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Results of Some Laboratory Water Vapor Corrosion Tests of BeO

Donald G. Salyards

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D. G. Salyards

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ABSTRACT

Some experimental corrosion rates were determined for unfueled, uncoated BeO tubes at temperatures of 1800°F, 2150°F, and 2600°F in moist air, and are compared to the theoretical corrosion rates. With one exception all tests were run at the mass flow rate of 0.0023 lb/sec and a water vapor content of 0.76 percent by weight. The experimental test apparatus and techniques are described.

FORWARD

This report was originally intended to include the dimensional stability and thermal stress resistance of BeO tubes but these topics have been treated in GE-ANP APEX's 756 and 757.

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Acknowledgement is made of the contributions by the following persons in the preparation of this report: J. B. Conway, J. Holowach, and R. A. Stanley

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1. INTRODUCTION

During the first four months of 1961 some water vapor corrosion tests on individual tubes were conducted in the laboratory to experimentally determine the time-temperature limitations (from the standpoint of water vapor corrosion) of unfueled, uncoated BeO tubes to be used in the reflector region of the reactor for the ACT. These tests were performed by passing moist air through the tube bore at a moderate flow rate. The tubes were heated by radiant heat transfer from a tungsten heating element surrounding the outside surface of the tube and this heat was conducted through the tube wall to the air flowing through the bore of the tube. The majority of these single tube tests were operated continuously up to 1000 hours at the test temperature except that the tube temperature was cycled every 24 hours to room temperature on a 2-hour combined cooling and heating schedule without any change in the flow of moist air through the bore. In addition, throughout the test the tube was removed from the furnace approximately five times for visual inspection and to measure the change in overall tube pressure drop due to corrosion of the BeO. The purpose of these tests was to determine the temperature regions of the reflector where it should be necessary to coat the tubes; in addition these test results were analyzed in such a manner that the experimental data obtained could be compared to the calculated corrosion rates as obtained from the theoretical equations reported by J. B. Conway (1).

2. EXPERIMENTAL TECHNIQUES

(a) Heating Equipment

A comprehensive treatment of the heating apparatus which was employed for these tests has been reported (2). One of the tubes was tested in an Inductively-Heated Tungsten Muffle Furnace and all the others were tested in a Resistively-Heated Tungsten Wire Helix Furnace. These furnace types are similar in that heat is transferred radiantly from the heating element to the BeO tube; however, the longitudinal temperature profile of the tube is somewhat different in each furnace. The maximum temperature of the inside wall of the tubes was measured in all instances by optical pyrometer sightings within the bore of the tube. This maximum measured temperature is dependent upon the optical sighting path, tube size, tube material, etc. Prior calibration tests were performed using Pt/Pt-10% Rh thermocouples embedded in the tube wall. The longitudinal temperature profile of the tube is a function of heating element length, test temperature, test heat flux, bore cooling air temperature, etc., so this aspect was also determined by prior calibration tests using thermocouples.

(b) Operational Characteristics and Measurements

All the tubes tested were hexagonal in outer configuration and were of the same nominal dimensions, i.e., 0.249-inch across flats, 0.167-inch inside diameter, and 4.42 inches long. The cooling air mass flow rate was 0.0023 lb/sec in nearly all the tests. With

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this moderate flow rate and the heating apparatus which was used, the longitudinal temperature profile of the tube is inherently non-isothermal. The corrosion mechanism is, of course, a function of the temperature level and therefore, because of the varying temperature along the length of the tube, the local corrosion rates are quite different. These conditions make overall weight loss measurements of the tube of little value in determining the quantity of BeO which has been hydrolyzed from the tube at a particular temperature (thereby permitting the weight-loss rate per unit area to be ascertained for that temperature). Further, redeposition of the hydrolyzed BeO within the bore of the tube as whiskers or crystals would subject such weight-loss determinations to some degree of error. Redeposition of BeO has been observed in zones where the wall temperature is decreasing in the direction of air flow as well as at the zone of maximum temperature. In a few instances redeposition has been observed in zones where the wall temperature was increasing in the direction of air flow.

