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Mechanical reliability of ceramic windows in high frequency microwave heating devices

Part 2 *Mechanical behaviour of the ceramics*

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The mechanical reliability was evaluated for the specific alumina and beryllia ceramics now used as microwave windows in the high-power ($\geqslant 200$ kW) high-frequency ($\geqslant 60$ GHz) gyrotron tubes being developed for plasma heating in fusion systems. Previous analysis of the stresses generated in the various window configurations and tube operating conditions indicated that significant tensile stresses are generated in the ceramic window by dielectric heating. As a result, we characterized both the static fatigue behaviour in the fluorocarbon fluid used to cool gyrotron windows and the inert strength distributions for these two ceramics. These data were then analysed in order to construct reliability diagrams for these two materials. Such diagrams revealed that the use of these specific ceramic materials will be limited by their time to failure at the tensile stresses imposed on them under the gyrotron operating conditions (60 GHz or greater and $\geqslant 200$ kW in a continuous wave (CW), TE_{02} mode (i.e., radial power distribution in beam exhibits two maxima). The fatigue behaviour and inert strengths of both materials could be improved by increasing their density (> 97% of theoretical) and employing a uniform fine ($\leqslant 10\,\mu$m) grain size. Such material improvements would permit significant increases in the mechanical reliability of the gyrotron microwave window.

1. Introduction

The development of high power microwave devices has been greatly accelerated by the interest in the use of microwave energy (i.e. 100 kW to 10 MW) at frequencies of up to a few hundred gigahertz for plasma heating in fusion systems [1]. Microwave tubes such as the gyrotron present major challenges not only in their theory and design but also in their fabrication. In addition, the high output powers and frequencies impose stringent requirements on the ceramic window materials utilized. The primary requirement is that of extremely low energy absorption to minimize window heating. This need for high "optical" quality in gyrotron window materials is reminiscent of similar requirements for high energy laser window materials [2, 3]. While recognizing the differences in absorption processes involved in materials in the different frequency regimes of interest in gyrotrons and laser windows, one is encouraged by the successes achieved in developing materials with both sufficient strength and very low and nearly intrinsic absorption properties for the laser applications. The initial achievements in high power gyrotrons and klystrons using beryllia (BeO), alumina (α-Al_2O_3), and sapphire (single crystal α-Al_2O_3) windows [4–6] are indicative of the possibilities for successful application of these materials in high energy microwave applications.

The selection of materials for any window application must include consideration of their ability to reliably withstand the imposed mechanical stresses (i.e. thermal stresses from microwave heating or structural stresses from system pressure differentials). This consideration arises because the materials that meet the stringent optical or

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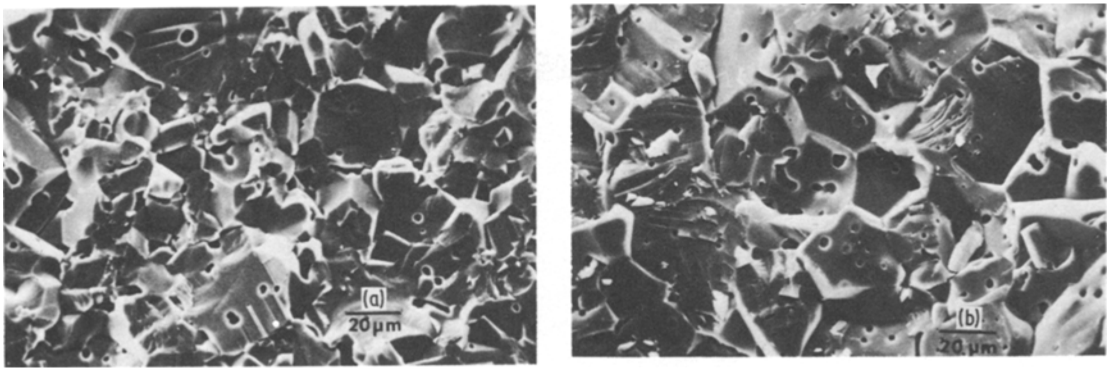


