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REACTION OF POROUS BERYLLIUM IN STEAM

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REACTION OF POROUS BERYLLIUM IN STEAM

FUSION SAFETY PROGRAM/ACTIVATION PRODUCTS TASK

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ABSTRACT AND SUMMARY

This report presents the findings of some additional investigations into the reaction of beryllium of approximately 88% theoretical density with steam. An earlier investigation¹ had shown this material to be extremely reactive compared to dense beryllium. Inductively heated samples developed self-sustaining reactions due to the exothermic heat of reaction at temperatures between 600°C and 700°C. These initial tests did not completely explain the mechanisms causing this behavior. The onset of thermal instability appeared to have a temperature dependent incubation period and some dependence upon specimen geometry. Additional tests have clarified this behavior. We have also obtained measurements of emissivity and permeability. This information will be helpful in the future in modeling reactor accident scenarios. The high effective surface area of porous beryllium has also allowed reaction rates to be obtained at temperatures as low as 400°C. This information for porous beryllium was then used to extend relationships for the reaction rates of dense beryllium in steam to these lower temperatures.

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1. INTRODUCTION

Beryllium has attractive features for use both in first wall components and in the breeding blanket of fusion reactors. Different forms of beryllium have different possibilities for use such as dense beryllium in blankets and plasma facing components (PFCs), plasma-sprayed in PFC components, and porous beryllium components in the blanket. A concern, however, is the reaction of beryllium with high temperature steam resulting from an accident that injects water into the plasma chamber. We have previously investigated the reactions of dense and 88% dense beryllium in steam from 600°C to 1230°C.¹ The investigation showed that the porous beryllium was significantly more reactive. Data at that time indicated that porous beryllium was about 200 times more reactive than dense beryllium. Samples of the porous material also became thermally unstable. Exothermic heat of reaction caused samples to spontaneously react when inductively heated at targeted temperatures of greater than or equal to 600°C. This behavior, although not completely understood, appeared to show some dependence upon time and specimen geometry.

Additional tests have been performed upon porous beryllium. The purposes of these tests were: a) to use the higher effective surface area of porous beryllium to obtain reaction rates for beryllium in steam below 600°C, b) to obtain a better understanding of conditions causing the thermal instability in porous beryllium, and c) to add to our porous beryllium volatility database. Permeability and emissivity were two parameters measured to assist in the second objective and to contribute to the database for future modeling efforts of reactor transients. Specimens of different geometries were tested in a resistance heated furnace where the mode of sample heating is by radiative heat. This allowed us to check for the influences of property changes, such as electrical resistance, which might impact the extent of heating caused by an induction field. This report presents the findings from these investigations.

2. CONCLUSIONS

1. The threshold temperature for the onset of thermal instability for the porous beryllium is between 650°C and 675°C. This temperature is common for both inductively and radiatively heated specimens. Earlier indications of temperature dependent incubation periods and geometry influences upon the onset of thermal instability, some as low as 600°C, were incorrect and caused by invalid temperature indications and control inherent to the experiments using induction heating.¹
2. Reaction rates of porous beryllium with steam depend both upon surface area and sample mass. Surface area initially provides the closest representation of reaction rates expressed as hydrogen production (liters/m²-s). However, as the reaction proceeds the samples develop greater porosity. This enhances the access of steam within the specimen resulting in a greater relative proportion of the

reaction occurring within the specimen. Reaction rates (liters/m²-s), consequently, increased relative to initial rates based upon external surface area.

3. The slope for the reaction rates of porous beryllium on the reciprocal temperature plot is not exactly parallel to that for dense beryllium as suggested previously.¹ This is likely related to the phenomena expressed in Item 2, i.e., greater relative amounts of reaction occur internally with progressively longer times and at lower temperatures.
4. Volatility was quite low for the porous beryllium specimens tested at temperatures below 600°C.

3. RECOMMENDATIONS

The results from this investigation have provided the basis for the following recommendations.

