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a Vacuum-Type Microcalorimeter

for Synchrotron-Radiation Measurements

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ABSTRACT

A previously developed total-absorption calorimeter was improved by replacing a thermostated air bath by a vacuum chamber in order to reduce the applicable photon energy up to several keV, and to extend the measurable photon intensity up to about 10 W of synchrotron radiation. The calorimeter maintained linearity over the power range from about 10 μW to a few W within 0.1%. Experiments with monoenergetic and white-spectrum photons have proven the designed performance, in which the heat power due to monoenergetic photons was measured within an uncertainty of 3%, and the heat power due to white-spectrum photons was up to about 2 W.

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1 Introduction

In a recent issue of this journal the present authors presented a new calorimeter which can be applied to the measurements of the absolute intensity of synchrotron radiation from 10 to 50 keV, having an intensity of 1 μW to 3.8 mW[1]. Although the calorimeter has been widely used for measurements of synchrotron radiation, the rather large thermostated air bath was a big barrier for applications to new fields, which was essential to accurately measure any subtle temperature change of the beam absorber. Besides, air of about 10 cm thickness in a thermostated air bath makes the application impossible for energy photons of less than 10 keV because of a large attenuation of the photon intensity.

Therefore, a vacuum-type calorimeter was developed by removing the thermostated air bath in order to facilitate applications and to extend the measurable range up to 10 W. In this paper, the characteristics and performance of the improved calorimeter are mentioned along with the experiments conducted for monoenergetic and white-spectrum photons at the Photon Factory of the National Laboratory for High Energy Physics.

2 Characteristics of the calorimeter

The basic principle in measuring the heat power due to photons is the same as that of the previous calorimeter. Namely, the present calorimeter comprises twin calorimetric sensors of a synchrotron-radiation beam absorber and a thermomodule along with a heat sink, which are placed in a vacuum chamber (Fig. 1). This is a twin-type heat-flow total-absorption microcalorimeter based on isothermal calorimetry[2]. The relation between the heat power ($P(\mu W)$) and the temperature (T_c(K)) of the beam absorber is given by

$$P = C(dT_c/dt) + k(T_c - T_e), \tag{1}$$

where
  C : heat capacity of beam absorber(J/K),
  k : coefficient of heat transfer of thermomodule(W/K),
  T_e: temperature of heat sink(K).
Since T_e is maintained constant for any input heat power in isothermal calorimetry, the thermal energy ($Q(\mu J)$) can be obtained by integrating the T_c-vs-time curve until T_c has returned to its initial value,

$$Q = \int P dt = k \int (T_c - T_e)dt. \tag{2}$$

The average photon intensity (N (photons/sec)) of energy (E (keV)) absorbed in the calorimeter is calculated from the thermal energy (Q) measured during Δt (sec) by

$$N = 6.242 \times 10^9 Q/E/\Delta t. \tag{3}$$

As shown in Fig. 1, photons are led through a 15 μm Be window to a vacuum chamber, in which the air pressure is kept at less than 10^{-2} Pa so as to make the attenuation less than 0.01% for 1-keV photons due to the air. All of the photon energy is absorbed in an Ag beam absorber of 17.32 J/K heat capacity. The Ag absorber has a cup shape in order to reduce the energy loss due to back-scattered photons and fluorescent-photon leakage. A thickness of 2 mm for the bottom of the Ag cup was determined such that 90-keV photons would be absorbed above 98%. The percentage energy loss due to the photons

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was estimated using the EGS4 code[3] above 10 keV and Greening's equation[4], as shown in Fig. 2, which is within 2% for photons up to about 90 keV.

The heat due to photon absorption in the beam absorber flows through a thermomodule made of compound semiconductors ($Bi_2Te_3 + Sb_2Te_3$ for p-type versus $Bi_2Ti_3 + Bi_2Se_3$ for n-type) into an Al heat sink of about 16 KJ/K heat capacity, and produces an electromotive force (EMF) at a rate of 53.34 mV/K due to the Seebeck effect. The heat loss from the beam absorber by thermal radiation is at most 0.01%, since the increment of the temperature of the sensor is less than 1 K for supposed maximum input powers.