Figure 1 Fracture surfaces of alumina and beryllia ceramics currently used for microwave windows in gyrotron tubes. (a) A bimodal grain size is evident in the alumina, in which regions containing 5 to 8 μm grains are distributed in a matrix of larger (~ 30 μm average) grains. (b) A uniform grain size averaging about 35 μm is observed in the beryllia. Both materials exhibit 1 to 3 μm diameter pores, which are distributed inter- and intragranularly.

electrical property requirements for microwave (and laser) windows are usually brittle and therefore fail catastrophically in short times, or over a period of time at rather low applied tensile (or bending) stresses without any plastic deformation. Initial gyrotron developments illustrate that the present windows can survive for considerable operating times under the thermal stresses developed. However, the increased power levels and frequencies required for the final tube designs will increase the thermal tensile stresses generated in the windows [7]. Window failure can result if the brittle mechanical behaviour of such materials is not thoroughly understood and included in a fracture mechanics design criterion. Such an approach has been successfully applied for laser [8] and Skylab [9] windows.

2. Experimental approach

The materials examined included samples obtained from a single isostatically pressed and sintered billet of Thermalox 995 standard BeO* and three discs of pressed and sintered AL 995 Al_2O_3.† The BeO ceramic had an average density of 2.904 g cm^{-3} (96.5% theoretical), faceted pores (1 to 3 μm diameter) both within grains and along grain boundaries, and an average grain size of about 35 μm. The Al_2O_3 ceramic had an average density of 3.84 g cm^{-3} (96.4% theoretical) with about 2 μm average inter- and intragranular pores distributed in a microstructure consisting of regions of 5 to 8 μm grains in a 30 μm grain size matrix. Typical microstructures for the two materials investigated are illustrated in Fig. 1.

Fracture strength and static fatigue data were obtained by employing four-point flexure tests for test span ratios (i.e. of distances between inner and outer load points) of 19.05:31.75 mm and 6.35:19.05 mm. The flexure test bars had cross-sectional dimensions of 2.54 by 2.79 mm and with edges chamfered at 45° (chamfer ~ 0.06 mm in width). The tensile surfaces consisted of 500-grit diamond-ground surfaces (grinding direction parallel to tensile axis of bar) for the BeO specimens and as-fired surfaces for the Al_2O_3 specimens. Test environments included liquid nitrogen and FC-75 fluorocarbon fluid‡ (22° C) for fracture strengths and FC-75 fluid held at both 22 and 48° C for static fatigue tests. The solubility limit for water of about 7 ppm in the fluorocarbon fluid was confirmed analytically.

3. Results and discussion

In order to establish the long-term mechanical reliability of these specific ceramics under the service stresses predicted by the stress analysis study [7], we established both the inert strength distributions and static fatigue characteristics for each material as discussed below. These were then employed to construct fatigue (time-to-failure

*A product of Brush-Wellman Corp., Elmore, Ohio. Test samples prepared by Ceradyne Corp., Santa Ana, California, USA.

†A product of Wesgo, Bellevue, California, USA.

‡A product of 3M Corporation, St. Paul, Minnesota, USA.

