DEVELOPMENT OF CRYOGENICALLY MICROWAVE LOSSY CERAMICS WITH ADJUSTABLE PROPERTIES*

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At cryogenic temperatures (below 20 K), most of the existing lossy materials become non-lossy, requiring the development of a new materials effective in these conditions. Results of an effort to develop a cryogenically lossy materials based on the AlN matrix are presented in the paper. Hot pressing with a wide range of possible lossy second phases was tried, followed by complex permitivitty measurements. A promising second phase was selected, produced and evaluated under cryogenic conditions at the Thomas Jefferson National Accelerator Facility (Jefferson Lab). The developed material system allows the dielectric permitivitty to be varied depending on the application requirements.

1. INTRODUCTION

Absorption of electromagnetic energy in the radio frequency and microwave ranges can take place via several mechanisms[1,2]. For most materials the absorption occurs through semiconducting phenomena, which require thermal excitation of charge carriers for an effective photon/phonon conversion. Whereas most applications that require microwave absorption occur at temperatures close to, or higher than room temperature, some accelerator applications exist[3,4] for which radiation emitted by electron beams must be intercepted and absorbed at temperatures between 2 K and 30 K.

In designing and testing components and systems capable of absorbing microwaves at low temperatures, it is essential that the absorbers (which in accelerator structures must be compatible with the ultra-high vacuum, 10^{-10} torr or lower) have little or no

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variation of the complex dielectric permitivitty between room temperature and the cryogenic temperatures. This can be accomplished through the use of artificial dielectrics[5] (composite materials with a dielectric matrix with an interspersed conductive and lossy phase) in the form of ceramics: this combination allows the use in the vacuum systems of accelerators and at the same time can provide the appropriate absorption of microwave energy.

In this paper we describe the development of artificial dielectric ceramics suitable for use in accelerators and based on Aluminum Nitride ceramic technology.

2. EXPERIMENTAL PROCEDURES

Commercially available powders were used in all tests, with basic properties shown in Table 1. Aluminum nitride (AlN) was used as the matrix in all cases. Second phases were selected based on commercial availability of refractory powders with mainly "metallic" (temperature independent) electrical conductivity, and their additions were limited to about 15 vol % (percolation limit). Based on prior experience[6], C(glassy) powder is known to lead to temperature independent losses, and it was used here as a standard. In addition, thermal conductivity improvements in this material were one of the objectives.

Powders were batched using wet mixing procedures in organic solvents (acetone or isopropyl alcohol), followed by a drying step. In addition to the AlN and a conductive phase, small amounts of sintering and thermal conductivity aids were added to the powder (1-5%). Powders were loaded in lined graphite dies (10x10x1.5 cm or 16 cm diameterx1.5 cm plates), followed by hot pressing at temperatures in the 1700-2000°C range and pressures up to 360 kPa (2,500 psi). In cases of pressureless sintering, a solvent-based binder was added to the powder, the powder was dried and dry-pressed or isopressed in a suitable die. Sintering was performed in a graphite crucible and an induction-heated furnace, under a flowing, inert gas atmosphere.

Table 1 Powder properties

Powder Type	Specific surface area (m^2/g)	Average particle size (µm)	Purity (%)	Oxygen content (%)
AlN (A)	1.1	4	99.5	0.8-1.0
AlN (B)	2.2	2-3	99.5	1.1-1.5
B4C	3-4	2-3	99+	.4
C(glassy)	2-3	4-6	99.8	-
CrN	0.8	4	99+	0.3
Mo_2C	2.3	1.3	99+	-
Mo2Si	0.7	4-5	99+	0.3
TiC	2.7-3.7	1-3	99-99.5	.3
Ti(.3C.7N)	1.7	2	99+	0.3
Ti(.5C.5N)	2.1	2	"	0.3
Ti(.7C.3N)	2.6	2	"	0.3
TiB2	2.9	2	99.5	-
TiN	1-3	2-5	99.5	0.4-0.8
ZrB2	0.8-1.5	2-4	99.2	0.4
ZrC	1.0-1.6	3-4	99	0.5
WC	1.2	1.2	99+	0.2