The net EMF from a calorimetric sensor is obtained by subtracting the EMF background of another sensor. The background level of the EMF was 5±3 μV in the present calorimeter, which is equivalent to the absorbed energy of about 3 μW. This background level is fairly larger than 0.15±0.01 μV in the previous calorimeter, showing that the vacuum chamber is inferior to a thermostated air bath for regulating the ambient temperature around the calorimeter.

Finally, the EMF output is converted to heat power using a calibration curve obtained electrically with Joule heat from a resistance of 100.0 ± 0.1 Ω reeled around the beam absorber. A calibration curve measured using a Joule-heat method from 16 μW to 1 W is shown in Fig. 3. The conversion factor determined by a least-squares fitting method was 1.5122 ± 0.00156 V/W. It guarantees that the present calorimeter maintains linearity within the power range from about 10 μW to a few W with an accuracy of 0.1%.

3 Monoenergetic synchrotron-radiation measurements

3.1 Experimental

The feasibility of the calorimeter was examined for monoenergetic photons of 8, 10, 15, 30 and 40 keV from the vertical wiggler beam line (BL-14C) [5] in the 2.5-GeV Synchrotron-Radiation Facility, the Photon Factory. The experimental arrangement in the shield hutch located in BL-14C is shown in Fig. 4. Monoenergetic photon beams are produced using a monochromator of Si(111) double crystals at 34 m distant from the light source, by which monoenergetic photons from 7.5 to 43 keV become available. The typical photon intensity was between 38.1 μW at 8 keV and 353 μW at 30 keV, which corresponded to fluence rates between 3.0×10^7 and 7.3×10^7 photons/sec, in which the typical energy resolution was about 60 eV at 30 keV. The beam size was changed using a collimator and a slit prepared at the exit of the beam line.

Photon beams were introduced into the absorber for between 3 to 10 minutes in order to obtain the absorbed energy above a few mJ. The incident beam intensity during the experiments has been relatively monitored using a free air ionization chamber[6] placed in front of the calorimeter, which was calibrated using the previous calorimeter[1].

Higher harmonics from the monochromator, which would affect on the results of the calorimeter significantly, were also monitored by measuring the scattered-photon spectra at 90 degrees from a Kapton foil of 80 μm thickness placed in a vacuum chamber using a HP-Ge detector. The central angle of higher harmonics in the Bragg-reflection curve slightly shifts from that of the fundamental wave due to the refraction effect, and its angle width is narrower than that of the fundamental wave. Therefore, its contribution was reduced to less than 0.1% during the experiments by detuning the angle of the crystals of the monochromator.

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3.2 Applicability for monoenergetic synchrotron-radiation

The output of the calorimeter for 8-keV photons is demonstrated in Fig. 5, where the output increases with the incident photon beam for 300 sec. and returns to the background level within about 30 min. after the incidence of the photon beam is stopped. The heat power is measured as 38.1 μW for about 40 min. by sampling the output with a personal computer at intervals of 0.1 sec. Similar experiments (12 times) were repeated while changing the beam energy, as summarized in Table 1. In order to investigate the reproducibility and reliability, we also show in this table the ratio along with the intensity measured by a free air ionization chamber. Corrections with linear attenuation coefficients[7] were made in order to obtain the ratio for the attenuation with air, Be and Kapton between the monitor and the beam absorber. The correction of attenuation was about 10% due to the air for 8-keV photon; the others were negligible. After the correction, the measured values under various photon energy and intensity agreed within $\pm 3\%$ with the monitor, and the dispersion was 1.7×10^{-2}.