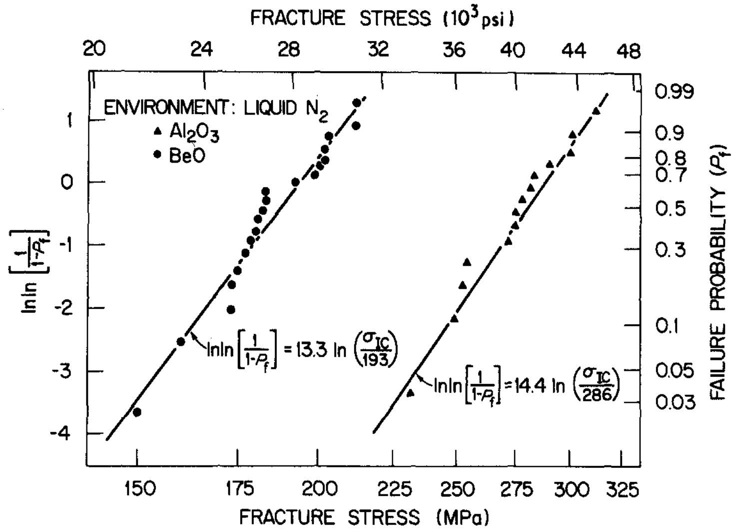


Figure 2 Distribution of four-point flexure strength under conditions in which slow crack growth is eliminated. This distribution shows the effect of the statistical nature of critical flaw parameters.

against applied stress) reliability diagrams for various probability of failure levels. The calculated service stress levels, based on the gyrotron tube operating conditions and pertinent properties of the ceramic [7], were then used to determine the life of the window material. Note that the service stresses utilized here are for specific window design and size and gyrotron operating conditions currently employed. As pointed out by the study on the service stresses, increasing the diameter of the window will decrease the tensile stresses generated in the window via dielectric heating. On the other hand, increasing either the frequency or the power level of the microwave beam will increase the tensile stress levels [7].

3.1. Inert fracture strength distributions

To obtain the statistical parameters required to describe the variability of fracture strength of these brittle materials due solely to the variability of critical flaw-size populations, flexure strengths were measured in a liquid nitrogen bath to avoid slow crack growth effects during loading. The results of these tests are shown in Fig. 2 as a plot that describes the probability that a specimen will fail when a known stress is applied. The data points, which represent the distribution of strengths obtained in four-point flexure, are then fitted by a linear regression analysis to

$$\ln \ln 1/(1-P_f) = m \ln (\sigma_{IC}/\sigma_0), \qquad (1)$$

where P_f is the probability of failure, m is the Weibull modulus reflecting the breadth of the flaw size range, σ_{IC} is the inert strength, and σ_0 is a constant. A large value of m reflects a relatively narrow flaw size range and thus a low variability in strength.

3.2. Static fatigue behaviour

Static fatigue tests were conducted in four-point flexure in a bath containing the fluorocarbon fluid maintained at 22 or 48° C. The time required for the flexure bar to fail upon application of a known stress level is then used to construct the fatigue plots (Fig. 3). The data at 22° C for the Al_2O_3 ceramic exhibit considerably more scatter than do the BeO data (Fig. 3), which undoubtedly reflect the less uniform, bimodal microstructure of the Al_2O_3 ceramic.

The BeO data at 22° C give the first firm evidence of a fatigue limit in a polycrystalline ceramic. At applied stresses of less than 40% of the mean inert strength of the BeO, no fatigue (slow crack growth) occurred in the FC-75 fluorocarbon at 22° C. Keep in mind that this does not reflect the statistical nature of failure, but only that the fatigue limit is reached at a probability of failure level of approximately 0.5 (about one out of two samples fail).

On the other hand, the fatigue problem is made more severe by increasing the temperature of the fluorocarbon. The average time to failure at a given applied stress at 48° C is about one tenth that at 22° C, as seen in Fig. 4.

Analysis of the data obtained is possible by linear regression methods except at longer times to failure in the case of BeO, in which nonlinear behaviour occurs. This nonlinear region was