Dielectric properties of densified materials, after basic density testing, were screened using HP85070C dielectric probe and HP8720ES vector network analyzer in the 1-20 GHz range. Materials showing sufficient loss characteristics were further explored and characterized. After adjusting the material properties of the most promising materials, sufficient amount of material was produced to manufacture samples in the configuration required for cryogenic testing at Jefferson Lab, where the cryogenic loss and brazeability of the materials were evaluated. Some samples were also evaluated at DESY for the Tesla project at a range of microwave frequencies (up to 40 GHz). In addition to dielectric property evaluation under cryogenic conditions, elevated temperature dielectric properties of some materials were measured up to 600°C[†].

3. RESULTS AND DISCUSSION

From the powders listed in Table 1, only two showed sufficient loss tangent (tg $\delta>0.05$ @2GHz) at high loadings to be considered further – ZrC and C(glassy). Based

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[†] Measurements performed by Microwave Properties North, Ontario, Canada

on this, a range of compositions with ZrC was produced under different processing conditions. Figure 1 shows effects of the peak hot-pressing temperature on the dielectric properties of the material (HP probe measurements), measured at 2GHz, as well as the ability to dial in the dielectric properties by adjusting the material composition. Thermal conductivities of the composites (Figure 2) were relatively high, and decreased with the second phase loading, as expected. Properties of optimized AlN-C(glassy) composite for the JNAF accelerator cryogenic application in comparison to prior version are given in Table 2.

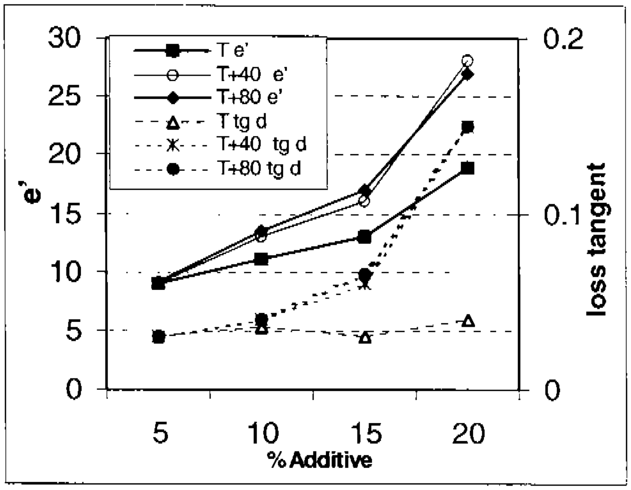


FIGURE 1 Dielectric properties (ambient) of AlN-ZrC composites, processed at three temperatures.