4 White synchrotron-radiation measurements

4.1 Measurements of a high-power beam

It is impossible to obtain a heat power greater than mW using monoenergetic photon beams in the Photon Factory. The white spectra from the BL-14C beam line were, therefore, used to test the feasibility of the calorimeter for measurements of a few mW to W. Photons with a white spectrum are generated by a vertical wiggler operated in the 3-pole 5-Tesla mode with an electron energy of 2.5 GeV[8]; the spectrum is accurately estimated using the calculational code STAC-8[9] developed for the shielding design of a synchrotron-radiation facility. In Fig. 6, the theoretically calculated spectrum is indicated by a solid line. Actually, the 37 m beam line to the experimental hutch has a couple of permanent filters (1.67×10^{-1} g/cm^2 Be, 5.37×10^{-4} g/cm^2 He and 4.035×10^{-3} g/cm^2 Al), which greatly attenuate the lower energy component of the spectrum. Consequently, the spectrum available for the calorimeter is indicated by the dotted line in Fig. 6.

In the white synchrotron-radiation experiments, the free air ionization chamber shown in Fig. 4 was removed from the experimental arrangement, since the intensity of white synchrotron-radiation was too strong to be monitored by the ionization chamber. Instead, a HP-Ge detector was utilized to monitor the relative intensity by measuring scattered photons into 90 degrees with a Kapton thin foil of 80 μm thickness arranged in a vacuum chamber, where the scattered photons were collimated by a lead collimator of 0.5 mm diameter and guided through a vacuum pipe of about 80 cm length to the detector.

Measurements were repeated for the beam power from 3 mW to about 2 W by changing the beam size. The results are summarized in Table 2, in which the ratio represents the variation from the average of the relative intensity measured by the HP-Ge monitor. The variation of the measurements is within almost $\pm 10\%$, and the dispersion is 4.1×10^{-2}. This larger uncertainty compared to that for monoenergetic photon beam comes from the poor monitoring device, in which the monitored photon intensity is slightly dependent on the beam size and position bombarded in the Kapton foil because of the variety of photon beams and of the foil thickness.

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4.2 Application to measurements of the white synchrotron-radiation intensity

Another experiment was carried out while eliminating the lower energy component of the incident photon beam by Al absorbers. As shown in the spectra calculated with the STAC-8 code of Fig. 6, the spectra of the lower energy component are easily changed by Al absorbers. The heat powers for the white spectra with various Al absorbers were measured by the calorimeter, while they were calculated from the spectra of the STAC-8 code using the following equation:

$$P_{cal} = A \int E\phi(E)dE, \qquad (4)$$

where

A : conversion factor (Watt/keV),

E : photon energy (keV),

$\phi(E)$: incident photon intensity of energy E calculated by STAC-8
: (photons/keV/mA).

A comparison between the measured and calculated heat powers is demonstrated in Fig. 7, where the calculated values are normalized to the measured one without an Al absorber. Both heat powers were in good agreement within about 5% for all of Al thicknesses, though the spectra were changed with the Al absorbers, as shown in Fig. 6. This result suggests that the STAC-8 code could be available for estimating the synchrotron-radiation spectra, while the present calorimeter is applicable for the measurement of an intense photon beam from synchrotron radiation.

5 Concluding remarks

A twin-type total-absorption calorimeter was improved in order to measure the absolute intensity of the photon beam of synchrotron radiation from a few keV to about 90 keV using a vacuum chamber instead of a thermostated air bath. It was shown that a heat power of 10 μW up to a few W could be measured with an accuracy of 0.1% from the calibration with Joule heat, except for the energy escape error. Experiments with monoenergetic synchrotron-radiation beams from 8 up to 40 keV verified that absolute photon intensity was measured within ±3% under various conditions. With white synchrotron-radiation measurements, it was demonstrated that the calorimeter was applicable with good accuracy for measurements of the beam power up to about 2 W.

