	18. SECURITY CLASSIFICATION OF THIS PAGE Unclassified	19. SECURITY CLASSIFICATION OF ABSTRACT Unclassified	20. LIMITATION OF ABSTRACT UL	

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## INTRODUCTION

Phased array antennas can steer transmitted or received signals either linearly or in two dimensions without mechanically oscillating the antenna. These antennas are currently constructed using ferrite phase shifting elements. Due to the type of circuit requirements necessary to operate these antennas, they are costly, large and heavy. Therefore, the use of these antennas has been limited primarily to military applications which are strategically dependent on such capabilities. In order to make these devices available for many other commercial and military uses, the basic concept of the antenna must be improved. If ferroelectric materials could be used for the phase shifting element instead of ferrites, phased array antennas would be totally revolutionized.

A ceramic Barium Strontium Titanate,  $Ba_{1-x}Sr_xTiO_3$ , (BSTO), phase shifter using a planar microstrip construction has been demonstrated<sup>11</sup>. In order to meet the required performance specifications, maximum phase shifting ability, the electronic properties in the low frequency (KHz) and microwave regions (GHz) must be optimized. As part of this optimization process, various composites of BSTO and non-ferroelectric oxides have been formulated. The BSTO-Alumina composite has a patent on its formulations and the other composites which are designated herein as BSTO-Oxide II and BSTO-Oxide III currently have a patent undergoing the filing process. All of these composites possess improved electronic properties. The comparison of the compositions and phase formation of the various BSTO-oxide ceramic composites will be made and related to their electronic properties. This report will outline some of the initial findings and compare them to the results found for pure BSTO. Finally, the 20 wt% BSTO-Alumina, 20 wt% BSTO-Oxide II, and 20 wt% BSTO-Oxide III specimens have been electronically aged and fatigued at high applied voltages which imitate operational voltage profiles. The results have been analyzed and appropriate conditioning of the specimens has been suggested.

## EXPERIMENTAL

### Ceramic Processing

Powder forms of Barium Titanate and Strontium Titanate were obtained from Ferro Corporation, Transelco Division, Pen Yan, N.Y. (product nos. 219-6 and 218 respectively), stoichiometrically mixed to achieve  $Ba_{0.6}Sr_{0.4}TiO_3$  and ball-milled in ethanol using 3/16" alumina media for 24 hrs. The resulting BSTO was then air-dried, calcined at 1100<sup>0</sup>C and mixed with either powder alumina (ALCOA Industrial Chemicals, Bauxite, AR, distributed by Whittaker, Clark, and Daniels, South Plainfield, N.J., product no. A16-SG) or a second oxide (oxide II) or a third oxide (oxide III) in the proper weight percent and ball-milled again in a slurry of ethanol using the alumina grinding media for an additional 24 hrs.

3 wt% of Rhoplex B-60A (Rohm and Haas Co., Philadelphia, PA) binder is added to the resulting BSTO-Oxide mixture. The mixture is then air-dried and dry-pressed uniaxially to a pressure of approximately 7000 p.s.i.. Sintering schedules were obtained by employing a deflectometer such as Mitutoyo digimatic indicator and miniprocessor (Mitutoyo Corp., Paramus N.J.). It should be noted that all of the examined samples have % liquid absorption of less than 2%.

Two metallization techniques were employed one involved painting on two circular, aligned electrodes, one on either side of the specimens, using high purity silver paint (SPI Supplies West Chester, PA) and attaching wires using high purity silver epoxy, Magnobond 8000, made by Magnolia Plastics, Inc., Chamblee, GA. The other technique utilized the screen printing of electrodes using silver conductive ink (FERRO #3350, Electronic Materials Division, Santa Barbara, CA) and wires were attached by dipping the specimens in a bath of 2% silver, 62% tin and 36% lead solder.

### Electronic Measurements

The dielectric constants,  $\epsilon'$ , loss,  $\tan \delta$ , % tunability were determined for all composites before application of high voltages. The % tunability of a material is determined using the following equation:

$$\% \text{ tunability} = \{ \epsilon'(0) - \epsilon'(V_{app}) \} / \{ \epsilon'(0) \} \quad (1)$$

The tunability measurements were taken with an applied electric field which ranged from 0 to 3.0 V/micron ( $\mu\text{m}$ ). The electronic properties given in the tables were measured at a frequency of 1 KHz. Capacitance measurements for all materials were taken using an HP4284A LCR meter. Further calculations were done to correct for the effect of fringe capacitance.

