

LOSS TANGENT MEASUREMENTS ON UNIRRADIATED ALUMINA – S. J. Zinkle and R. H. Goulding (Oak Ridge National Laboratory)

OBJECTIVE

The objective of this report is to summarize some of the available unirradiated room temperature data on the loss tangents of several commercial grades of alumina.

SUMMARY

Unirradiated room temperature loss tangent data for sapphire and several commercial grades of polycrystalline alumina are compiled for frequencies between 10^5 and 4×10^{11} Hz. Sapphire exhibits significantly lower values for the loss tangent at frequencies up to 10^{11} Hz. The loss tangents of 3 different grades of Wesgo alumina (AL300, AL995, AL998) and 2 different grades of Coors alumina (AD94, AD995) have typical values near $\sim 10^{-4}$ at a frequency of 10^8 Hz. On the other hand, the loss tangent of Vitox alumina exhibits a large loss peak ($\tan \delta \sim 5 \times 10^{-3}$) at this frequency.

PROGRESS AND STATUS

Introduction

Ceramic insulators are required for radiofrequency (RF) heating components in magnetic fusion energy systems. The insulator is required to transmit megawatts of RF beam power and therefore must have low amounts of dielectric power loss (i.e., low loss tangent) in addition to good thermal conductivity and mechanical strength [1-4]. In order to avoid overheating of the insulator, the loss tangent must be less than $\sim 10^{-3}$ for ion cyclotron range of frequencies (ICRF, ~ 50 -100 MHz) and less than $\sim 10^{-6}$ for electron cyclotron range of frequencies (ECRF, ~ 100 GHz). Alumina has been identified as one of the leading candidates for near-term RF systems. A large amount of work has been performed over the past few decades to measure the dielectric properties of alumina in the unirradiated [2-10] and irradiated [2,3,10-12] conditions. Most of the published neutron irradiation results on the loss tangent of alumina are summarized in ref. 13. The present report summarizes some of the published information on the room temperature dielectric loss tangent of unirradiated single crystal and polycrystal alumina at frequencies between 10^5 and 4×10^{11} Hz. This compilation is not intended to be an exhaustive summary of all existing data, but is instead a representative listing of typical results.

Results and Discussion

The room temperature loss tangents of 5 different commercial grades of polycrystalline alumina produced by GTE Wesgo and Coors Ceramics were measured at a frequency near 100 MHz as part of a larger program investigating the effects of gamma ray and pulsed neutron irradiation on prompt dielectric properties of ceramic insulators [14,15]. Some of the unirradiated values were not published in previous reports and are therefore summarized here for completeness. In order to provide a more comprehensive data set, room temperature loss tangent measurements on sapphire [4], Vitox (polycrystalline 99.9% Al_2O_3) [4,11], Wesgo AL995 [8,9], Wesgo AL300, and Coors AD995 [7,9] at frequencies between 10^5 and 4×10^{11} Hz have been compiled from published reports. Details concerning the measurement techniques may be found in the original reports. Some limited data quoted by the manufacturers [16,17] are included for purposes of comparison.

Table 1 summarizes the measured chemical composition of the Wesgo, Coors and Vitox grades of alumina [4,19-21]. It should be noted that the chemical analyses were performed on different heats of material compared to the dielectric measurements. However, heat-to-heat variations in the chemical composition of

Table 1. Chemical analysis (wt. %) for some of the commercial polycrystalline grades of alumina. Chemical analysis performed by Coors Analytical Laboratory [19] unless otherwise noted. Representative uncertainties in the impurity measurements quoted by the analysis laboratories are noted. The alumina concentration was obtained by subtraction of the measured impurity content.

Element	Wesgo AL995	Wesgo AL995 [20]	Wesgo AL300	Coors AD94	Coors AD998	Vitox [4,21]
Al ₂ O ₃	99.3	99.5	97.3	93.4	99.72	99.9
Si	0.22±0.02	0.137±0.006	1.4 ± 0.1	4.0 ± 0.4	0.085±0.009	<0.01
Fe	0.076	0.034	0.081	0.18	0.040	0.0027
Ca	0.095	0.0424	1.1 ± 0.1	0.26	0.042	0.0054
Mg	0.31±0.03	0.249±0.005	0.033	0.68	0.077±0.008	0.038
Zr	0.008	0.007	0.009	0.65	0.006	0.00033
Ba	<0.005	<0.0002	<0.005	0.76	<0.005	
Na	0.027	0.021	0.039	0.045	0.028	0.0013
Ti	0.005	0.01	0.003	0.02	0.003	
K	0.007	<0.002	0.007	0.06	0.004	
Co		<0.005				
Cr		<0.001				<0.00005
Ni		<0.002				
Cu						0.00065
Ga						0.0030
Sr						0.0004
Zn						0.0018

