Manufacturing of a graphite calorimeter at Yazd Radiation Processing Center

Farhood Ziaie

Abstract In this work, a few quasi-adiabatic graphite calorimeters of different dimensions are described. These calorimeters have been manufactured by ourselves and studied for accurate absorbed dose measurements in 10 MeV electron beam. In order to prove the accuracy and reliability of dose measurements with the use of the self designed graphite calorimeters (SDC), an inter comparison study was performed on these calorimeters and Risø’s graphite calorimeters (SC, standard calorimeter) at different doses by using a Rhodotron accelerator. The comparison shows conclusively SDC of the optimal size, the results agreeing with those obtained with the SC within 1%.

Keywords graphite calorimeters • absorbed dose • radiation processing • self designed calorimeters (SDC) • standard calorimeter (SC) • Rhodotron accelerator

Introduction

High-intensity, high-energy (4 to 12 MeV) electron beams are widely utilized in the radiation processing of a large variety of industrial products. The standardization of these electron beams is a vital facet of the evaluation of the absorbed dose delivered to the product. The desired effects on various materials are achieved only at certain doses, requiring that the dose be well known in order to assure total process completion. Calibration of electron beams in term of depth-of-cure is a necessary part of and overall quality assurance program. Various models of graphite calorimeters have been previously constructed in order to address the issues of industrial electron beam accelerator calibrations [2, 4, 7–9]. These models generally consist of a single graphite disk surrounded by an expanded polystyrene foam for thermal insulation. One version of a previously used NIST-designed calorimeter is shown in Fig. 1. It consists of a small graphite disk with an imbedded thermistor (connected to a digital meter), surrounded by a thick graphite ring in order to provide more realistic side-scatter conditions. Calorimeters of this type can provide detailed information on electron beams and have been successfully used in inter comparisons with the Risø National Laboratory in Denmark and at the National Physical Laboratory (NPL) in the United Kingdom [11]. Calorimeters have been used for making routine and reference dose measurements at electron accelerators for radiation processing [3, 5, 6, 10]. These calorimeters are operating at atmospheric pressure. The temperature is needed to be stable enough to allow a read-out only before and after irradiation, so that no on-line measurement is needed. The calorimeters are usually irradiated by passing them through a swept electron beam on a conveyor system. These calorimeters
are commonly used for 4–12 MeV electrons, under conditions where the calorimetric body is not totally absorbing. In this paper, I present experimental results of a few graphite calorimeters of different dimensions for accurate absorbed dose measurements which were tested with a 10 MeV electron beam.

Accelerator

Irradiations were performed with the scanned beam from a high-energy electron accelerator, Rhodotron TT200 type, at the nominal electron energy of 10 MeV, at beam current 4 mA, and with a scan width of 100 cm, at a scan frequency of 100 Hz. The accelerator was provided with a variable-speed conveyor, to pass the calorimeters or other materials through the swept beam. The distance between the accelerator output window and the conveyor was about 120 cm.

Construction details

The calorimeters were made of high-purity graphite (99.95%, nuclear grade) with a density of 1.70 g/cm³. In this work, several size of disks were made with 2, 3, 9, and 14 cm in diameter and 1.2 cm in thickness. This thickness was about one-third of the electron range for the 10 MeV electron beam that was calibrated. All 4 disks were used for the measurements alone. I also mounted the 2 or 3 cm disks in the middle of the 9 and 14 cm graphite rings (with the same thickness of the disks) for further dose measurements. The disks were thermally insulated from the ring by a nominal 0.5 mm air gap (Fig. 1). Small-expanded polystyrene beads separate the disks from the surrounded rings. The rings were used to contribute side-scattered electrons to the core in the broad-beam geometries. All assemblies were placed in the polystyrene foam as a thermal insulation, at least 4 cm thick on all sides.