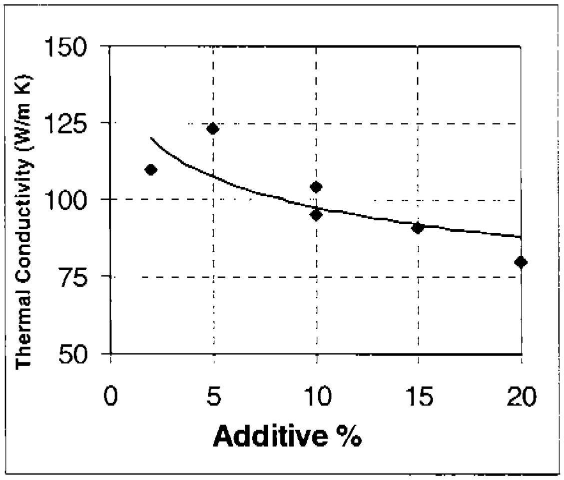


FIGURE 2 Thermal conductivity of AlN-ZrC lossy composites

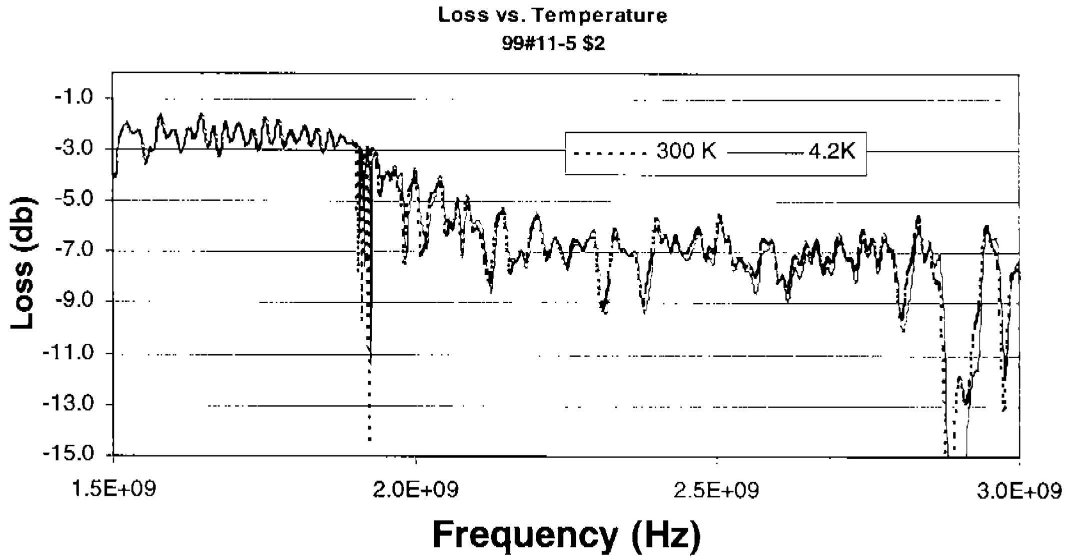
Table 2 Properties of optimized AlN-C(glassy) lossy composite

Property	Optimized composite	1995 version
e' @2(GHz)	20-25	20-25
tgδ	.13-.18	.13-.18
Thermal conductivity (W/m K)	85-130	50-55

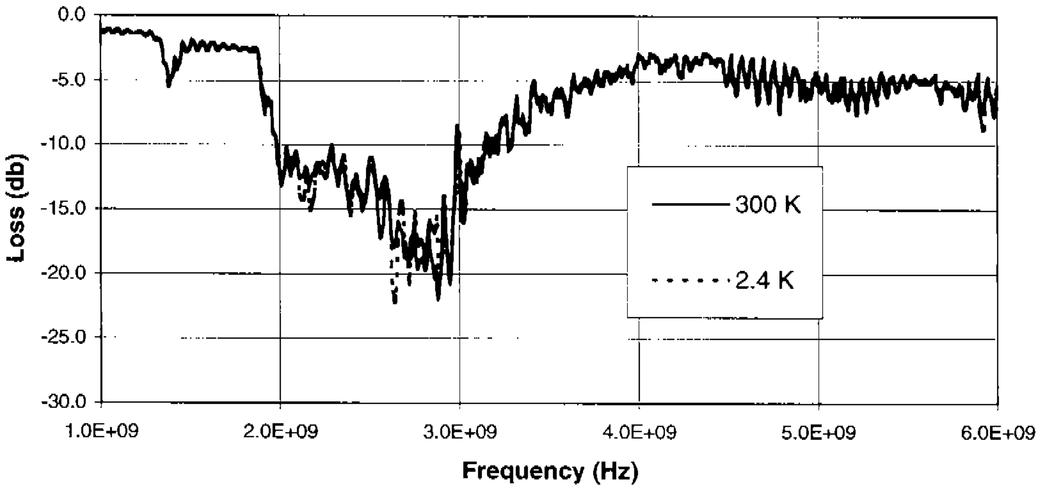
To verify the cryogenic loss of both types of lossy composites, enough material was produced from the most suitable composites and absorbers were made from them for outgassing tests performed at Jefferson Lab, as well as cryogenic testing in a wave guide (Figure 3) after bonding (Figure 4). Loss in Figure 3a is lower than for the material in Figure 4b, a result of higher loss tangent of the AlN-C(glassy) material.

Outgassing tests showed that all samples could be outgassed in the wave guide to 10^{-9} torr in the same amount of time as without them.

