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FAIRCHILD

ENGINE AND AIRPLANE CORPORATION

~~RESTRICTED DATA~~

~~ATOMIC ENERGY ACT OF 1946~~

~~SPECIFIC RESTRICTED DATA CLEARANCE REQUIRED~~

THE HEAT CAPACITY OF BERYLLIUM CARBIDE
POWDER IN STAINLESS STEEL CAPSULES AS A
FUNCTION OF TEMPERATURE (400° - 1800°F)

PROJECT NC-Ph-80902

(METHOD OF NC-Ph-80720)

NEPA 932-SCR-32

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ABSTRACT

The mean heat capacity of a sample of powdered beryllium carbide, a possible moderator material for a nuclear reactor, was determined as a function of temperature. A thin walled stainless steel capsule containing the material was heated in an electric furnace and dropped into a water calorimeter. The following linear equation for mean heat capacity over the temperature range 50°C - T°C as a function of temperature, T, was derived from the data by the method of least squares:

$$C_p = 0.299 + 2.16 \times 10^{-4} T \text{ } ^\circ\text{C} \pm 0.005 \text{ cal gm}^{-1}(\text{ } ^\circ\text{C})^{-1}.$$

The numerical value of C_p expressed by this equation is also equal to the mean heat capacity in BTU lb⁻¹(°F)⁻¹. /end

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INTRODUCTION

The heat capacity of a material determines the temperature to which a given mass of substance is raised by the gain of a given quantity of heat. A substance of high heat capacity per unit mass can absorb more heat for a given temperature rise than a substance of low heat capacity per unit mass. The measurement of heat capacity (per unit mass) as well as the total heat capacity is important in the analysis of reactor behavior. For example, the low total heat capacity of the Los Alamos Fast Reactor is one of the factors which makes control difficult.

As outlined in NEPA 816-SCR-28, the heat capacity of materials under consideration for project use is being measured by two general methods. The first of these includes the copper block drop method, (Method NC-Ph-80720), which has been employed for powdered materials contained in metal capsules. The second includes thermal radiation methods intended for use at temperatures above 1500°F.

The objective of this project was to obtain values of the mean heat capacity of beryllium carbide powder as a function of temperature from room temperature to 400°C - 1000°C (752°F - 1832°F) with an error of 15% or less. The experimental procedure was almost identical with that described in NEPA Technical Report NEPA-816-SCR-28. The present method, however, used thin walled stainless steel capsules rather than copper capsules for containing the samples. Data from these experiments were treated more rigorously than those of previous experiments. It was possible to use the least squares method and statistical evaluation because of the large number of experimental values obtained. Two equations, one linear and one parabolic, were fitted to the data, using the least squares technique. The parabolic equation was discarded because of uncertainties in the coefficient of the squared term.

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APPARATUS AND METHOD

The method consisted of heating the sample in a furnace to a known temperature, dropping it into a copper block in a water calorimeter, and measuring the heat evolved in cooling the sample to equilibrium temperature in the calorimeter. The sample was sealed in a stainless steel capsule having a screw top. The mean heat capacity of the empty capsule and the heat loss by the container during the drop were accounted for by "blank" experiments made with an empty capsule. The temperature of the sample was measured by a chromel-alumel thermocouple.

The essential parts of the apparatus were identical with those described in NEPA-816-SCR-28, with the following exceptions:

- a. Stainless steel was used for the capsules, the advantages over copper being lower density and less corrosiveness.
- b. The furnace temperature was controlled automatically by a Bristol Pyrometer Indicator Controller.
- c. The capsule was suspended in the furnace by a wire, the upper end of which was fastened to a rigidly held brass connection. By turning a screw on the lower end of the connection, the capsule and wire were released, thus allowing them to drop toward the calorimeter.

A typical cooling curve of temperature versus time may be found in Figure 3 of NEPA-816-SCR-28. The maximum calorimeter temperature was obtained by extrapolating the curve back to zero time, the instant of drop.