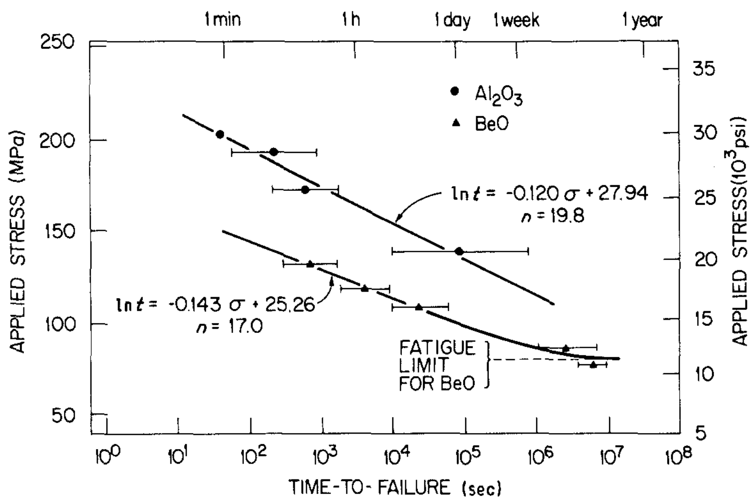


Figure 3 Static fatigue behaviour of ceramics in fluorocarbon fluid (~ 7 ppm H_2O), showing time to failure dependence on applied flexure stress level at 22° C. Note that the BeO data exhibit a fatigue limit. Both curves represent time to failure for a 0.5 probability of failure.

treated by incorporating a fatigue limit and fitting the data by iterative methods. The linear portions of the plot of time to failure, t, against applied stress, σ, data in Fig. 3 were analysed by two different approaches. To derive the stress corrosion susceptibility exponent n and the A parameters used in the lifetime prediction, the following was used:

$$\ln t = -n \ln \sigma + A'. \quad (2)$$

A more general form,

$$\ln t = b_1\sigma + b_0, \quad (3)$$

can be used to describe the stress corrosion process (e.g. activation volume). The terms b_1, b_0, and A' are constants. The values of n, 19.8 and 22.1, respectively, determined by static fatigue (Fig. 3) and by dynamic fatigue [10] are in excellent agreement for the alumina ceramic tested in the fluorocarbon fluid.

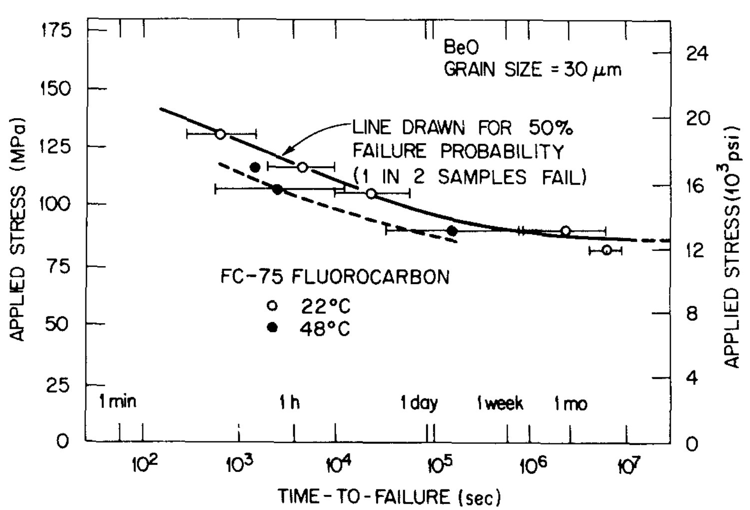
The static fatigue limit in the BeO was described by using the general integral representation of fatigue life:

$$t = (2/\sigma^2 Y^2) \int_{\sigma(K_{IC}/\sigma_{IC})}^{K_{IC}} (K_I/V)\,dK_I, \quad (4)$$

where the crack velocity, V, is determined by iteration processes by

$$V = \Omega_2 \exp(\Omega_3 K_I)\{1 - \exp[-L(K_I - K^*)]\}, \quad (5)$$

where K_I is a crack tip stress intensity factor and is less than K_{IC}, the critical stress intensity factor; Y is a geometric factor; and Ω_2, Ω_3, L, and K^* are constants obtained by iteration; L and K^* characterize the fatigue limit.



FOR THE ESTIMATED SERVICE STRESS OF 91 MPa (13.2 ksi) WINDOW FAILURE WOULD OCCUR IN APPROXIMATELY 3+ DAYS

Figure 4 Increase in temperature decreases the static fatigue life of BeO tested in fluorocarbon fluid. Such increases in temperature could occur in the gyrotron tube as a result of dielectric heating of the ceramic and cooling fluid.