these well-established commercial ceramics are thought to be less than the uncertainty in the chemical measurements. The chemical compositions for two of the materials measured at a frequency of 100 MHz (Coors AD995 and Wesgo AL998) are not yet available, and will be reported in a future progress report. Two independent chemical analyses have been performed on the U.S. fusion heat [18] of Wesgo AL995, which provides some indication of the absolute accuracy of the chemical measurements.

The results of the loss tangent measurements are summarized in Figures 1 and 2. Figure 1 compares the loss tangent for sapphire [4], Vitox [4,11] and Wesgo AL995 [8,9,15,17]. Sapphire exhibits very low values of loss tangent ($<10^{-5}$) for frequencies up to $\sim 10^{10}$ Hz. The intrinsic loss peak in alumina occurs at a frequency of $\sim 10^{13}$ Hz [22], which causes the loss tangent of sapphire to increase rapidly with frequency above 10^{10} Hz. The loss tangent of Wesgo AL995 is approximately an order of magnitude larger than that of sapphire over a wide frequency range. The Wesgo AL995 loss tangent converges toward the sapphire value at frequencies above $\sim 10^{11}$ Hz, due to the intrinsic alumina loss peak at high frequencies. The dielectric loss tangent of Vitox is characterized by a very pronounced loss peak at frequencies near 10^8 - 10^9 Hz.

A further comparison of the loss tangents of the different Coors and Wesgo grades of alumina is shown in Fig. 2, where the low-loss and high-loss curves associated with sapphire and Vitox, respectively have been omitted for clarity. All five of the Coors and Wesgo grades of alumina have room temperature loss tangent values near 10^{-4} at a frequency of 100 MHz. With the exception of the higher loss tangent for AD995 quoted in the Coors technical literature [16], very good agreement is found between the loss tangent values reported in different studies on a particular grade of alumina. This indicates that heat-to-heat variations in the dielectric properties of these commercial grades of alumina are small.

All five of the Coors and Wesgo grades of polycrystalline alumina appear to be suitable for potential application in ICRF heating systems, since their unirradiated room temperature loss tangents are well below 10^{-3} . Previous work has found that the unirradiated loss tangent of low-loss grades of alumina in the 1 MHz-10 GHz range of frequencies is relatively insensitive to temperature between 20 and $\sim 300^\circ\text{C}$ [4-6].

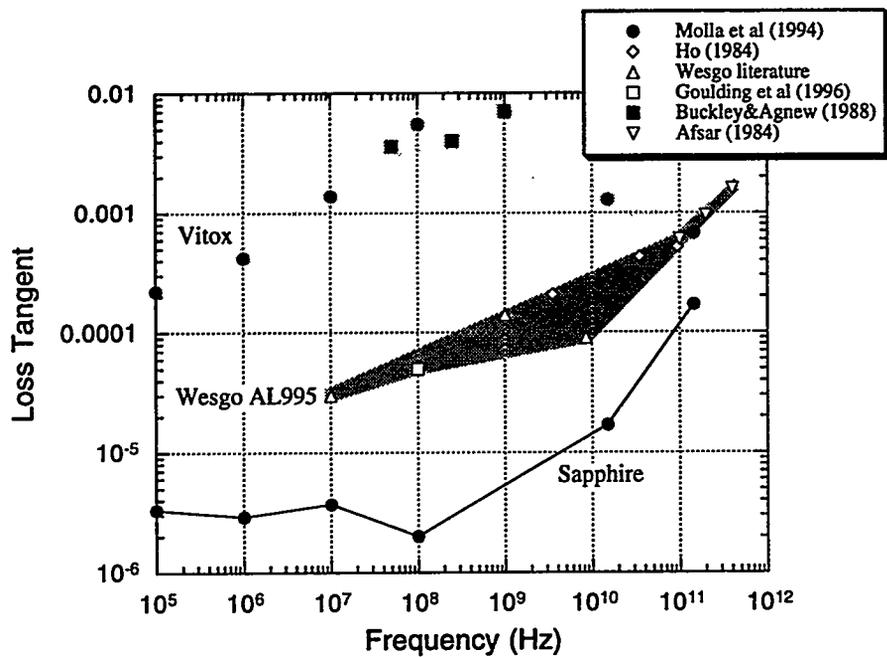


Fig. 1. Room temperature loss tangent vs. frequency for single crystal alumina [4] and two commercial grades of polycrystalline alumina [4,8,9,12,15,17].