This demonstrated a complete functionality of both materials in cryogenic environments under vacuum as well as bondability to Cu.



a. AlN-ZrC lossy



b. AlN-C(glassy)

FIGURE 3 Waveguide insertion loss at ambient and cryogenic temperatures with two different lossy materials with no change in loss detected. Testing performed on brazed samples. (Note difference in frequency range)

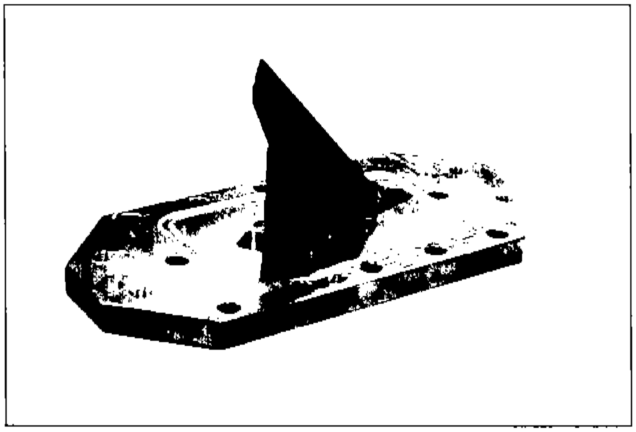
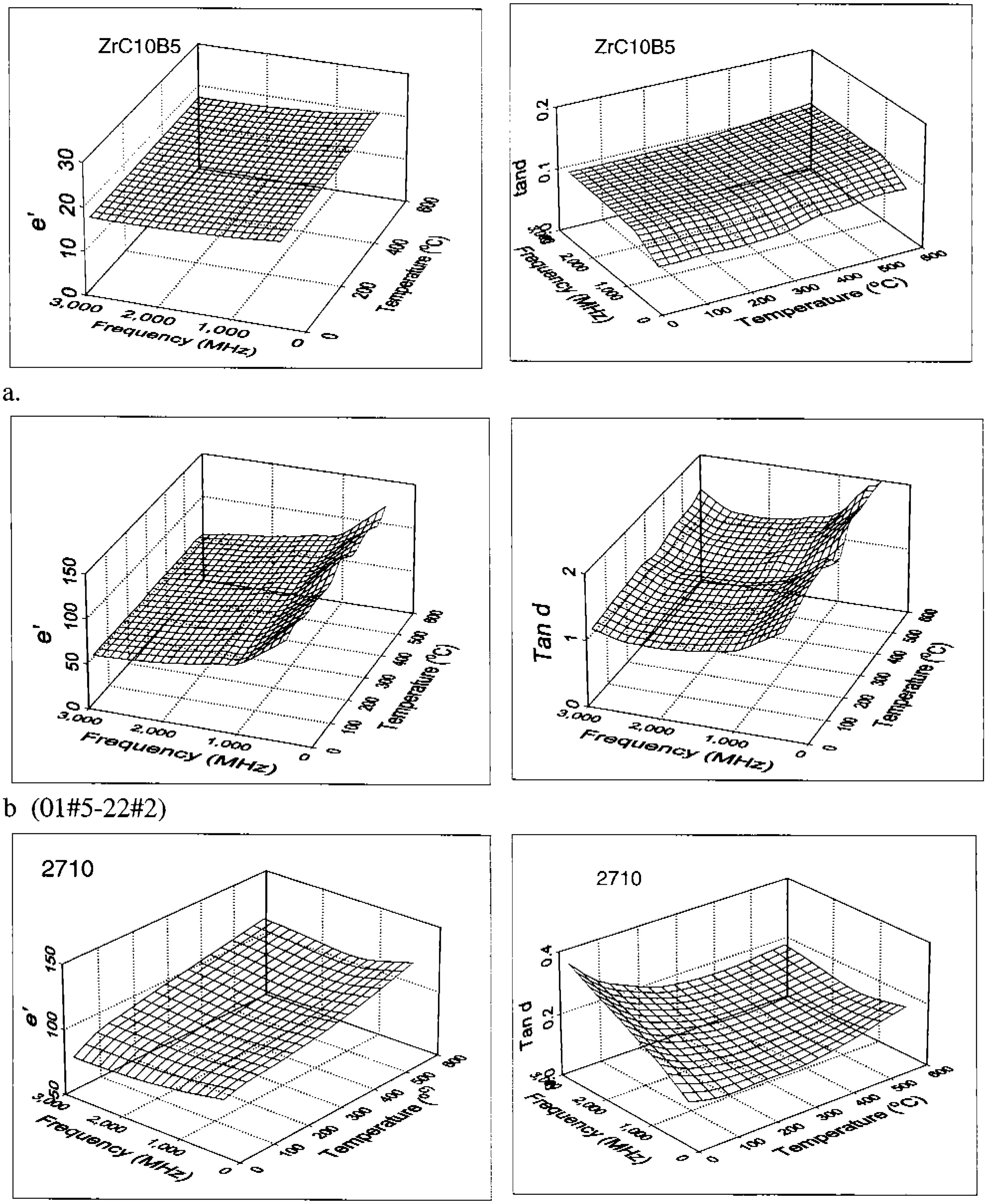