1. Porous beryllium of approximately 88% full density becomes thermally unstable near 675°C. From a safety point of view, it would be best to avoid using this form of beryllium because it is high reactivity. If the use of this material is unavoidable, its use should be limited to temperatures sufficiently below this threshold temperature, e.g., 600°C if exposure to steam is possible.
2. The reaction rate for the porous beryllium from 400°C to 700°C in terms of [liters H₂(STP)/m²-s] may be represented by Equation (1). Temperature is expressed as degrees K.

$$\begin{aligned} \text{Reaction Rate (liters/m}^2\text{-s)} &= 12.3 \times 10^4 e^{-12,500/T}, & (1) \\ \text{Temperatures: } &\geq 400^\circ\text{C} \leq 700^\circ\text{C} \end{aligned}$$

3. Reaction rates for dense beryllium can be represented for temperatures from 600°C to 900°C by Equation (2) shown below and reported in Reference 1. The constant R is 8.317 J/mole-K and temperature (T) is expressed as degrees K.

$$\begin{aligned} \text{Reaction Rate (liters/m}^2\text{-s)} &= 4.8 \times 10^9 e^{-215,000/RT}, & (2) \\ \text{Temperatures: } &\geq 600^\circ\text{C} \leq 900^\circ\text{C} \end{aligned}$$

For reaction rates of dense beryllium at temperatures below 600°C, and for a measure of conservatism, we recommend using Equation (1) developed for porous beryllium divided by a factor of 100. Thus reaction rates for dense beryllium below 600°C may be expressed as Equation (3).

$$\begin{aligned} \text{Reaction Rate (liters/m}^2\text{-s)} &= 12.3 \times 10^2 e^{-12,500/T}, & (3) \\ \text{Temperatures: } &\leq 600^\circ\text{C} \end{aligned}$$

4. The emissivity measured for porous beryllium when initially exposed was near 0.25 and increased with exposure. This value of 0.25 was obtained from a single wavelength (1.65 μm) and at an angle of approximately 30 degrees from normal. This adds to the database but additional information for total emittance would be needed for use in thermal modeling.
5. The permeability of gases in porous beryllium has been determined. The constant (K_a) characteristic for this material was found to be 0.98 millidarcy. The following relationship, Equation (4), is recommended for use when applying this constant in modeling other gases and geometries.

$$K_a(0.98 \text{ millidarcy}) = (MQL) / (A\Delta P) \times 1000 \quad (4)$$

M is the viscosity of the gas in centipoise. Q is flow rate (cm^3/s), L is sample length (cm). A is sample area in cm^2 . ΔP is the absolute pressure drop across sample in atmospheres. A centidarcy is a flow of 0.01 cm^3/s of a fluid with one centipoise viscosity through a 1-cm cube under a pressure difference of one atmosphere.

4. EXPERIMENTAL PROCEDURES AND RESULTS

4.1 General Test Procedures

Tests in steam were performed in two different test set-ups having many similar conditions. One system provided induction heating and was contained in a glove box as described in Reference 1. The other system provided radiative heating in a 36-cm long muffle furnace contained in a ventilated hood. Both systems had a gas-tight glassware assembly which allowed the samples to be purged with argon and preheated. Tests were initiated by switching to 470°C preheated steam supplied at 0.17 g/s. Both test chambers were 6.4 cm in diameter and produced a steam velocity of 0.26 m/s at the specimen surface. Samples were then heated to the targeted temperature. Steam temperatures did not increase significantly above 470°C for experiments using induction heating, however, the steam was heated to approximately the targeted test temperatures in the muffle furnace. Gas flow was directed through a condensing system and into a collection system. Complete recondensation of steam allowed hydrogen, the residual product gas, to be used as a means to measure reaction rates. The various glassware components were cleaned with acid solutions and analyzed with inductively coupled plasma atomic emission spectroscopy (ICP-AES) to check for the possible transport of beryllium oxidation products.

The porous material used for this investigation had the same background as that used for our previous investigation.¹ The powder was Brush-Wellman structural powder, SP-200-F, having particle sizes of 10 to 12 μm . This powder had been isostatically cold pressed into billets at 415 MPa and then sintered for 4 hours at 1100°C in a vacuum of 0.013 Pa. Although samples were prepared from different billets of approximately 44 mm

diameter, bulk densities were all within the range of $1.63 \pm 0.05 \text{ g/cm}^3$. Apparent porosities ranged from 10.5 to 13 percent. Samples from the various billets could therefore be assumed to have quite similar structure. Two difference sizes of specimens were used. Both were approximately 25.4 mm in diameter, but, Type A specimens were 2.54 mm thick and Type B specimens were 12.70 mm thick.

4.2 Emissivity Measurements

Emissivity measurements were obtained using the test set-up described in Reference 1. An induction coil was used to heat a sample contained in a quartz test chamber. A thermocouple embedded into a sample 25.4 mm in diameter by 2.54 mm thick was used to monitor temperature and to manually adjust the power on the induction generator. An infrared pyrometer sensing a wavelength of $160 \mu\text{m}$ and having an emissivity adjustment was focused upon the specimen surface at an angle of about 30 degrees from normal. The sample was viewed through quartz windows to reduce interference of the infrared signal. The emissivity settings were adjusted during the course of the test to match indications from the infrared pyrometer with those from the thermocouple. The goal was to measure emissivity changes with time as the test progressed.