TABLE I Fracture mechanics parameters derived for the window materials

Parameter	995S BeO	AL 995 Al_2O_3
K_{IC} (MPa $m^{1/2}$)	4.8	4.5
n	17	19.8
A'	1.07×10^{-11}	7.34×10^{-13}
Y	$\pi^{1/2}$	$\pi^{1/2}$
m	13.3	14.4
σ_0 (MPa)	193	286

3.3. Fatigue reliability diagram

When combined with the probability-of-failure data, the measured static fatigue behaviour can be used to predict the fatigue behaviour for various levels of probability of failure (i.e. 1 − probability of surviving). One can recognize that the predictions are influenced by the number of data points in the sets (i.e. the confidence limits for each probability level will decrease with increasing number of data points). However, it is extremely useful to observe what happens when desired probability of failure levels are prescribed.

This is accomplished by applying the analysis for the time to failure, t, at various applied stress levels, σ_a,

$$t = \frac{(2K_{IC}/\sigma_0)^{2-n}}{AY^2(n-2)}\sigma_a^{-n}\left[\ln\ln\frac{1}{1-P_f}\right]^{(n-2)/m}, \tag{6}$$

where the values of the required parameters (Table I) are obtained from the experimental data in Figs. 2 and 3 except for the values of the critical stress intensity factor, K_{IC}, which were obtained from the applied-moment double-cantilever test specimens [11].

The resultant design diagrams for the Thermalox 995 standard BeO and the AL 995 alumina are shown in Figs. 5 and 6, respectively.

Using such design diagrams and the stresses imposed on the window materials by dielectric heating due to microwave beam-material interactions [7] one can, using Equation 6, predict the lifetime of the window. This has been accomplished for face cooled 3.175 cm radius windows that are 0.24 cm thick for a TE_{02} microwave beam operating at 28, 60, and 100 GHz at a continuous wave (CW) power level of 200 kW (refer to Table II of [7]) for two different surface heat transfer conditions. As seen in our Table II, for face cooled windows, the applied tensile stress levels due to dielectric heating increases with increasing operating frequency with an attendant decrease in time to failure in both the BeO and Al_2O_3 ceramics. Note that the calculated window stresses for different microwave beam frequencies based in the stress analysis in [7] included the effects of the dependence of dielectric properties upon microwave frequency (Table III, [7]). Decreasing the allowed probability of failure from one failure in one thousand windows to one in two windows also increases the time to failure. One can see that increasing the surface heat transfer, by increasing the flow of the FC-75 coolant or by decreasing its temperature by passing it through a heat exchanger, will increase the window life. The estimated time to failure even for the increased heat transfer conditions indicate limited lifetimes for

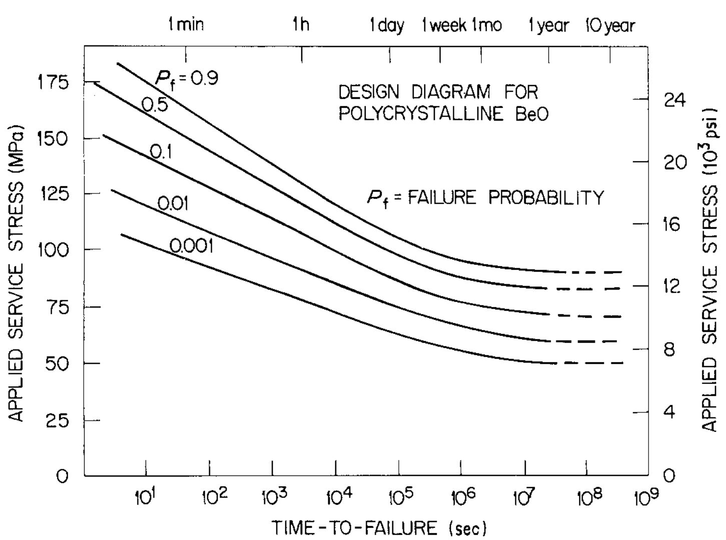


Figure 5 Design diagram for BeO ceramic represents time to failure plotted against applied flexure stress level for various probability of failure levels at 22° C. The service stress level was determined by analysis of radial and axial tensile stresses generated in a double-disc window structure (each disc being about 76 mm diameter and 2.5 mm thick), in which fluorocarbon coolant passes between the two parallel discs. The gyrotron operating conditions consisted of 200 kW power at 60 GHz.