Acknowledgments

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Table 1. Intensity of monoenergetic SR measured using the calorimeter
(Ratio: $\bar{x}$=1.000, σ^2=0.017)

Energy[a] (keV)	Current[b] (mA)	Beam Size[c] (mm^2)	Power[d] (μW)	Ratio[e]
40	287	4.5×11.5	305.	1.006
40	282	4.5×11.5	292.	0.996
30	273	6.0×11.5	316.	0.995
30	270	6.0×11.5	353.	1.019
15	262	10.5×11.5	179.	0.978
15	258	10.5×11.5	187.	0.987
10	252	10.5×12.0	94.5	0.978
10	248	10.5×12.0	86.6	0.978
10	245	10.5×12.0	93.2	1.010
8	237	11.0×12.0	40.6	1.029
8	234	11.0×12.0	39.4	1.017
8	231	11.0×12.0	38.1	1.007

[a]Photon energy.

[b]Storage ring current.

[c]Photon beam size.

[d]Calorimeter output.

[e]Ratio of the mersurements to the monitor.

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Table 2. Intensity of white SR measured using the calorimeter
(Ratio: $\bar{x}$=1.000, σ^2=0.041)

Current[a] (mA)	Beam size[b] (mm^2)	Power[c] (mW)	Ratio[d]
315	28.0	2180.	0.938
311	28.0	2220.	1.013
308	28.0	2290.	1.043
305	28.0	2060.	1.088
302	28.0	2060.	1.086
298	18.8	1350.	1.059
296	18.8	1304.	1.052
290	18.8	1262.	1.059
286	18.8	1224.	1.064
269	3.142	249.1	0.982
267	3.142	249.2	0.985
252	3.142	234.6	0.957
355	3.142	338.4	0.969
279	0.785	3.069	0.898
275	0.785	3.428	0.899
272	0.785	62.31	0.958
271	0.785	61.58	0.958

[a]Storage ring current.
[b]Photon beam size.
[c]Calorimeter output.
[d]Ratio of the mersurements to the monitor.
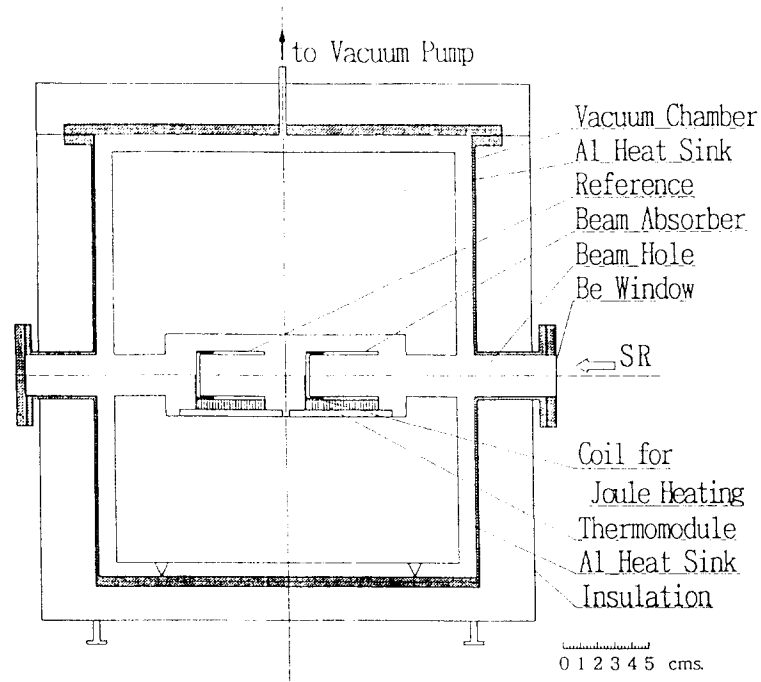
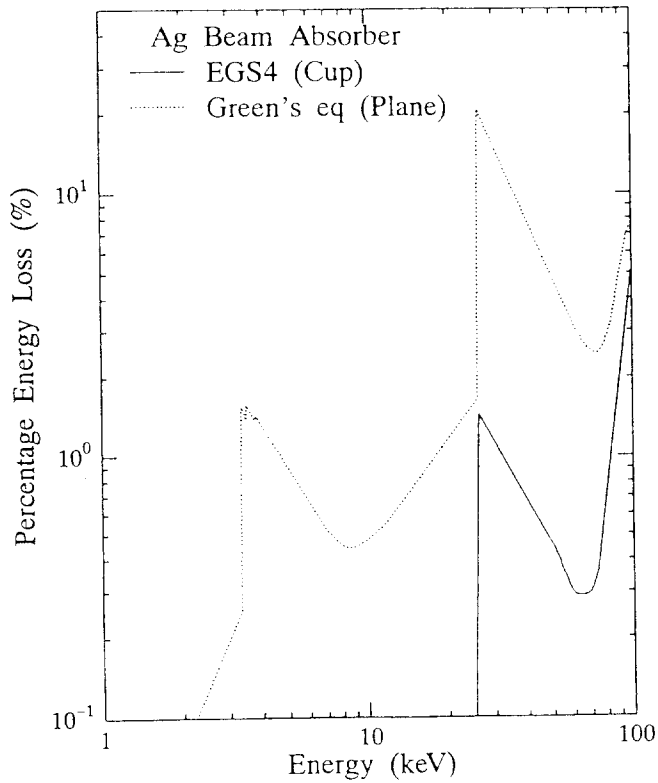Fig. 1. Cross-sectional view of the twin-type total-absorption calorimeter assembly.