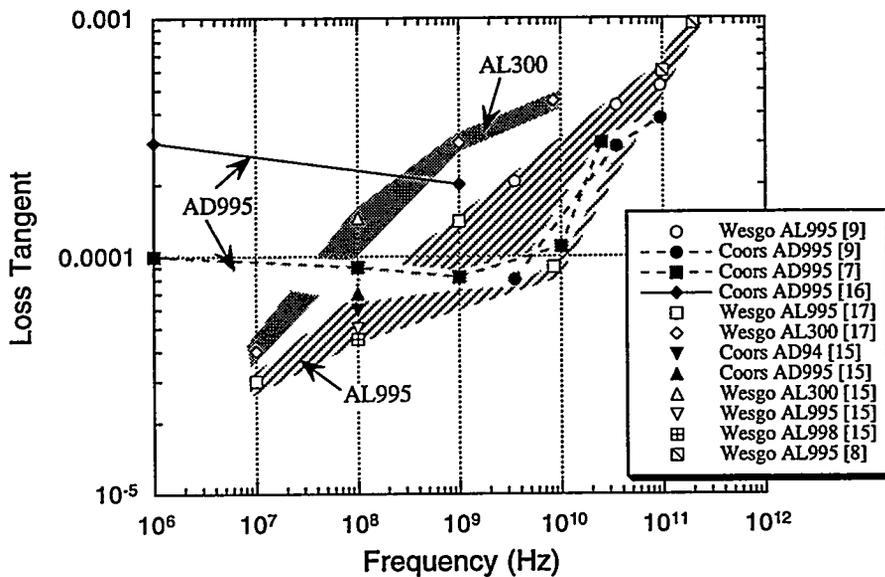


Fig. 2. Room temperature loss tangent vs. frequency for Wesgo AL300, AL995, AL998 [8,9,15,17] and Coors AD94, AD995 [7,9,15,16] grades of polycrystalline alumina.

Neutron irradiation damage would increase the loss tangent. Extrapolation of the available loss tangent data on alumina irradiated with fission neutrons near room temperature [13] suggests that the loss tangent would approach 10^{-3} after a neutron fluence of $\sim 10^{25}$ n/m² ($E > 0.1$ MeV), which corresponds to a damage level of ~ 1 dpa.

The cause of the large loss peak at ~ 100 - 1000 MHz in the Vitox grade of polycrystalline alumina is uncertain. This loss peak does not occur in any of the five Wesgo and Coors grades of alumina investigated in this report. The major impurity in Vitox is 380 ppm Mg. However, both Coors AD998 and Wesgo AL300 grades of alumina have similar Mg levels (Table 1) without the corresponding large loss peak near 100 - 1000 MHz. Therefore, the loss cannot be strictly associated with a specific concentration of Mg. Previous work has found that impurities such as Mg and Si can have a significant effect on the loss tangent at low frequencies (≤ 1 MHz), but do not have a noticeable effect at ~ 9 GHz [5,6]. The low-frequency losses were shown to be associated with increased electrical conductivity in the impurity-doped specimens. The conductivity contribution to the loss tangent becomes insignificant at frequencies above ~ 10 MHz since it is inversely proportional to frequency. Therefore, this mechanism would not explain the appearance of a loss peak at 100 - 1000 MHz in Vitox.

Ho [9] noted that the dielectric loss tangent at GHz frequencies in sintered alumina did not correlate with impurity concentration. Instead, the location and chemical form of the impurity (which is dependent on the details of the sintering process) was found to be the most important factor. For example, a laboratory-prepared Al₂O₃ -0.2% MgO specimen was found to have a factor of two higher loss tangent at 35 GHz than any of the Coors or Wesgo grades of alumina [9]. The lowest loss tangents were observed in commercial grades of alumina that were sintered for sufficiently long times to eliminate impurities within the grains and to produce relatively pure secondary phases (located at grain boundaries). The presence of large sintering pores at the grain boundaries did not have a deleterious effect on the millimeter-wave losses, whereas fine porosity apparently produced increases in the loss tangent [9]. The grain size in Vitox (~ 1.4 μm) [4] is considerably smaller than in the other commercial grades of alumina (~ 30 μm) investigated in this report. Due to the larger grain boundary surface area per unit volume in fine-grained materials, impurities need to be controlled to much lower concentration levels to achieve the same loss properties as larger-grained materials [9]. Further work is needed to determine if the loss peak at 100 - 1000 MHz in Vitox can be eliminated by modifying the sintering process.