Most of the previous laboratory water vapor corrosion studies have employed a measured increase in pressure drop to indicate the extent of hydrolysis taking place. It is not possible to convert this type of data into determinations of the weight-loss rate per unit area and such measurements are merely useful indicators of the physical damage or change sustained by a tube. Most attempts to correlate such data from various tests have not been very satisfactory. These pressure-drop measurements are influenced by too many parameters and interpretation is difficult. The change in pressure drop may be the result of roughening of the inner tube surface by the water vapor corrosion, redeposition of the hydrolyzed BeO in forms of varying aerodynamic drag characteristics, deposition of foreign material on the inner tube surface, change in dimensions of the flow passage, etc. In addition, since the change in pressure drop has been measured in a cold-flow apparatus, some fragile shapes of redeposited BeO might have been dislodged in the interim between furnace operation, thermal cycle to room temperature, and pressure-drop testing. Further, all the parameters leading to variations of pressure drop are dependent upon temperature and because of the longitudinal temperature gradients of the tube, which are characteristic of these particular tests, only a portion of the tube is exposed to the temperature level of interest. Therefore a given measured change in pressure drop requires a special interpretation because localized characteristics may be quite different from what the overall pressure drop change would indicate.

A better insight into the localized-damage phenomenon can be obtained by using cold-flow apparatus which is designed to measure and record the longitudinal static pressure along the axis of the tube. This apparatus yields the longitudinal static-pressure profile by slowly traversing the bore of the tube with a small diameter static-pressure probe while recording the static pressure and traverse distance on a two-function plotter. The pressure lead from the static-pressure probe is connected to an electrical transducer and the static pressure is recorded by a single pen on a strip chart. The linear probe movement is synchronized to the chart drive of the recorder. Typical

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pressure profiles for a tube are shown in Figure 1. The approximate constant-slope curve of the tube before test is an indication of a constant-area flow passage with uniform frictional losses. This apparatus can detect small local changes in static pressure which cause the trace to exhibit waves of small amplitude and frequency. Such traces have been observed with coated tubes in which the coating appears to be rippled. The friction factor for a tube can be computed from the slope of the static-pressure profile. The pressure profile after testing is also shown in Figure 1. This tube has had BeO hydrolyzed from the inner surface in the center section so that in this region the flow area has been increased. The bore of this tube was purposely cleaned with a pipe cleaner before the post-test trace was made so that fragile BeO deposits would be removed. In this instance the local increase in the static pressure at a particular longitudinal location is largely the effect of expanding flow area, but often such a local increase will include substantial frictional effects if there is an appreciable increase in the overall pressure drop of the tube. The change in the cross-sectional area can be computed by applying the equations for an isentropic expansion; oftentimes, however, frictional effects would make such a solution difficult to obtain with certainty and accuracy. Therefore, these pressure profiles cannot always be converted by computations into precise weight loss determinations which are required to obtain corrosion rate data. The pressure profile shown in Figure 2 is from a tube that had large random crystal deposits (about 0.015-inch high) in the bore. The trace is not smooth because these deposits caused fluctuations in the static pressure. The approximate constant slope at the inlet portion of the tube corresponds to a smooth-bore tube and indicates the absence of water vapor corrosion in this relatively low-temperature region during the test. The downstream end of the tube also indicates the absence of hydrolysis in this area since the pressure gradient is nearly constant and about equal to that of the inlet region. The trace in this downstream region is not exactly smooth but this may be attributed to a very slight redeposition of BeO or perhaps foreign material because the inner surface of the tube was slightly discolored in this area.

None of the above mentioned techniques, i.e. overall weight-loss measurements, overall pressure-drop-change measurements, or static-pressure-profile determinations used in conjunction with compressible-flow calculations, offered sufficiently accurate means to determine the corrosion rate at a particular temperature in this type and method of hydrolysis testing. A method which does permit the various corrosion rates to be ascertained is based on volumetric-displacement measurements. A schematic diagram of the test equipment used in determining the magnitude of the cross-sectional area of the tube by volumetric-displacement methods is shown in Figure 3. The tube is soaked in the tank, which contains water plus a wetting agent, for about one hour and then is attached by a wire to the single-pan analytical balance. The linear micrometer screw is then manually operated to lower the water vessel until an incremental change in the total weight of plus 0.1 gm is read on the balance. The linear movement of the vessel is then read directly from the micrometer scale. The incremental volume of the tube which has been immersed in the water is then equal to 0.1 cc (0.1 gm = 0.1 cc for SP.GR. = 1) and the average solid cross-sectional area can then be

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Figure 1 - Longitudinal static-pressure profile of an unfired, uncoated BeO tube before and after test (Test #9).