Fig. 2. Energy escape with photons from Ag beam absorbers. The solid line represents the
value for a cup-shaped absorber calculated with the EGS4 code, and the dotted line
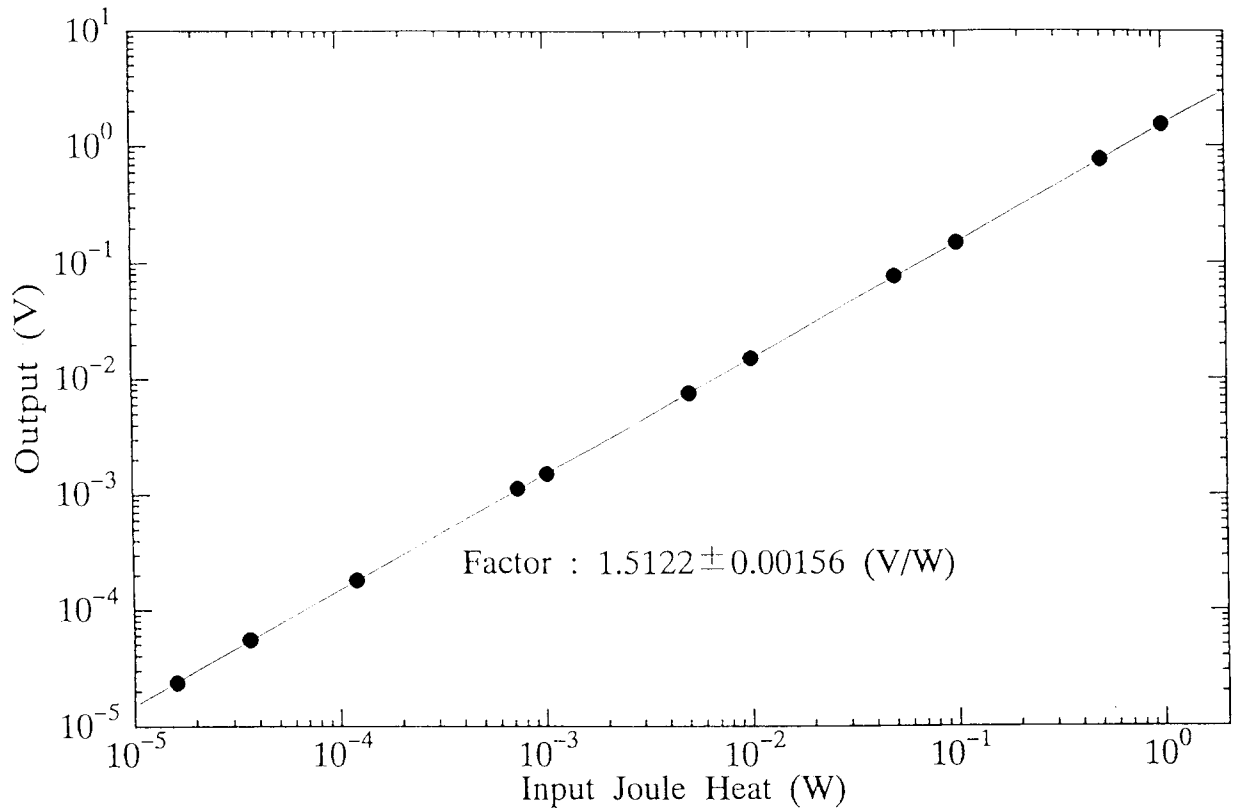for a plane one with Greening's equation.[1]