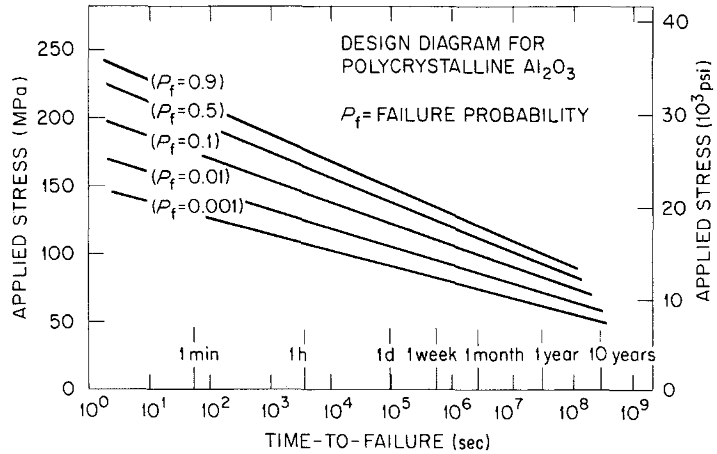


Figure 6 Design diagram for alumina ceramic based on static fatigue behaviour at 22° C. Service tensile stresses analysed for conditions comparable to those used for BeO windows.

microwave frequency levels of ⩽ 60 GHz for these window sizes even at high allowable probabilities of failure.

As pointed out in Part 1 [7], increasing the window radius with respect to the wave guide radius lowers the maximum applied tensile stress due to dielectric heating. This has a significant effect on extending the lifetime of the window as shown for the face cooled alumina window, Table III. Note these lifetime predictions are for a 60 GHz microwave beam operating in the TE_{02} mode at a 200 kW CW power level. Thus relatively minor changes in the window dimensions can substantially improve the performance of the gyrotron.

Use of the fatigue data for the flexure bars with the calculated service stress levels in a window disc will yield a somewhat greater reliability for the design life of a window disc than would strength data based on on full size windows. This is related to two factors. First, the service stresses have been calculated in a manner requiring that the energy level and energy density remain *constant* with no spurious increases in local energy density as might occur in actual tube operation. Second, the fatigue data are based on samples having both smaller surface area and volume than the actual window. Because both the strength distributions and the time to failure increase with decreasing surface area or volume, the reliability diagrams will predict slightly longer time to failures (at each chosen probability of failure level) than may occur in an actual window.

For either material, the design diagrams indicate that about one of two windows will fail in approximately 1 month under such calculated service stresses at a temperature of 22° C. On the

TABLE II Influence of operating frequency of microwave beam and surface heat transfer conditions on lifetime of window

	Material					
	BeO	BeO	BeO	Al_2O_3	Al_2O_3	Al_2O_3
Frequency (GHz)	28	60	100	28	60	100
Normal surface heat transfer*						
σ_a [MPa (10^3 psi)]	20.3(4.1)	69.6(10.1)	151.7(22.0)	68.9(10.0)	196.5(28.5)	373.4(54.2)
t_f (P_f† = 0.5)	50639 yr	29.0 day	23.7 sec	1.446×10^7 yr	1.29 h	⩽ 1 sec
t_f (P_f† = 0.001)	22.7 yr	18.7 m	⩽ 1 sec	133.8 yr	⩽ 1 sec	⩽ 1 sec
Increased surface heat transfer†						
σ_a [MPa (10^3 psi)]	17.2(2.5)	57.2(8.3)	106.2(15.4)	51.7(7.5)	123.4(17.9)	255.1(37.0)
t_f (P_f‡ = 0.5)	8.2398×10^7 yr	1.5 yr	1.35 h	1.4117×10^{10} yr	10.6 yr	8.1 sec
t_f (P_f‡ = 0.001)	3.691×10^4 yr	5.75 h	2.2 sec	1.377×10^5 yr	0.9 h	⩽ 1 sec