Figure 2 - Longitudinal static-pressure profile of an unfueled, uncoated BeO tube after test with random crystal deposits (Test #11).

Figure 3 - Schematic arrangement of volumetric-displacement-measurement apparatus.

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computed by dividing 0.1 cc (volume) by the linear movement.

Several checks on this procedure have been made using tubes which have not been tested. A cross-sectional profile of one such tube is shown in Figure 4 and is constant within ± 0.5 percent along the length of the tube. The cross-sectional area was also computed based on the across-flats dimension measured with a standard micrometer caliper and a thickness gage to measure the wall thickness at the midpoint between corners (thinnest section of the wall). The results of these measurements are also shown in Figure 4 and are in excellent agreement with the cross-sectional area determined by the volumetric-displacement method.

This method permits local cross-sectional-area changes to be determined which can then be directly converted into corrosion rate data providing other dimensional changes such as matrix growth or swelling do not occur simultaneously. The stability of the across-flats dimension of the tube after test is sufficient evidence to confirm the absence of extraneous dimensional changes.

3. EXPERIMENTAL RESULTS

Ten tubes were tested in conjunction with this water vapor corrosion study on unfueled, uncoated BeO reflector pieces for the ACT for intervals of time between 800 and 1008 hours. The maximum tube temperature was 1800°F for three of these tubes and was 2150°F for the other seven. One other tube was operated at 2600°F for 146 hours with a cooling-air mass flow rate of 0.004 lb/sec instead of 0.0023 lb/sec which was used for all other tests. All tests were run with an inlet cooling-air temperature of 70°F and a water vapor content of 0.76 percent (50°F dew point) by weight in this air. With these conditions of mass flow rate, cooling-air temperature, tube-test temperatures, and tube dimensions, the tubes were operated at the maximum heat fluxes of 0.095, 0.12, and 0.21 BTU/sec-in² for the test temperatures of 1800°F, 2150°F and 2600°F respectively. The three tubes tested at 1800°F were run continuously for 800 hours each with only a single temperature cycle to room temperature at the conclusion of each test. The tube tested at 2600°F was run in this same manner. The seven tubes tested at 2150°F were all cycled to room temperature every 24 hours on a 2-hour combined cooling and heating schedule without any change in the flow of the moist air through the bore. In addition, two of these seven tubes were run with the cooling-air water content increased from 0.76 percent to 1.5 percent for 5 hours of each 100 hours of operation. All the tubes, with the exception of the three tested at 1800°F, were operated close to atmospheric pressure. The cooling air was simply exhausted from the exit end of the tube into a plenum maintained at atmospheric pressure. This plenum was maintained at 60 psig for the three tubes tested at 1800°F.

Table 1 presents the experimentally determined corrosion rates for the eleven tests. These rates have been calculated from the post-test cross-sectional profiles obtained by volumetric-displacement measurements. For illustrative purposes, the profiles in Figures 5 through 8 represent one tube for each of the three test temperatures and also one tube with the water content cycled to 1.5 percent. In these figures the reciprocal cross-sectional area is plotted as a function of specimen length since

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Figure 4 - Average cross-sectional area profile of an unfired, uncoated BeO tube as received.