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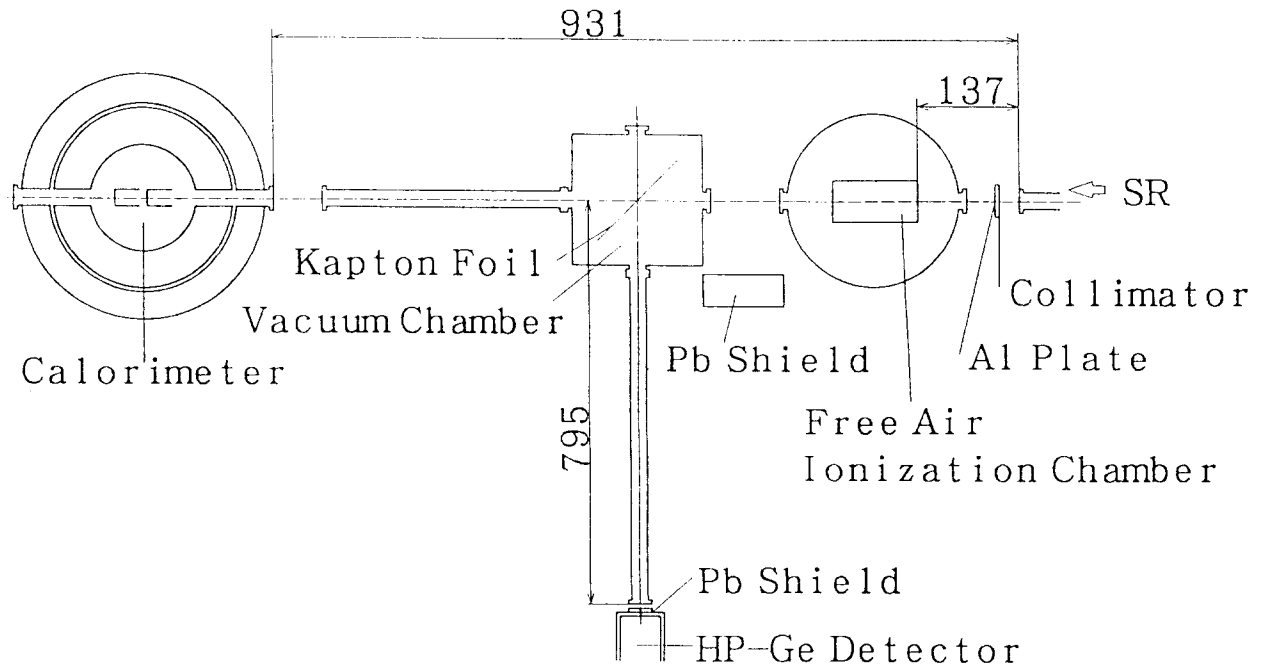Fig. 3. Calibration curve from output to heat power measured with Joule heat.