Table 1. Emissivity measurements for 600°C test.

<u>Time, minutes</u>	<u>Thermocouple, °C</u>	<u>Infrared Pyrometer, °C</u>	<u>Emissivity</u>
0	600	602	0.24
12	596	602	0.25
15	600	602	0.27
First temperature decay occurred at this time.			
0	600	602	0.29
12	605	602	0.31
17	602	602	0.36
22	600	602	0.49
24	604	602	0.85
25	--	602	0.93

The results from a sample tested at 600°C are summarized in Table 1. Emissivity was initially measured as 0.24 at the beginning of the test. There was a gradual increase to a value of 0.27 after 15 minutes. The test

Figure 1. Temperature decay curves measured for a sample of porous beryllium reacted in steam at 600°C. First cooling after 15 minutes and second cooling after 40 minutes.

was then interrupted and a temperature decay occurred as shown in Figure 1. The steam supply was shut off and a minimum flow of argon of about 0.2 liters/minute was maintained during the cool-down. The sample was then reheated to 600°C in steam. Measured emissivity values continued to gradually increase and reached values of near 0.50 after another 22 minutes. The sample had by then developed a pinkish color. Within three minutes the samples became red and the emissivity settings had to be increased to 0.93 to maintain a match between the thermocouple and infrared pyrometer. Power and steam supply to the sample was then shut off and a second temperature decay obtained. This time-temperature plot is also included in Figure 1.

The trend of increasing emissivity with time shown in Table 1 agrees with some emissivity changes reported in the literature. Data compiled by Touloukian and DeWitt² show progressively higher emissivity values for tungsten with 30 percent and 45 percent porosity compared to polished fully dense tungsten. Oxidation can also increase emissivity. These same authors show that polished, grit blasted and oxidized niobium at 800°C provide emissivities (hemispherical total emittance) of about 0.15, 0.30, and 0.70, respectively. An example is also given whereby emissivity (normal total emittance) for beryllium metal increased from 0.5 to 0.9 when heated from 900°C to 1200°C, and remained at the latter value when cooled back to 900°C. This same reference also shows that the normal spectral emittance of beryllium oxide is quite dependent upon minor material differences. Emittance values were shown to vary from less than 0.05 to 0.55 at the wavelength corresponding to our experiment. Direct comparisons of emissivity values are complex. However, a trend of higher emittance with the development of additional porosity or oxidation seems logical.

An increase in emissivity, as indicated above, and an increase in surface area due to sample swelling would increase radiative heat losses from a sample. This effect would oppose the onset of a self-sustaining reaction. Possible influences upon the onset of thermal instability may therefore be higher reaction rates due to greater internal reaction with time, or invalid temperature indications for the inductively heated specimens. Subsequent information presented from radiatively heated specimens shows a) there is some increase in sample reaction rates with time, but more importantly, b) the embedded thermocouple loses contact with the specimen as the sample swells. This causes invalid temperature indications and an over-heating of samples tested with the induction heating method. This also raises some question about the integrity of emissivities measured during the latter stages of the test reported in Table 1.

4.3 Radiatively Heated Samples (Temperature Dependence)

A series of the Type A (thin) specimens was tested in the resistance heated shell furnace to determine the temperature at which thermal instability developed. The purpose was to compare this temperature with the thermal instability temperature of 675°C determined for inductively heated specimens.¹ One thermocouple, embedded in the specimen, supported the specimen in the test chamber. Two additional thermocouples were used. One monitored steam temperatures about four millimeters upstream from the specimen. The other measured temperatures near the inner diameter of the

Figure 2. Hydrogen release from the 2.54-mm thick (Type A) specimens tested between 600°C and 700°C.

furnace chamber. The thermocouple embedded in the specimen was used to monitor and make minor power adjustments when changes in temperature by all thermocouples concurred. However, if the specimen thermocouple showed a sudden increase or changed in disagreement with the other thermocouples then no power correction was made. This method of testing provided a better match between the specimen and test environment temperatures and a lower possibility of overheating the sample compared to the induction heating method.