The experiment was divided into two parts. The first part consisted of the determination of the mean heat capacity of the stainless steel capsule as a function of temperature. This calibration of the "blank" was done in order to provide values of the apparent mean heat capacity of the capsule under

the actual experimental conditions. The second part consisted of the determination of the mean heat capacity of the capsule filled with the beryllium carbide sample, from which data the mean heat capacity of the sample was calculated. An analysis of the beryllium carbide powder is shown in Table 3. Each determination of heat capacity was calculated from the following equations:

$$(C_p)_c, T_{av} = \frac{K_w(T_3 - T_2)}{M_c(T_1 - T_3)} \quad (1)$$

for the empty capsule, and

$$(C_p)_s, T_{av} = \frac{K_w(T_3 - T_2) - (C_p)_c M_c(T_1 - T_3)}{M_s(T_1 - T_3)} \quad (2)$$

for the sample. $(C_p)_c, T_{av}$ was the apparent mean heat capacity of the capsule at T_{av} , $(C_p)_s, T_{av}$ that of the sample at T_{av} , M_c the mass of the capsule, and M_s that of the sample. T_1 was the temperature of the capsule before dropping, T_2 that of the calorimeter at the same time, T_3 the extrapolated maximum temperature of the calorimeter, and T_{av} the average of T_1 and T_3 . K_w was the calculated water equivalent of the calorimeter, defined by the relation

$K_w =$ mass of calorimeter cup x heat capacity + mass of water x heat capacity + mass of immersed portion of stirrer x heat capacity + mass of copper block x heat capacity + mass of immersed portion of thermometer x heat capacity.

A linear equation was fitted to each of the two sets of data. This was of the form

$$C_p = A + BT \quad (3)$$

A, as defined by the least squares method, was

$$A = \frac{\left(\sum_1^N T_i^2 \cdot \sum_1^N (C_p)_i \right) - \left(\sum_1^N T_i \cdot \sum_1^N T_i (C_p)_i \right)}{\left(N \sum_1^N T_i^2 - \left[\sum_1^N T_i \right]^2 \right)} \quad (4)$$

and B, defined similarly, was

$$B = \frac{N \sum_1^N T_i (C_p)_i - \left(\sum_1^N T_i \cdot \sum_1^N (C_p)_i \right)}{\left(\sum_1^N T_i^2 - \left[\sum_1^N T_i \right]^2 \right)} \quad (5)$$

In the equations above, T_i was the temperature in $^{\circ}\text{C}$ of the i th determination, $(C_p)_i$ the i th calculated mean heat capacity in $\text{cal gm}^{-1} (^{\circ}\text{C})^{-1}$, and N was the total number of determinations. The summation $\sum_1^N$ extended over the entire set of calculated values.

The probable absolute errors were found from the following equations:

$$E_1 = \frac{.6745}{\sqrt{(N-1)}} \sum_1^N \left[(\bar{C}_p)_i - (C_p)_i \right]^2 \quad (6)$$

for a single determination, and

$$E_2 = \frac{0.6745}{\sqrt{N(N-1)}} \sum_{i=1}^N [(\bar{C}_p)_i - (C_p)_i]^2 \quad (7)$$

for any point on the least squares curve.

$(\bar{C}_p)_i - (C_p)_i$ was the vertical distance from the i th experimental point to the curve, N was the total number of points and the summation $\sum_{i=1}^N$ extended over all points.

The probable absolute error for the final expressions for the mean heat capacity of the sample was found by taking the square root of the sum of the squares of three separate errors. These were (1) the probable error of the total heat content of the capsule as determined in the "blank" calibration, since this error was inherent in the calculation of the mean heat capacity of the beryllium carbide, (2) the same probable error repeated since it appeared again experimentally in the drops using full capsules, and (3) the probable error calculated from the spread of values around the curve for the mean heat capacity of the sample. Expressed mathematically, the errors were

$$E_3 = \sqrt{\frac{(E_{cM_c})^2 + (E_{cM_c})^2 + (E_{sM_s})^2}{M_s}} \quad (8)$$

for a single determination, and

$$E_4 = \sqrt{\frac{\frac{1}{N} [(E_{cM_c})^2 + (E_{cM_c})^2 + (E_{sM_s})^2]}{M_s}} \quad (9)$$

for any value in the final expression for $(C_p)_s$ as a function of the sample temperature.

E_c was the probable absolute error of a single measurement of the $(C_p)_c$ of steel and E_s that of a single measurement of $(C_p)_s$, the mean heat capacity of the sample. M_c and M_s were, respectively, the masses of the capsule and the sample.