Further electronic data were acquired after applying large dc fields to the materials. Again, the capacitance measurements for all materials were taken using an HP4284A LCR meter. The temperature during acquisition of both the aging and fatigue data was held constant to within  $\pm 1/2$   $^{\circ}\text{C}$ .

Electronic aging was accomplished by determining the drift in the dielectric constant of the specimen at zero and elevated voltages and the return of the specimen to the zero value dielectric constant was observed. The dielectric constants of the materials were measured at zero bias and then again when a field of  $+1.5$  V/ $\mu\text{m}$  was applied. The field was sustained for 30 minutes and a reading was taken at one minute intervals during this time period. The field was then decreased to zero and the dielectric constant was again recorded.

The fatigue data was accomplished by determining the dielectric constant of the part at zero bias, then again upon the application of  $+1.5$  V/ $\mu\text{m}$  and when the field was decreased to zero again and then the dielectric was recorded at a bias of  $-1.5$  V/ $\mu\text{m}$  and again as field was decreased to zero. The above consisted of one cycle and the specimens were subjected to at least two and a half to five cycles.

## RESULTS AND DISCUSSION

### SEM and X-ray Diffraction Analysis

SEM examination of the individual bulk ceramic layers of the BSTO-Alumina composite revealed that a second phase became apparent in alumina additions as low as 10 wt%. The phase became

more dominant at 35 wt% alumina and then disappeared at the 60 wt% alumina content. Likewise, the x-ray results revealed that when alumina is added to BSTO in small amounts, < 20 wt%, a second phase of Barium Aluminum Titanate,  $Ba_3Al_{10}TiO_{20}$ , is formed. In compositions having between 20 and 40 wt% alumina another second phase of Barium Aluminum Titanate forms, ie.  $BaAl_6TiO_{12}$ . In this composition range we are also seeing traces of Barium Aluminum Oxide. At 60 wt% and 80 wt% alumina we were unable to discern which phase was most prevalent or even if all three phases were present and at 80 wt% alumina definite  $Al_2O_3$  peaks became apparent.

SEM examination of the individual BSTO-Oxide II and BSTO-Oxide III compositions showed very little microstructural difference with added percentages of oxide. The x-ray results for the BSTO-Oxide II composites reveal that initially oxide II appears to be absorbed into the BSTO lattice structure. At compositions from 5-50 wt% oxide II, BSTO is the more dominant pattern, but at 60 wt%, oxide II becomes predominant. It is also interesting to note that no second phase materials were detected at any of the compositions for this composite. Similar results were observed for the BSTO-Oxide III composites. The only variation for this composite is that traces of oxide III are apparent even with only a 1 wt% addition.

### Electronic Properties

The results for the electronic properties of the BSTO-Alumina composites are shown in Table 1. The dielectric constant of the specimens is quickly reduced for compositions up to 35 wt% alumina at which point the rate of reduction in the dielectric constant is diminished. The dielectric loss,  $\tan \delta$ , reported here for all specimens includes the loss caused by the metal contact, improved metallization for these materials will definitely result in loss  $\tan \delta < 0.01$ . The tunability of the composites is maintained at reasonable levels (>10%) up to 20 wt% alumina at which time the tunability decreases rapidly. Table 2 contains the electronic data for the BSTO-Oxide II ceramic composites. As shown in Table 2 the loss tangents of the materials

TABLE 1. Electronic Properties of BSTO-Alumina Ceramic Composites.

<u>Alumina</u> <u>Content (wt%)</u>	<u>Dielectric</u> <u>Constant</u>	<u>Loss</u> <u>Tangent</u>	<u>% Tunability</u>	<u>Electric</u> <u>Field (V/um)</u>
0.0	3299.08	0.0195	19.91	0.73
1.0	2606.97	0.0122	22.50	0.76
5.0	1260.53	0.0630*	13.88	0.67
10.0	426.74	0.0163	4.79	0.39
15.0	269.25	0.0145	5.72	0.87
20.0	186.01	0.0181	3.58	0.48
25.0	83.07	0.0120		
30.0	53.43	0.0135	5.13	2.21
35.0	27.74	0.0029	0.51	0.83
40.0	25.62	0.1616*		
60.0	16.58	0.0009	0.01	0.60
80.0	12.70	0.0016		
100.0	8.37	0.0036		

\* samples had poor contacts

TABLE 2. Electronic Properties of BSTO-Oxide II Ceramic Composites.