Figure 2 shows records of the amounts of hydrogen collected with time for Type A (thin) specimens tested between 600°C and 700°C. The sample heated to 700°C developed a self-sustaining reaction within four minutes. The other thermocouples never registered temperatures exceeding 700°C during the test. They also lagged behind the specimen thermocouple during the temperature excursion. This demonstrates that chemical energy release from the beryllium:steam reaction was responsible for the temperature excursion. The sample heated to 650°C was held at that temperature for 32 minutes. The sample then fell forward off the thermocouple and into a slightly higher temperature zone. The sample increased its reaction rate and within two to three minutes underwent a temperature excursion. The Type A sample tested at 600°C was releasing hydrogen at a linear rate after 96 minutes into the test. The sample then fell off of the thermocouple. The reaction rate for this sample increased, but, the sample did not develop a self-sustaining reaction. Results from the larger Type B specimens tested between 400°C and 600°C are shown in Figure 3. Hydrogen release was linear during the latter stages of the 600°C test and was terminated after 62 minutes. Hydrogen release initially appeared parabolic but then became linear for both the 3-hour 400°C and 2-hour 500°C tests.

This test series has shown two things: a) thermocouples that are firmly embedded into samples of porous beryllium will become loose as the reaction with steam proceeds, and b) the temperature at which 88 percent dense beryllium develops a self-sustaining reaction in steam is near 675°C. We know that porous beryllium swells and develops additional porosity as it reacts.¹ This causes thermocouple contact with the specimen to degrade. Erroneously low readings result as the thermocouple junction is cooled by steam. In our previous tests using induction heating, we responded by increasing power levels which initiated thermal instability. This explains the delayed, or time-dependent, onset of thermal instability and the geometry effect that we initially believed to have happened in our previous study. But in reality, samples were actually over-heated and this was dependent upon time. The rate for sample swelling is temperature dependent, hence, it would take longer for thermocouples to provide incorrect indications at lower temperatures. Thermocouples are also embedded more deeply into larger specimens providing better security, again, increasing the time required to obtain invalid temperature indications. Temperatures near the wall of the test chamber, or three to four centimeters in front of the specimen, in the radiatively heated set-up were about 20°C to 25°C higher than at the center of the gas stream where the specimen was located. Therefore, in conclusion, the actual temperature at which this material becomes unstable is between 650°C and 675°C.

Figure 3. Hydrogen release from the 12.70-mm thick (Type B) specimens tested at 400°C, 500°C, and 600°C.

4.4 Radiatively Heated Samples (Geometry Dependence)

Geometric effects upon reaction rates are provided from tests of samples of both Type A and Type B at 600°C. Hydrogen releases with time are shown for these samples in Figure 4. We used releases during the first hour to evaluate relative reaction rates with respect to initial surface area and sample mass. The comparison between these two samples is given in Table 2. The Type B specimen had an external surface area of 20.3 cm² compared to 12.2 cm² for the Type A specimen. This is a ratio of 1.66. The mass of the Type B was 10.206 g and the Type A specimen was 2.023 g. This provides a ratio of 5.05. Table 2 shows that the quantity of hydrogen generated by the Type B specimen relative to the Type A specimen increased from 1.9 to 2.7 over the duration of the tests. This shows that surface area provides the better parameter upon which to base reaction rates. However, there is an increasing dependence upon sample mass with time. The curves in Figure 4 also show an increasing release with time. This would correspond with the development of additional porosity and greater internal access of steam resulting in a larger proportion of the reaction taking place within the specimen.

Table 2. Influence of specimen geometry upon hydrogen production.

<u>Time,</u> <u>minutes</u>	<u>Volume, cm³</u> <u>Type A Specimen</u>	<u>Volume, cm³</u> <u>Type B Specimen</u>	<u>Ratio:</u> <u>Type B/Type A</u>
0	0	0	--
5	27	50	1.9
10	43	95	2.2
20	86	195	2.3
30	135	300	2.2
40	193	435	2.3
50	259	615	2.4
60	320	820	2.6
65	352	935	2.7

4.5 Temperature Dependence of Reaction Rate

Samples were tested at temperatures from 400°C to 700°C in the resistance heated furnace. This extends below temperatures obtainable with the induction heating system. A summary of the test data acquired from radiatively heated specimens are given in Table 3. This additional

Table 3. Test data from porous beryllium in steam.

Temp., °C	Heating Method	Specimen Type	Time, minutes	Reaction Rate, $\text{liters/m}^2\text{-s}$	Measured Emissivity
557	Inductive	A	4	5.2E-2	---
649	Same sample	A	6.5	1.6E-1	0.14 - 0.23
600	Inductive	A	15	3.3E-2	0.24 - 0.27
600	Same sample	A	25.5	8.8E-2	0.29 - 0.93
696	Radiative	A	4	4.1E-1	---
656	Radiative	A	32	1.8E-1	---
594	Radiative	A	96	7.0E-2	---
595	Radiative	B	65	1.0E-1 (6.0E-2) ^a	---
494	Radiative	B	120	7.7E-3 (4.6E-3) ^a	---
395	Radiative	B	180	2.4E-3 (1.5E-3) ^{a,b}	---

^aThe rate for the 594°C-Type A was 60% of that for the 595°C-Type B test at 65 minutes. This correction was therefore applied to all Type B specimens.