TABLE I
WATER-VAPOR CORROSION OF UNFUELED, UNCOATED BeO TUBES

Test No.	Specimen Number	Inside Surface Temp, °F	Dew Point, °F	Mass Flow, lb/sec	Time On Test, hr.	Experimentally-Determined Corrosion Rate, gm-BeO/sec-cm ²	Theoretical ⁽¹⁾ Corrosion Rate, gm-BeO/sec-cm ²	Theoretical ⁽¹⁾ Temp. that would yield Exp. Corrosion Rate, °F *	Theoretical ⁽¹⁾ Dew Point that would yield the Exp. Corrosion Rate, °F **
1	N2250-3	1800	50	0.0023	800	Not measurable	$9.5(10^{-10})$		
2	N2257-1	1800	50	0.0023	800	"	$9.5(10^{-10})$		
3	#6	1800	50	0.0023	800	"	$9.5(10^{-10})$		
4	N2250-4	2150	50	0.0023	1000	$2.50(10^{-9})$	$10.5(10^{-9})$	1925	15
5	N2253-2	2150	50	0.0023	1008	$3.07(10^{-9})$	$10.5(10^{-9})$	2100	43
6	N2253-3	2150	50	0.0023	1008	$7.57(10^{-9})$	$10.5(10^{-9})$	2090	41
7	N2254-1	2150	50	0.0023	1008	$4.45(10^{-9})$	$10.5(10^{-9})$	2000	30
8	N2254-3	2150	50	0.0023	1008	$7.18(10^{-9})$	$10.5(10^{-9})$	2080	40
9	N2257-3	2150	50/70	0.0023	974	$7.46(10^{-9})$	$11(10^{-9})$	2090	41
10	N2257-4	2150	50/70	0.0023	974	$6.52(10^{-9})$	$11(10^{-9})$	2065	38
11	#12	2600	50	0.004	146	$7.38(10^{-8})$	$14(10^{-8})$	2450	34

* Assuming that the test was operated at the designated mass flow and dew point continuously

** Assuming that the test was operated at the designated mass flow and temperature continuously.

(1) See Reference (1) in Bibliography

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Figure 5 - Longitudinal reciprocal cross-sectional-area profile of an unfired, uncoated BeO tube after test (Test #3).

Figure 6 - Longitudinal reciprocal cross-sectional-area profile of an unfused, uncoated BeO tube after test (Test #6).

Figure 7 - Longitudinal reciprocal cross-sectional-area profile of an unfueled, uncoated BeO tube after test (Test #9).

Figure 8 - Longitudinal reciprocal cross-sectional-area profile of an unfueled, uncoated BeO tube after test (Test #11).

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this is a more convenient way to handle the physical volumetric-displacement measurements. The longitudinal temperature profiles of the tubes in the test zone are also plotted on these figures. From these data the local corrosion rate can be computed as follows:

Referring to Figure 7 -

Initial reciprocal cross-sectional area of the tube, $A_{RX_1} = 5.04 \frac{1}{\text{cm}^2}$

(This is an average value from the inlet region of the tube which is not subjected to the test temperature).

Initial cross-sectional area of the tube, $A_{X_1} = 0.1984 \text{ cm}^2$

Post-test reciprocal cross-sectional area of the tube,

$$A_{RX_0} = 5.33 \frac{1}{\text{cm}^2}$$

(Interpolated from Figure 7, location of maximum temperature in this sample calculation).

Post-test cross-sectional area of tube, $A_{X_0} = 0.1876 \text{ cm}^2$

Net decrease in cross-sectional area

$$A_{X_1} - A_{X_0} = 0.1984 - 0.1876 = 0.0108 \text{ cm}^2$$

$$\text{Percent decrease in cross-sectional area} = \frac{0.0108}{0.1984} \times 100 = 5.44$$

Assuming that the tube was of uniform density, the percent loss in weight would be the same as the percent decrease in cross-sectional area. (The following computations are not based upon a segmental basis, but rather as if the entire tube had been subjected to 2150°F instead of only the short region).

Tube weight before test, $M = 6.5955 \text{ gm}$

Total calculated loss in weight
after test $\Delta M = 6.5955 (.0544)$
 $= 0.3588 \text{ gm}$

Tube I.D., $D = 0.167 \text{ inch}$

Tube I.D. area, $A_s = \text{Circumference} \times \text{length}$
 $= \pi (0.167) (4.42)$
 $= 2.319 \text{ in}^2$
 $= 14.96 \text{ cm}^2$

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Time on test,

$$t = 974 \text{ hrs.}$$

$$= 3.506 (10^6) \text{ sec.}$$

Therefore,

$$\begin{aligned} \text{Experimental corrosion rate, } W \frac{\text{gm-BeO}}{\text{sec-cm}^2} &= \frac{\Delta M}{t(A_s)} \\ &= \frac{0.3588 \text{ gm}}{(3.506 \times 10^6 \text{ sec})(14.96 \text{ cm}^2)} \\ &= 6.84(10^{-9}) \frac{\text{gm}}{\text{sec-cm}^2} \end{aligned}$$

This experimental corrosion rate does not take into account the change in exposed surface as corrosion progresses, nor that the tube was cycled every 24 hours as described previously. The time on test used in the calculation above should be adjusted accordingly. Using 893 hours as the time on test ($974 \times \frac{22}{24} = 893$)

$$W = 7.46(10^{-9}) \frac{\text{gm}}{\text{sec-cm}^2}$$

This corrosion rate may be expressed in terms of mils of thickness of BeO removed from the cylindrical inner surface by merely computing the final I.D. taking into account the decrease in cross-sectional area of 0.0108 cm^2 (0.00167 in^2).