EXPERIMENTAL DATA

Fifty-three calibration runs were made over the temperature range 500°C - 1000°C (932°F - 1832°F) with stainless steel capsules. Of these, thirty-two were made with a thin walled hollow steel capsule, five with a solid steel capsule, and the remainder with a hollow capsule filled with pure Al_2O_3 powder used as a standard substance. The data for these are found in Table I. The best linear equation for the mean heat capacity of stainless steel over the temperature range 50°C - T°C as a function of temperature T °C calculated by the method of least squares, was (See Figure 1)

$$(C_p)_c = 0.119 + 0.169 \times 10^{-4} T^\circ\text{C} \text{ cal gm}^{-1}(\text{°C})^{-1} \quad (10)$$

The probable errors, as calculated from equations 6 and 7 were 0.005 cal $\text{gm}^{-1}(\text{°C})^{-1}$, for a single observation and 0.00064 cal $\text{gm}^{-1}(\text{°C})^{-1}$ for any point on the curve. The error represented by these two values was less than 5%.

Twenty-one determinations were made over the temperature range 400°C - 1000°C (752°F - 1832°F) with the stainless steel capsule filled with beryllium carbide powder. The mean heat capacity of the stainless steel capsule, given in equation 10, was used as a standard for calculating the mean heat capacity values of the beryllium carbide sample. The equation determined by the least squares method using the values in Table II was (See Figure 2)

$$(C_p)_s = (0.299 + 2.16 \times 10^{-4} T^\circ\text{C}) \text{ cal gm}^{-1}(\text{°C})^{-1} \quad (11)$$

where $(C_p)_s$ was the mean heat capacity over the temperature range (50°C - T°C). The percentage of impurities present in the sample is shown in Table III.

The probable absolute errors for the above expression for C_p , were calculated from equations 8 and 9. These were 0.34 cal $\text{gm}^{-1}(\text{°C})^{-1}$ for a single determination, and 0.005 cal $\text{gm}^{-1}(\text{°C})^{-1}$ for any value obtained from equation 12 or from the graph in Figure 2.

DISCUSSION

The mean heat capacity over the temperature range 300°C - 1000°C (572°F - 1832°F) was calculated from a series of heat content determinations. All of the data were evaluated by the method of least squares in order to obtain the best curve of mean heat capacity as a function of temperature.

The probable errors were calculated on the assumption that the deviations obeyed a binomial distribution. No attempt was made to take into account the constant errors of the experiment. It is important to note that the errors involved in the determination of the mean heat capacity of the steel capsule were multiplied in the final determinations on beryllium carbide powder by a factor approximately equal to the ratio of the mass of the capsule to the mass of the sample.

The total error of the final expression for mean heat capacity was estimated to be less than 10%. Contributing factors to this error were the following:

- 1) Errors in the thermocouples and thermometers.
- 2) Errors arising from impurities in the sample.
- 3) Non-reproducible heat losses by radiation and convection during the time of fall of the capsule.
- 4) Errors in the extrapolated maximum calorimeter temperature.
- 5) Errors arising from a change of mean heat capacity of the capsule due to an oxide film formed during successive drops.
- 6) Errors arising from the difference of the true calorimeter water equivalent and the calculated one.
- 7) Errors caused by the heat of stirring. These were probably small in comparison with other errors.

All of the results over the range of temperature 300°C - 1000°C (572°F - 1832°F) may be expressed by the equation

$$(C_p)_{\text{Be}_2\text{C}} = (0.299 + 2.16 \times 10^{-4} T^{\circ}\text{C} \pm 0.005) \text{ cal gm}^{-1}(\text{°C})^{-1} \quad (12)$$

The numerical value of C_p found by this equation is also equal to the heat capacity in BTU lb⁻¹(°F)⁻¹.