Fig. 4. Experimental configuration to measure the absolute intensity of synchrotron-radiation beams at BL-14C of the Photon Factory.
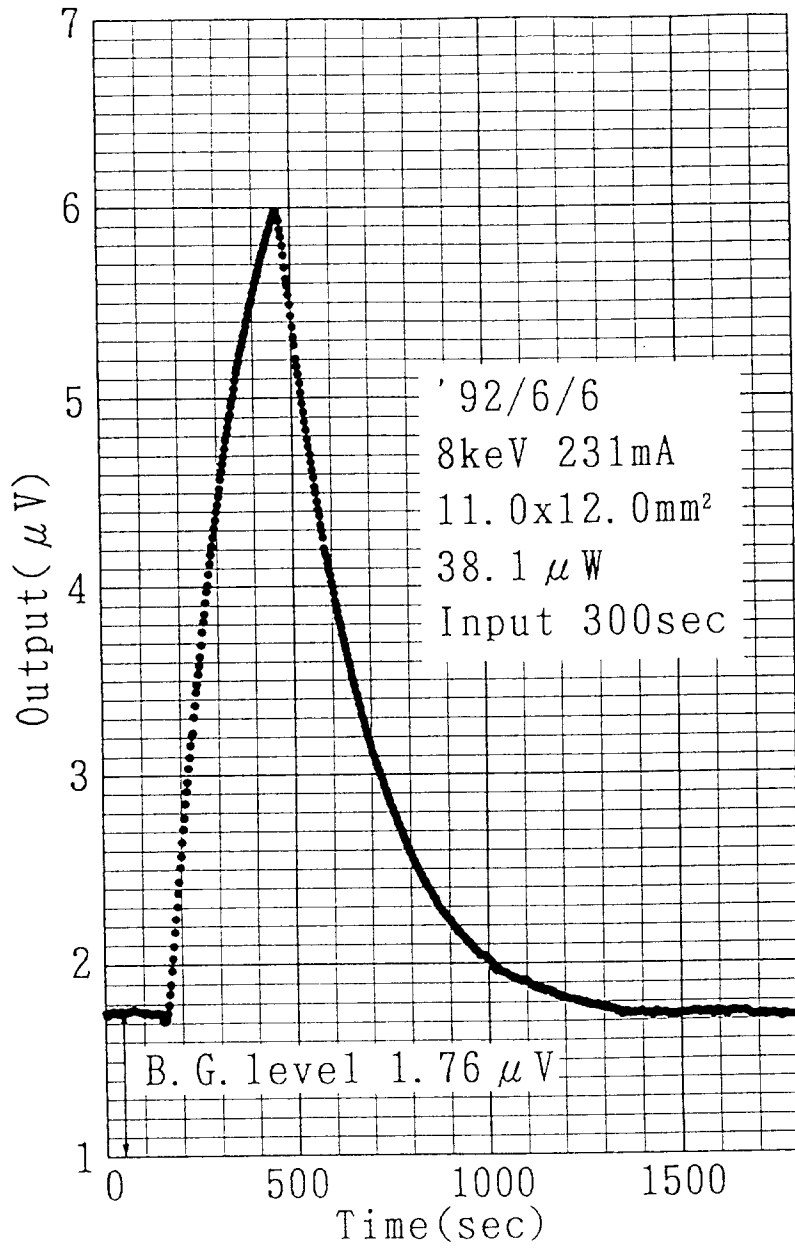
Fig. 5. Measured calorimeter output for monoenergetic synchrotron radiation of 8 keV.
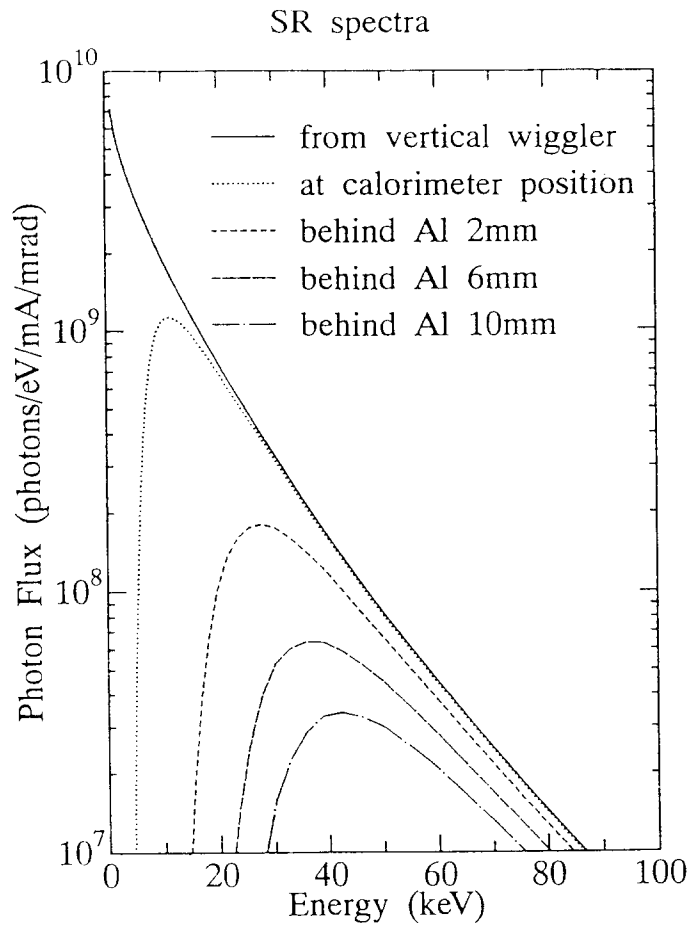
SR spectra