<i>Oxide II</i> <i>Content (wt%)</i>	<i>Dielectric</i> <i>Constant</i>	<i>Loss</i> <i>Tangent</i>	<i>% Tunability</i>	<i>Electric</i> <i>Field (V/μm)</i>
0.0	3299.08	0.0195	19.91	0.73
1.0	2696.77	0.0042	46.01	2.72
5.0	2047.00	0.0138	12.70	0.76
10.0	1166.93	0.0111	7.68	0.68
15.0	413.05	0.0159	5.07	1.11
20.0	399.39	0.0152	5.39	0.76
25.0	273.96	0.0240	6.02	1.02
30.0	233.47	0.0098	1.21	0.73
35.0	183.33	0.0091	5.87	0.95
40.0	162.26	0.0095	0.70	0.71
50.0	92.73	0.0071	1.67	1.12
60.0	69.80	0.0098		
80.0	17.31	0.0056		
100.0	15.98	0.0018	0.05	0.27

are relatively low (<0.02). It appears that as the dielectric constant decreased the loss was lowered. The dielectric constant of the composites decreases with the addition of oxide II. The electronic data for the BSTO-Oxide III composites are shown in Table 3. The semi-log plot of the dielectric constants of the BSTO-Alumina composites, the BSTO-Oxide II composites and the BSTO-Oxide III composites is shown in Fig. 1. As shown in Fig. 1, the rate of reduction is similar for both composites for compositions < 20 wt%. However, between 20 wt% - 50 wt% the rate of reduction in the dielectric constant is less than that of the BSTO-Alumina composites. The decrease in the dielectric constant for the two sets of composites is again similar from 60 wt%-100 wt% additive content. However, the magnitude of the dielectric constant for all of the BSTO-Alumina composites is less than that of the BSTO-Oxide II and BSTO-Oxide III composites. This may be due to the formation of the second phases in the BSTO-Alumina composites. As shown in Fig. 2, the loss tangent of the BSTO-Oxide III composites are extremely low for most all compositions (decreases slightly with an increase in oxide III). These formulations could therefore be used at higher operating frequencies, i.e., at millimeter wave range, @ 77 GHz. As shown in Fig. 1, the other electronic properties are similar to BSTO-Oxide II, except for the fact that the dielectric constants are even higher for these composites in the range 15-60 wt%. The tunability decreases slowly with increase in oxide III content and the composites exhibit high tunabilities (>10%) up to 60 wt% oxide III which was not the case for the other oxides.

The results for the aging of the composites are shown in Fig. 3. The graph shows the normalized (change in the dielectric constant) versus time in minutes. As stated previously the specimens were held at zero bias and at 1.5 V/μm for thirty minutes. The percentage drift (or change) in the dielectric constants at these voltages has been determined. The data indicates that the BSTO-Alumina (20 wt%) had a 0.727% change in the dielectric constant before bias and showed a 1.102% drift at bias and a 0.267% drift when again returned to zero. Likewise, the BSTO-Oxide II (20 wt%) specimen showed 2.280% drift before biasing, at bias showed a 0.373 % drift, and



TABLE 3. Electronic Properties of BSTO-Oxide III Ceramic Composites.

<i>Oxide III Content (wt %)</i>	<i>Dielectric Constant</i>	<i>Loss Tangent</i>	<i>% Tunability</i>	<i>Electric Field (V/<math>\mu</math>m)</i>
0.0	3299.08	0.0195	19.91	0.73
1.0	1276.21	0.0015	16.07	2.32
5.0	1770.42	0.0014		
10.0	1509.19	0.0018		
15.0	1146.79	0.0011	7.270	1.91
20.0	1079.21	0.0009	15.95	2.33
25.0	783.17	0.0007	17.46	2.45
30.0	750.93	0.0008	9.353	1.62
35.0	532.49	0.0006	18.00	2.07
40.0	416.40	0.0009	19.81	2.53
50.0	280.75	0.0117*	9.550	2.14
60.0	117.67	0.0006	11.08	2.70
80.0	17.00	0.0008	0.61	1.72
100.0	13.96	0.0009		