TABLE I

TEMPERATURE AND HEAT CAPACITY DATA FOR #303 STAINLESS STEEL

Run Number	Experimental Body	Weight (gm.)	Furnace Temp. T, °C	Mean Heat Capacity (C_p) _c (cal gm ⁻¹ °C ⁻¹)
1	Al ₂ O ₃ in capsule	43.5	506	.119
2	Al ₂ O ₃ in capsule	51.6	508	.125
3	Al ₂ O ₃ in capsule	51.6	524	.120
4	empty capsule	51.5	530	.126
5	empty capsule	51.6	542	.130
6	empty capsule	51.5	552	.127
7	empty capsule	64.9	598	.128
8	Al ₂ O ₃ in capsule	65.5	600	.126
9	Al ₂ O ₃ in capsule	65.5	612	.128
10	empty capsule	64.9	646	.132
11	Al ₂ O ₃ in capsule	65.7	673	.134
12	empty capsule	61.8	681	.137
13	empty capsule	64.9	685	.135
14	Al ₂ O ₃ in capsule	65.7	694	.131
15	empty capsule	61.8	705	.132
16	empty capsule	61.8	710	.133
17	empty capsule	61.8	720	.142
18	empty capsule	61.8	720	.125
19	empty capsule	61.8	720	.138
20	empty capsule	61.8	721	.136
21	empty capsule	64.9	746	.128
22	empty capsule	65.5	750	.129
23	Al ₂ O ₃ in capsule	65.5	752	.129
24	Al ₂ O ₃ in capsule	64.9	752	.125
25	empty capsule	55.1	760	.124

TABLE I (continued)

TEMPERATURE AND HEAT CAPACITY DATA FOR #303 STAINLESS STEEL

<u>Run Number</u>	<u>Experimental Body</u>	<u>Weight (gm.)</u>	<u>Furnace Temp. T, °C</u>	<u>Mean Heat Capacity (C_p) (cal gm⁻¹ °C⁻¹)</u>
26	empty capsule	65.0	845	.143
27	empty capsule	64.9	848	.138
28	empty capsule	65.6	860	.145
29	empty capsule	65.7	860	.132
30	empty capsule	65.0	860	.128
31	empty capsule	54.9	910	.128
32	empty capsule	55.1	921	.130
33	empty capsule	64.3	955	.133
34	empty capsule	55.1	1005	.136
35	empty capsule	54.5	1015	.126
36	empty capsule	46.3	1022	.128
37	empty capsule	42.9	1040	.131
38	Al ₂ O ₃ in capsule	61.8	1042	.152
39	Al ₂ O ₃ in capsule	51.6	1048	.150
40	empty capsule	46.3	1050	.135
41	solid capsule	125.6	1053	.132
42	Al ₂ O ₃ in capsule	48.3	1061	.155
43	Al ₂ O ₃ in capsule	48.8	1065	.140
44	empty capsule	50.5	1071	.142
45	solid capsule	125.8	1075	.132
46	empty capsule	49.5	1076	.144
47	empty capsule	50.5	1095	.145
48	empty capsule	45.9	1105	.133

TABLE I (continued)

TEMPERATURE AND HEAT CAPACITY DATA FOR #303 STAINLESS STEEL

<u>Run Number</u>	<u>Experimental Body</u>	<u>Weight (gm.)</u>	<u>Furnace Temp. T, °C</u>	<u>Mean Heat Capacity (C_p) (cal gm⁻¹ °C⁻¹)</u>
49	solid capsule	127.3	1130	.136
50	empty capsule	42.9	1140	.130
51	empty capsule	44.4	1145	.135
52	solid capsule	126.5	1150	.128
53	solid capsule	127.1	1158	.127

TEMPERATURE AND HEAT CAPACITY DATAFOR BERYLLIUM CARBIDE POWDER.TABLE II

Run Number	Furnace Temperature T_1 , $^{\circ}\text{C}$	Mean Heat Capacity (C_p) _s , cal gm ⁻¹ $^{\circ}\text{C}^{-1}$
1	397	.391
2	403	.403
3	413	.389
4	418	.413
5	412	.396
6	422	.342
7	422	.395
8	604	.448
9	606	.432
10	609	.426
11	609	.415
12	805	.415
13	802	.496
14	805	.459
15	811	.482
16	816	.473
17	816	.439
18	1008	.500
19	1009	.519
20	1011	.540
21	1012	.526

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TABLE III

ANALYSIS OF BERYLLIUM CARBIDE POWDER GRADE 46 MANUFACTURED BY
FANSTEEL

<u>Material</u>	<u>Analysis Before Heating</u> <u>% Total Weight</u>	<u>Analysis After Heating</u> <u>% Total Weight</u>
C (Combined)	32.00	29.7
H ₂ O	6.40	1.7
BeO	9.30	15.21
C (Free)	2.17	2.82
Be ₃ N ₂	1.97	5.11
Insolubles	0.20	0.5
Be (Combined)	48.06	44.6

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Figure 1

Mean Heat Capacity of #303 Stainless Steel

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Figure 2

The Mean Heat Capacity of Beryllium Carbide
Powder Over the Temperature Range 50°C.-1700°C.
as a Function of Temperature T.