*Surface heat transfer conditions described by Equation 5, Cases 1 and 2, Table II, [7].
†Surface heat transfer conditions described by Equation 6, Cases 4 and 5, Table II, [7].
‡P_f is the probability of failure level.

TABLE III Influence of window radius on the time to failure of face cooled alumina windows*

Window radius (cm)	3.81	5.08	7.62
Maximum applied tensile stress [MPa (10^3 psi)]	196.5(28.5)	144.7(21.0)	120.6(17.5)
Time to failure, t_f at probability of failure, $P_f = 0.5$	1.29 h	82.7 day	18.4 yr
0.001	≤ 1 sec	69.7 sec	1.57 h

*Microwave beam operating at 60 GHz, 200 kW CW in TE_{02} mode for surface heat transfer condition based on Equation 5, [7].

other hand, if only one in 1000 windows are allowed to fail, then the service lifetime would be less than 1 h. Obviously, neither of these results is satisfactory if long lifetimes and high survivability are desired for the window. In addition, the present results, which show decreasing fatigue life with increasing temperature of the fluorocarbon fluid (Fig. 4), point out that the above predicted lifetimes will decrease with any increase in the temperature at the ceramic–coolant interface due to microwave heating.

These materials are, however, adequate for short-term testing during the development of 60 GHz tubes, although reliable long-term tube operation may not be achieved with such materials.

Therefore, one needs to look for material improvements that can be achieved in the near term. Recent findings on the fatigue behaviour of anisotropic noncubic polycrystalline alumina indicate that greater fatigue resistance can be obtained by reducing the grain size [12]. It is also well known that the critical fracture strength of polycrystalline ceramics can be increased by increasing their density and/or reducing their grain size. Thus, substantial improvements in the mechanical reliability of the window can be obtained by additional refinements in processing the materials to achieve both densities greater than 97% theoretical and uniform grain sizes of less than 10 μm. In fact, a small effort in progress has shown that BeO ceramics having densities greater than 99% theoretical and grain sizes of less than 5 μm can readily be achieved by hot-pressing fine (submicron) BeO powders obtained by ball milling and the use of less than 4 wt% MnO additions [13]. Furthermore, the analysis of the service stress reveals that increasing the window diameter can decrease the applied tensile stress and, hence, increase the time to failure. Studies are under way to determine the mechanical reliability of windows made from such improved materials and due to changes in the window design.

4. Conclusions

1. The mechanical properties pertinent to the gyrotron window reliability of both the Thermalox 995 standard BeO and the AL 995 Al_2O_3 ceramics have been determined. These properties include the critical (inert) fracture strength distribution, and static fatigue behaviour in the liquid fluorocarbon environment used as a window coolant at 22 and 48° C.

2. These results show that environmentally assisted slow crack growth (fatigue) occurs in both of the polycrystalline ceramics in the fluorocarbon fluid, which contains only 7 ppm H_2O (the solubility limit). The fatigue rate is increased nearly ten-fold by increasing the test temperature from 22 to 48° C.

3. Design diagrams based on the above data reveal that these materials are probably not satisfactory for *long* service life and *high* survivability levels at the estimated service stress levels determined by analysis of microwave heating in the TE_{02} mode. However, such materials are probably adequate for tube design development and short-term testing except at operating frequencies of ≥ 60 GHz.

4. In the near term, advanced materials could be obtained by improvements in the fabrication of the BeO and Al_2O_3 ceramics. Materials having both densities greater than 97% theoretical and uniform grain sizes of less than 10 μm offer the potential for significantly improving the mechanical reliability of gyrotron windows.

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