\* samples had poor contacts

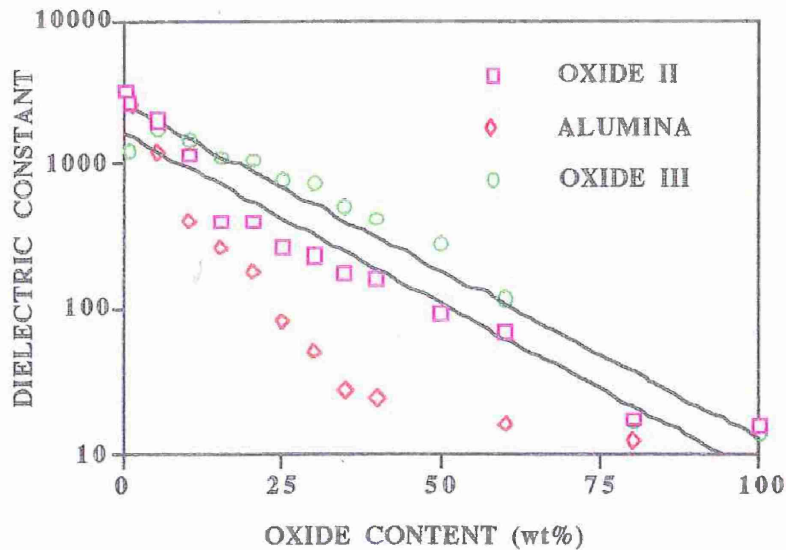


Figure 1. Semi-log plot of the Dielectric Constant versus Oxide Content (wt%) for BSTO-Alumina, BSTO-Oxide II, and BSTO-Oxide III composites.

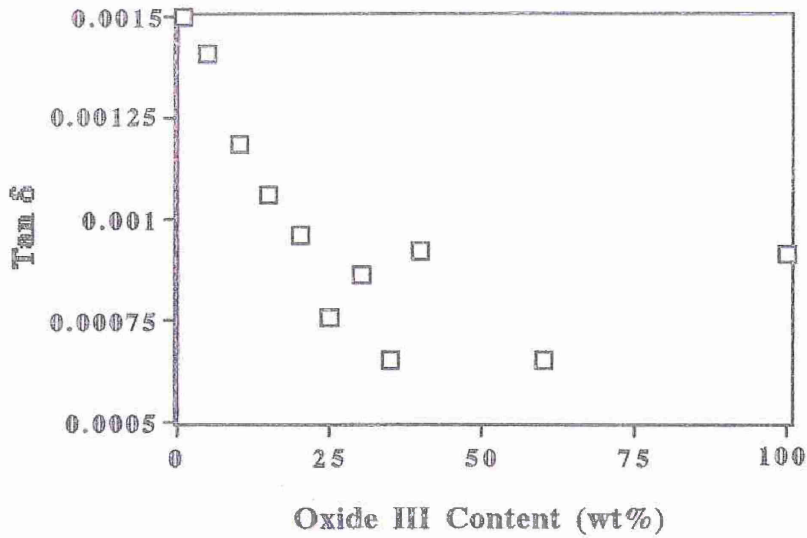


Figure 2. Loss Tangent versus Oxide III Content (wt %) for BSTO-Oxide III Composites.

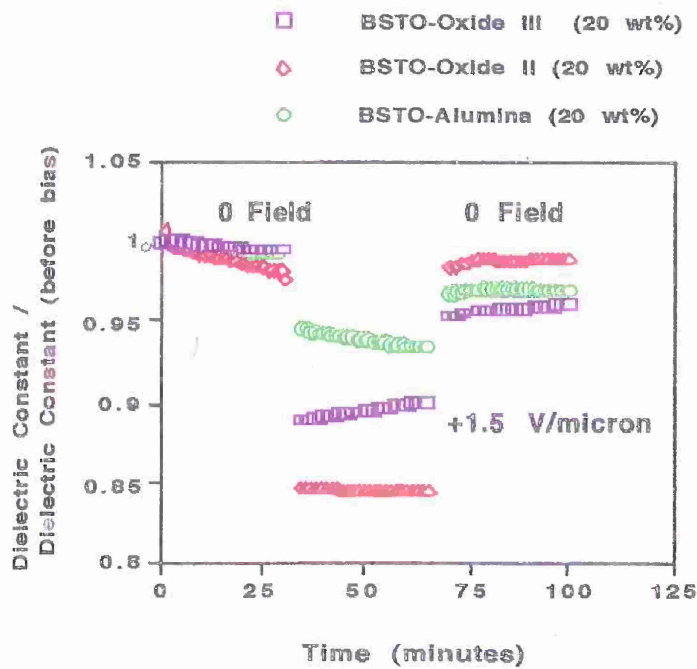


Figure 3. Fractional Dielectric Constant (Dielectric Constant/Dielectric Constant [before bias]) versus Time (minutes) for BSTO-Oxide Composites (20 wt%) measured at 1 KHz.

when returned showed a 0.452% drift. The BSTO-Oxide III sample showed a 0.410% before biasing, a 1.304% drift at bias, and a 0.808 % drift when returned to zero. It is apparent none of the specimens showed significant drifting in their dielectric constants. However, it was noticed that the dielectric constant at zero bias was less when the specimens were returned to zero after biasing. The original value of the dielectric constant of the virgin material was however, regained after the specimen was left sitting for a period of time (the exact recovery time required has not yet been identified). This indicates no remnant polarization existed as expected for paraelectric materials of this type. However, the decreased value of the dielectric constant led us to examine the fatigue experienced by the specimens.