FIGURE 4 Brazed lossy ceramic segments to Cu waveguide segment after the cryogenic test.

To demonstrate the same materials operate just as well at elevated temperatures (which is the case for lossy components in high power electron devices), high temperature dielectric properties were measured on some of the developed materials, and compared to a BeO-SiC lossy material commonly used in high power devices. An example is shown in Figure 5, where a moderately lossy AlN-ZrC and high loss AlN-C(glassy) are compared to a high loss BeO-40%SiC lossy material. The important point here is that the dielectric properties of the new materials are virtually independent of temperature to at least 600°C, whereas the SiC based lossy materials show a considerable temperature dependence.

Figure 6 presents averaged results of a series of waveguide complex permitivitty measurements in four frequency bands (3-40GHz) on three of the developed materials. This demonstrates the range of frequencies of materials' effectiveness. For the Tesla accelerator, material Zr10B5 has been selected. Delivered components from this material have been brazed and are waiting for further testing.



a.

b (01#5-22#2)

c

FIGURE 5 Temperature dependence of e' and loss tangent for a- AlN-ZrC; b- AlN-C(glassy) and c- AlN-SiC lossy material.

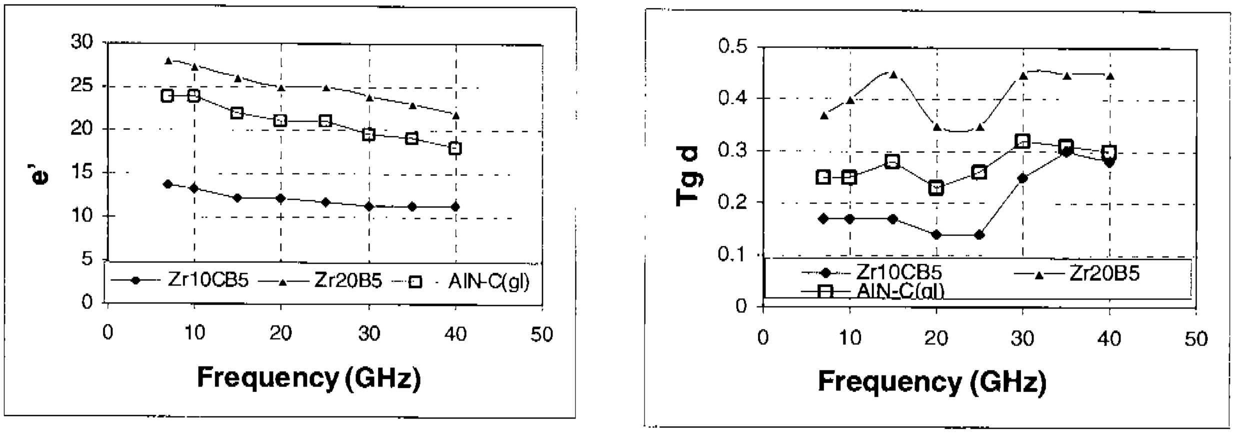


FIGURE 6 Complex permitivitty of selected materials. (by Andreas Joestingmeier)

4. CONCLUSIONS

Two families of AlN based materials have been developed, both with virtually invariant dielectric loss from 2 to 900 K. These materials are vacuum compatible, can be brazed to Cu, have selectable dielectric properties and have higher room temperature thermal conductivities than comparable AlN based lossy materials.

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