Initial tube bore area

$$\begin{aligned} A_{B_i} &= \pi/4 (0.167 \text{ in})^2 \\ &= 0.02190 \text{ in}^2 \end{aligned}$$

Post-test tube bore area

$$\begin{aligned} A_{B_o} &= 0.02190 + 0.00167 \\ &= 0.02357 \text{ in}^2 \end{aligned}$$

Therefore, Post-test tube I.D.

$$D_o = 0.1732$$

Therefore, Corrosion rate, W mils/hr

$$\begin{aligned} &= \frac{D_o - D}{2(t)} = \frac{(0.1732 - 0.167)(10^3)}{2(893)} \\ &= .00347 \text{ mils/hr} \end{aligned}$$

or

$$W = 3.47 \text{ mils/1000 hr}$$

The above-calculated post-test tube I.D., $D_o = 0.1732$ is in excellent agreement with that which can be calculated from the static-pressure profile for

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this tube which is shown in Figure 1. The post-test maximum deviation of this profile from the pre-test static-pressure profile corresponds to the same longitudinal location on the tube as that of the maximum reciprocal cross-sectional area of the tube shown in Figure 7, as would be expected. Using the local increase in static pressure in this region in conjunction with the flow conditions of the traverse apparatus, and employing the relationships for the isentropic expansion of a perfect gas, the calculated post-test tube I.D. in this region is 0.1736 inch. This measurement technique was employed as a check against the corrosion rates determined by the volumetric-displacement technique for some of the other tubes tested, and nearly the same level of agreement was obtained in each instance as that above.

The difficulty in utilizing over-all weight-loss measurements to determine corrosion rates in tests of this type is illustrated by weight data on the tube that was discussed above. The pre-test weight was 6.5955 gm and the post-test weight was 6.5462 gm so that the loss in total weight was 0.0493 gm. In Figure 7 the area under the reciprocal cross-sectional-area curve and above the original 5.04 l/cm² base line at the inlet of the tube represents the volume of BeO which has hydrolyzed from the bore of the tube. If this area is distributed uniformly along the length of the tube, the overall reciprocal cross-sectional area is changed from the original 5.04 l/cm² base line to a final 5.10 l/cm² value. This represents 1.209% change in the cross-sectional area which would also be a 1.209% decrease in the total weight of the tube assuming, as before, uniform material density. This would yield a calculated 0.0797 gm loss in the tube weight instead of the 0.0493 gm which was measured. This apparent discrepancy between the calculated and measured weight loss can be explained, in part at least, by the shape of the reciprocal cross-sectional area in the downstream region of the tube. In all cases the curve comes below the average value of the upstream region which indicates that some material, probably BeO, has been deposited in this area.

Post-test inspection of the tubes run at 1800°F indicated no visual damage to the quality of the bore surface which is substantiated by the negligible increase in overall pressure drop of these tubes after test. The tubes run at 2150°F showed some damage to the bore surface in addition to loss of material. A few very tiny crystal deposits were observed in the tubes during the various inspection points in the hot zone as well as downstream of the hot zone. In addition, some roughening of the downstream area was generally observed which was probably the result of material deposition in this region. The crystals which could be easily seen with the naked eye were very fragile and some were dislodged from time to time by the flow of air employed with the overall pressure-drop measuring apparatus. The tube run at 2600°F had many random crystal deposits in the bore which were not easily dislodged. These are shown in the photograph of Figure 9. It is interesting to compare the longitudinal locations of these crystals to the longitudinal temperature profile of Figure 8 and the static pressure profile of Figure 2. The lower longitudinal half section in Figure 9 has many more crystal deposits than does the upper half section which indicates an asymmetric redeposition tendency in this particular test.