Fig. 4 shows the fatigue experienced by the composites. The tunability of all of the specimens, after the initial biasing cycle, is constant throughout application of the fields, even though the specimens experience typical capacitor-like logarithmic damping of their dielectric constants. For pre-conditioning of the materials, the BSTO-Alumina constants require 5 or more biasing cycles for stabilization of their dielectric constants, whereas the BSTO-Oxide II and BSTO-Oxide III require about 2 to 2 1/2 cycles. Since the dielectric constants of the materials were observed to return to their pre-bias values, the voltage conditioning should be done after each start-up period for the antenna or, if the antenna is left without application of electric field. The time after which the voltage application has to be done has not yet been determined.

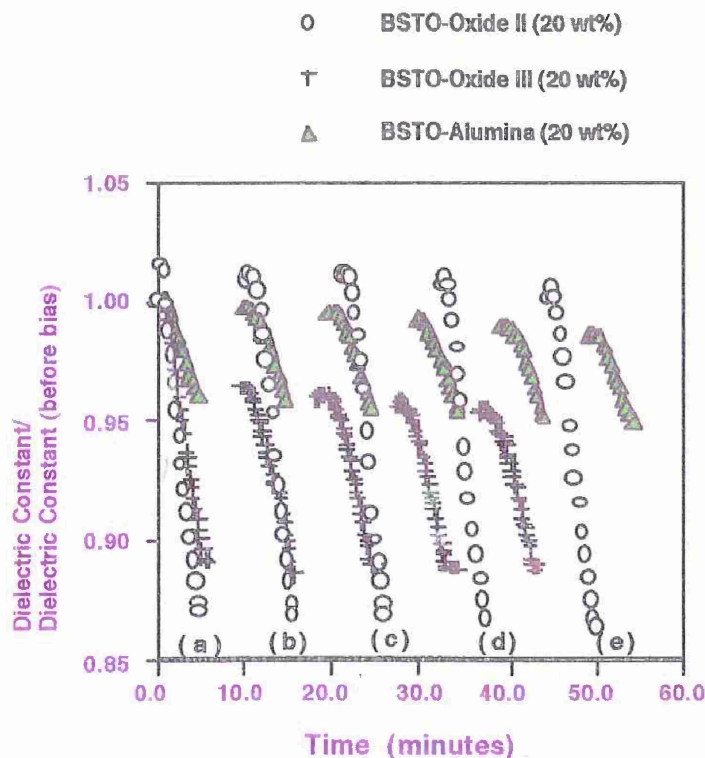


Figure 4. Normalized Dielectric Constants vs. Time for BSTO-Oxide Composites (20 wt%) (a = +1.5 V/ $\mu$ m, b = -1.5 V/ $\mu$ m, c = +1.5 V/ $\mu$ m, d = -1.5 V/ $\mu$ m, and e = +1.5 V/ $\mu$ m).

## CONCLUSIONS

Composites of BSTO and non-ferroelectric oxide ceramics have been fabricated and characterized. The composites have all demonstrated adjustable electronic properties. The dielectric constant of the BSTO-Alumina composites decreases faster than the BSTO-Oxide II and the BSTO-Oxide III composites from 20-50 wt% alumina content and is related to the formation of multiple second phases for the BSTO-Alumina composites. The BSTO-Oxide III composites exhibited the lowest loss tangents and the overall highest dielectric constants. The low loss tangents make higher operating frequencies possible. All composites showed very little drifting of the dielectric constants at zero fields or with an applied bias of 1.5 V/ $\mu\text{m}$ . The samples did exhibit typical capacitor-like damping of their dielectric constants but the before bias value of the dielectric constants of all specimens returned after a period of time (of which has presently not been exactly determined). The tunabilities of all of the specimens did not vary with voltage cycling.

It was noticed that pre-conditioning of the specimens is necessary to achieve a steady-state value for the zero-bias dielectric constants. This was achieved with 2 to 2 1/2 voltage cycles for the BSTO-Oxide II and BSTO-Oxide III specimens and over 5 cycles for the BSTO-Alumina composites.

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