Temperature measurement system

The temperature measurement system was a commercial digital read-out unit employing a thermistor sensor. The thermistor was oval in shape, about 1.9 mm in diameter and 4 mm long (model VEC P32A 180, calibrated in Risø). A hole, slightly larger than the thermistor was drilled to the center of the core, 2 mm in diameter and 7 mm deep, and the thermistor was fixed in place. We made sure that approximately no air was trapped in the hole that could reduce thermal conduction. The thermistors used in the calorimeters were of high stabilities. The temperature correlation as a function of thermistor resistance over the temperature range of 0°C to 60°C were determined as [11].

\[
T = B(\ln R + A)^{-1} - T_K
\]

where \( R \) is the measured thermistor resistance in ohms. The constant values A, B and \( T_K \) for the used thermistors and coded in the Risø National Laboratory are listed in Table 1.

### Experimental

In this work, 7 types of calorimeters called the self designed calorimeters, SDC, were irradiated under 10 MeV electron beam on the conveyor with 260–640 cm/min speed. The calorimeters are labeled with two numbers in parenthesis such as SDC(\( n_1, n_2 \)) which indicate the core and ring diameters in cm, respectively. Figure 2 shows a typical recording of the calorimeter temperature increase as a function of time. The points labeled \( T_1 \) and \( T_2 \) indicate the temperatures at the start point and the end of irradiation times. Periods I, II, and III show a slow temperature drift before irradiation, a rapid increase during irradiation, and a slow cooling after irradiation, respectively. To limit heat losses during irradiation, the electron beam irradiation period should be short in comparison to the thermal decay constant. The absorbed dose in each disk is calculated by [1, 4].

\[
D = \Delta T \cdot C_g
\]

where \( D \) is the absorbed dose in Gy, and \( \Delta T \) is the change

![Fig. 1. A prior model of a graphite calorimeter used for studies of electron accelerators.](image1)

![Fig. 2. Typical recording of the calorimeter temperature increase as a function of time.](image2)

<table>
<thead>
<tr>
<th>Type</th>
<th>Code</th>
<th>A</th>
<th>B</th>
<th>( T_K )</th>
</tr>
</thead>
<tbody>
<tr>
<td>SDC</td>
<td>1114</td>
<td>5.911</td>
<td>4536.89</td>
<td>315</td>
</tr>
<tr>
<td>SC</td>
<td>1015</td>
<td>5.9779</td>
<td>4582.23</td>
<td>317</td>
</tr>
</tbody>
</table>

Table 1. Constant values in eq. (1) as used for thermistors, and coded in Risø [11].
in temperature at the midpoint of the irradiation period \((T_z^{'2} - T_z^{'1})\).

The value of \(AT\) is determined by extrapolation of the temperature drift rates before and after irradiation to the midpoint of the irradiation period. This method of dose calculation by temperature midpoint analysis compares very well with the more rigorous methods of irradiation calculation by temperature midpoint analysis compares the midpoint of the irradiation period. This method of dose calculation by temperature midpoint analysis compares very well with the more rigorous methods of irradiation by use of the equal-area triangle method [8]. The specific heat capacity, \(C_g\), of the graphite used for the disks and rings construction were determined as a function of temperature from equation (1).

\[
(3) \quad C_g = 644.2 + 2.86 \times T \quad [J/(kg\cdot°C)]
\]

For any given calculation involving the specific heat capacity, the average temperature during the irradiation period (i.e., \((T_{max} + T_{min})/2\)) is used to determine the appropriate value to use in subsequent dose calculation. The beam uniformity for the accelerator was measured by irradiating radiochromic films with a scanning spectrometer. The electron beam had a good uniformity, being uniform within 2% over the diameter of the calorimeter disk [12]. Before each irradiation the calorimeter bodies were left to reach a heat equivalent temperature as the irradiated environment. According to the given references, the calorimeter temperature should approximately be constant for about 10 min before irradiation (typically, the change is less than 0.1°C) [1, 9].

In order to have a reference value for dose comparison, during the experiment for each of the self designed calorimeter, a standard calorimeter, SC, made by the Risø National Laboratory was irradiated simultaneously. For getting a reliable result, more than 50 irradiations were done and standard deviations were calculated and compared.