Conclusions

Sapphire and the five Coors and Wesgo grades of polycrystalline alumina are worthy of further consideration for ICRF insulator applications, whereas Vitox exhibits unacceptably large losses in this frequency range.

Future Work

Room temperature dielectric property measurements at 50 and 100 MHz will be performed on various unirradiated and neutron-irradiated ceramic insulators. The postirradiation measurements will be performed on ceramic insulators irradiated in HFIR at 60 - 600°C to damage levels of ~ 1 and ~ 15 dpa (hydraulic rabbit and CTR60/61 target capsules) and in FFTF MOTA 2B at 420 - 800°C to damage levels of ~ 30 to 60 dpa.

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REFERENCES

1. J. L. Scott, F. W. Clinard, Jr., and F. W. Wiffen, *J. Nucl. Mater.* **133&134** (1985) 156.
2. H. M. Frost and F. W. Clinard, Jr., *J. Nucl. Mater.* **155-157** (1988) 315.
3. R. Heidinger, *J. Nucl. Mater.* **179-181** (1991) 64.
4. J. Molla, R. Heidinger and A. Ibarra, *J. Nucl. Mater.* **212-215** (1994) 1029.
5. L. M. Atlas, H. Nagao and H. H. Nakamura, *J. Am. Ceram. Soc.* **45** (1962) 464; **46** (1962) 196.
6. G. S. Perry, *Brit. Ceram Soc. Trans.* **69** (1970) 177.
7. W. H. Gitzen, Ed., *Alumina as a Ceramic Material* (Amer. Cer. Soc., Westerville, OH, 1970).
8. M. N. Afsar, *IEEE Trans. Microwave Theory Tech.* **MTT-32**, No. 12 (1984) 1598.
9. W. W. Ho, Millimeter Wave Dielectric Property Measurement of Gyrotron Window Materials, ORNL/SUB/83-51926/1 (April 1984); ORNL/SUB/83-51926/2 (April 1985).
10. R. Heidinger, *J. Nucl. Mater.* **173** (1990) 243.
11. S. N. Buckley and P. Agnew, *J. Nucl. Mater.* **155-157** (1988) 361.
12. R. Heidinger and A. Hofmann, H-U Nickel and P. Morajitra, *Fus. Eng. Des.* **18** (1988) 337.
13. L. W. Hobbs, F. W. Clinard, Jr., R. C. Ewing, and S. J. Zinkle, *J. Nucl. Mater.* **216** (1994) 291
14. R. E. Stoller, R. H. Goulding, and S. J. Zinkle, *J. Nucl. Mater.* **191-194** (1992) 602; also Fusion Reactor Materials semiann. prog. report for period ending Sept. 30, 1990, DOE/ER/1313/9, p. 317.
15. R. H. Goulding, S. J. Zinkle, D. A. Rasmussen, and R. E. Stoller, Fusion Reactor Materials semiann. prog. report for period ending Sept. 30, 1993, DOE/ER/1313/15, p. 434; also *J. Appl. Phys.* **79**, No. 5 (1996) in press.
16. Material Properties Chart, Coors Ceramics Co., Structural Division, Golden, Colorado.
17. Technical bulletin, Wesgo Division, GTE Products Corp., Belmont, California.
18. R. E. Stoller, Fusion Reactor Materials semiann. prog. report for period ending March 31, 1990, DOE/ER/1313/8, p. 299.
19. Coors Analytical Laboratory, Golden, Colorado, Lab. analysis #90-04229 (Dec. 21, 1990).
20. Forschungszentrum Karlsruhe, IMF-I chemical analysis 360/94 (October, 1994).
21. G. P. Pells and M. J. Murphy, *J. Nucl. Mater.* **183** (1991) 137.
22. G. Link and R. Heidinger, in 18th Int. Conf. on Infrared and Millimeter Waves, eds. J. R. Birch and T. J. Parker, *proc. SPIE* vol. 2104 (1993) 150.