^bThe detection limit is $4\text{E-}4 \text{ liters/m}^2\text{-s}$.

information has been combined with previously reported data^{1,3-4} and is shown in Figure 5. Data obtained for the porous beryllium from the two different methods of sample heating show good agreement. The one datum point for the 700°C induction test seems too high, but it is also probably the one with the greatest uncertainty. The rapid onset of thermal instability causes higher uncertainties in time and temperature. Reaction rates for the porous beryllium are about 50 and 100 times higher than dense beryllium at 700°C and 600°C, respectively, and about one-fourth of the rates obtained for plasma sprayed samples. With the incorporation of the new data, the slope for the porous beryllium on the Arrhenius plot does not lie parallel to that for dense beryllium. Increasing contributions from internal reaction are likely responsible for this behavior. We have fit the data for the porous beryllium to an Arrhenius relationship for temperatures from 400°C to 700°C. This relationship is shown below as Equation (1).

Figure 4. Hydrogen production from specimens of two different geometries at 600°C. Small specimen (Type A) is 2.54 mm thick. The larger specimen (Type B) is 12.70 mm thick.

Figure 5. Current data from the porous beryllium combined with previously reported data for porous beryllium,¹ dense beryllium,^{1,3} and plasma sprayed beryllium.⁴

$$\text{Reaction Rate (liters/m}^2\text{-s)} = 12.3 \times 10^4 e^{-12,500/T} \quad (1)$$

The datum point at the lowest temperature, i.e., 400°C, appears to lie above the curve determined for the Arrhenius relationship for the porous beryllium in Figure 5. If this is true, linear extrapolation of the curve for dense beryllium to temperatures below 600°C could result in values which are too low. We propose that reaction rates for dense beryllium below 600°C be represented by 1/100th of the reaction rates of porous beryllium, i.e., Equation (1), to provide a factor of conservatism.

4.6 Permeability Measurements

Permeability measurements were made on a sample of the porous beryllium using procedures similar to those described in ASTM Standard Practice C 577. The sample was a Type A specimen with a 25.4-mm diameter and a 0.52-mm thickness. This sample was sealed in a chamber and nitrogen was used as the penetrating gas. The flow rate (Q) was varied from 34.4 to 275 cm³/min. The upstream pressure varied from 34.47 to 347.7 kPa above ambient, i.e., 5 psig to 50 psig. These tests were performed at room temperature and the viscosity of nitrogen was taken as 0.01765 centipoise. Calculations of the permeation constant were made based upon Equation (4).

$$K(\text{millidarcys}) = (MQL) / (A\Delta P) \times 1000 \quad (4)$$

where M is the viscosity of the gas (centipoise), Q is flow rate (cm³/s), L is sample length (cm), A is sample area (cm²), ΔP is the absolute pressure drop across sample (atm.).

Results from this test were used to extrapolate back to a reciprocal pressure of zero to provide a constant (K_a) for this material of 0.98 (millidarcys). Permeability for other gases, e.g., steam, may be obtained by substituting the viscosity of that gas into Equation (4). This relationship could be used to calculate steam penetration into porous beryllium. An application could be calculations of steam penetration and reaction within porous beryllium in breeder blankets.

4.7 Volatility Measurements

Differences in test configurations have made the direct comparisons of volatility data difficult. The test chamber in the electrical resistance heated furnace was positioned horizontally. The sample could not be removed without dislodging oxide within the chamber. Volatilized products were therefore collected only from the two condenser chambers located downstream. The deposition rates on the two condenser chambers and the flux from the specimen based upon the beryllium collected from these two components are shown below in Table 4.

Table 4. Summary of volatilization results.

Temp., °C	Deposition rate first condenser (g/m ² -s)	Deposition rate second condenser (g/m ² -s)	Flux from specimen, (μg/m ² -s)
700	8.0E-6	2.5E-6	132
600	8.6E-7	3.1E-7	14
500 & 400	1E-7	3E-8	1

These values are in reasonable agreement with the flux values which we have previously reported.¹ The values reported for temperatures below 800°C for dense beryllium were generally below 100 μg/m²-s. The above information is useful as an addition to the beryllium volatility database at the INEL. This information will be used in modeling the distribution of beryllium for accident scenarios to determine possible dose exposures for toxicity considerations.

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