**Results and discussion**

The absorbed dose to the calorimeter core in practice, \(D_c\), is calculated from equation given in [11]:

\[
(4) \quad D_c = (T_z^{'2} - T_z^{'1} - T_a) \cdot C_g \cdot K_1 \cdot K_2
\]

where \(T_z\) and \(T_a\) are the measured temperature before and after irradiation, respectively. \(T_a\) is a compensative heat transfer parameter of the conveyor and accelerator structure already measured to be 0.35°C. \(C_g\) is the specific heat of graphite. \(K_1\) is a correction factor for the change of temperature as a function of time after irradiation where no extrapolation is used. \(K_2\) is the calibration factor derived from inter-comparisons with reference dosimeters (for the used SC it is 1.03 measured, in Risø). In each measurement, the ratio of Dose (SDC)/Dose (SC) was calculated and the standard deviation was found by using the formula:

\[
(5) \quad S_{n-1} = \sqrt{\frac{\sum (x_i - \bar{x})^2}{n-1}}
\]

where the \(x_i\) is the Dose (SDC)/Dose (SC) ratio, \(\bar{x}\) is the average value over the \(x_i\)'s, and \(n\) is the number of measurements. Also the relative uncertainty, \(RU\), for each SDC are calculated using the formula:

\[
(6) \quad RU = \frac{S_{n-1}}{x} \times 100 \quad [%]
\]

Table 2 shows the results derived from measurements done by using the SDCs. In eq. (4) the coefficient \(K_1\) is defined as the ratio of dose calculated by extrapolating the temperatures up to the midpoint of the irradiation time \((T_z)\), to the non-extrapolated dose value using the measured temperature after irradiation \((T_z)\). Then, due to the heat loss in the calorimetric body after irradiation, \(K_2\) values will increase as a function of time. Figure 3 shows the variation of \(K_1\) coefficient values as a function of time after irradiation for the SDC(3,14), using the linear extrapolation. To determine the \(K_2\) value, the FWT-60 film dosimeters were used as a reference dosimeter. This film dosimeter was put on the surface center of the SDC(3,14) graphite disk and was irradiated by 10 MeV electron beam using the conveyor system. The average dose ratios calculated by the used SDC and FWT-60 film dosimeters were considered as \(K_2\) coefficient (Table 3).

**Conclusion**

As shown in Table 2 by comparing the results of the SDC(2,0), SDC(3,0), SDC(9,0), and SDC(14,0), one can

\[
\text{Fig. 3. Variation of } K_1 \text{ as a function of time after irradiation for SDC(3,14), using linear extrapolation.}
\]
conclude that increasing the core diameter causes a decrease of the relative uncertainty in the measured dose values. This is due to a decrease of heat capacity of the absorber, graphite disks, and dropping down of the heat loss rating from the disks, consequently.

On the other hand, comparison between SDC(2,9) and SDC(2,0), SDC(3,9) and SDC(3,0), and SDC(3,14) and SDC(3,0), shows the effect of the graphite ring or jacket on the decrease of the relative uncertainty in the measured dose values, which, in turn, are due to a better semi-adiabatic condition. We can also conclude that the graphite ring with higher diameter, SDC(3,14), gives the best result comparing to the other rings. Also the comparison of SDC(3,14) and SDC(14,0) shows the air gap effect in reducing the heat loss and more heat stability in SDC(3,14) core, where the heat capacity is the same as with the SDC(14,0) one.

Table 3. Measurement results using SDC and FWT-60 film dosimeters simultaneously and $K_2$ coefficient calculation.

<table>
<thead>
<tr>
<th>No.</th>
<th>SDC dose (kGy)</th>
<th>FWT dose (kGy)</th>
<th>$K_2 = \text{Dose (FWT)/Dose (SDC)}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>4.89</td>
<td>5.11</td>
<td>1.05</td>
</tr>
<tr>
<td>2</td>
<td>8.11</td>
<td>8.34</td>
<td>1.03</td>
</tr>
<tr>
<td>3</td>
<td>12.29</td>
<td>12.49</td>
<td>1.02</td>
</tr>
<tr>
<td>4</td>
<td>21.44</td>
<td>21.90</td>
<td>1.02</td>
</tr>
<tr>
<td>5</td>
<td>26.38</td>
<td>27.51</td>
<td>1.04</td>
</tr>
</tbody>
</table>