Fig. 6. Calculated spectra with the STAC-8 code of white synchrotron radiation generated by the vertical wiggler of the BL-14C, and led to the shield hutch through filters in the beam line. Synchrotron-radiation spectra attenuated by Al plates are also shown in the figure.
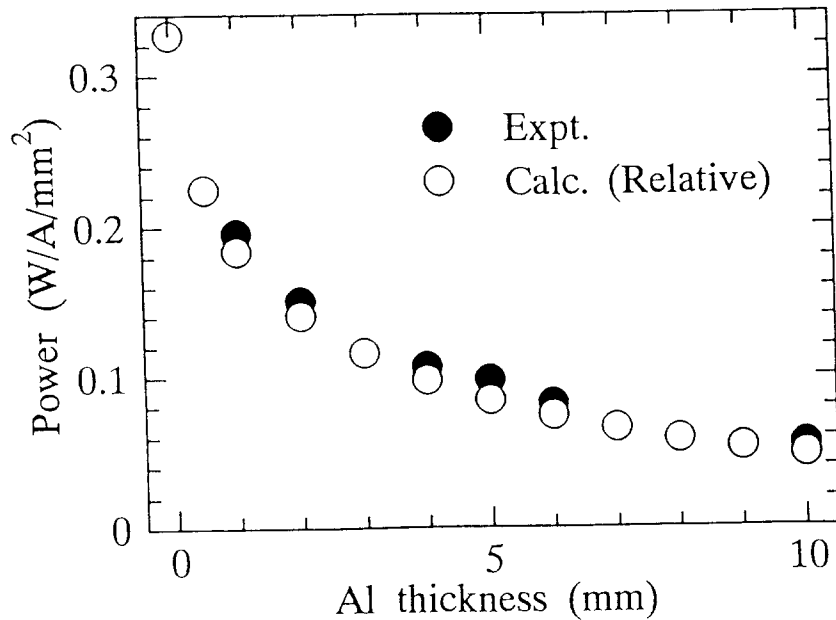

Fig. 7. Heat powers of white synchrotron radiation attenuated by Al absorbers. The solid circles represent the values measured with the calorimeter, and the open circles the values calculated by the STAC-8 code. The calculations are normalized to the measured value without Al absorbers.