The massive crystal deposits shown in the photographs in Figure 10 resulted during some earlier experiments conducted in conjunction with some mechanical-properties-evaluation tests. Although these experiments were not related to the above water vapor corrosion tests of unfueled, uncoated BeO tubes,

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nevertheless these photographs provide a very descriptive illustration of intensive redeposition of hydrolyzed BeO. This tube was fueled, uncoated BeO, 0.225-inch inside diameter; the cooling-air mass flow was cycled between 0.0065 and 0.003 lb/sec, and the pressure was 25 psig. The test was run for 1000 hours and the water vapor content was 0.76 percent by weight (50°F dew point). The longitudinal half-section shown in Figure 10 (a) had a sharply increasing temperature profile in the direction of the air flow (the extreme upstream end of this section was nearly outside the heated furnace volume). This sample was actually from the first (inlet) of three tubes aligned to form a single air flow channel, and the butt joint between this section and the middle section was about 0.5 inch beyond the downstream end shown in (a). The temperature measured with a thermocouple near this downstream region was about 2300°F. At this point about 6 mils have been removed from the inner surface of the bore. The extent of this reaction correlates very well with the theoretical (1) corrosion rate for these conditions. A nearly exact agreement is obtained if 2250°F is taken as the temperature level at this zone instead of 2300°F.

4. SUMMARY

The corrosion rates which were determined experimentally in these tests are generally in excellent agreement with the calculated corrosion rates as obtained from the theoretical equations (1) of the hydrolysis mechanism. The greatest deviation from the theoretical values occurred in test #4, test #7, and test #11. All the other results have slightly lower values than the theoretical values but are well within the accuracy limitations of the test instrumentation, test control apparatus, and the experimental measurement techniques. The theoretical values which are used in Table 1 were derived from the approximate equation (1) and this method yields higher values than the more accurate equations (1). A closer agreement between the experimental values and the theoretical values would result if the more accurate equations were utilized for the comparison. Included in Table 1 are the theoretical values of temperature and dew point that would yield the experimentally-determined corrosion rate, assuming all other parameters except one were exactly at their proper values. These tabulations show the small deviation in either of these two parameters that would permit exact agreement between the experiment and theoretical corrosion rates. No explanation can be offered for the deviation margins of test #4, test #7, or test #11. Of all the control aspects of these tests, the dew point was the most difficult to maintain. The type of air-humidification apparatus and dew point-measurement equipment which was used did not permit the optimum control which can be obtained with more extensive systems.

The corrosion rates could not be determined for the three tests run at 1800°F because the volumetric-displacement-profile curves were essentially straight lines. The negligible corrosion rate which follows from this observation is in agreement with the theoretical corrosion rate values. For the test conditions and exposure times, the quantity of material removed from the bore of the tube would be only on the order of 0.6% of the total. This low corrosion rate is beyond the accuracy of the volumetric displacement measurement technique. Tubes in this temperature region could certainly be operated in the ACT without coatings to prevent hydrolysis.

Tubes in the 2150°F temperature region would probably require protective cladding. While the rate of material volatilization for the particular

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reactor operating conditions at this temperature level is not significant, the reduction of flow in the air channels from the redeposition of the BeO would have to be considered. The overall pressure-drop change for the seven tubes tested at 2150°F was an increase in all cases in the range of ~~from~~ 10% to 25% but was very changeable for any particular tube at the various inspections. The seemingly erratic data ~~were~~ probably the result of the temperature cycling and the cold-flow pressure-drop testing. It was noted that the visual appearance of the bore was often altered after these pressure-drop measurements, and it is presumed that some crystals were dislodged in the process. These observations emphasize the difficulty in trying to obtain quantitative results from this type of overall pressure-drop measurement. Additional experiments are required to develop a laboratory method that would accurately evaluate the pressure-drop change that would occur in a single ACT air-flow channel. Such a method would have to simulate all the parameters that affect the kinetics of the corrosion and redeposition of BeO.

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Figure 9 - Unfueled, uncoated BeO tube after test (Test #11) showing crystal deposits in the bore (the tube is shown split into two longitudinal half sections).

Air Flow →

PHOTO 23783

(a) Longitudinal half section (about 6X)

(b) End view before sectioning
(about 6X)

PHOTO 23781

NEG. NO. 176

(c) Tube wall (about 35X)

Fig. 10 - Fueled, uncoated BeC tube after test showing massive crystal deposits

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