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PROPERTIES OF RUTHENIUM OXIDE RESISTORS AS SENSITIVE ELEMENTS OF COMPOSITE BOLOMETERS

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ABSTRACT

The performance of Ruthenium oxide termistors on sapphire as bolometers for particle detection around 100 mK is investigated.
It is shown that the modest pulse height of the signals is due to a reduced thermal responsivity in pulse conditions with respect to the zero frequency value and not to large effective capacitance of the bolometer.

Indeed, the effective thermal capacitance of the RuO_2, measured in pulse conditions with time constant up to tens of milliseconds is compatible with only Debye and electronic contributions while nuclear terms do not contribute.

The low pulse response is attributed to poor thermal diffusivity in the inhomogeneus structure of the thick film sensor.

Comparison with the performance of a sapphire-germanium bolometer shows also limits due to device noise in the energy resolution of the RuO_2 bolometer.

The low thermal capacity in dynamic regime of materials with high nuclear moments support the feasibility of high resolution special bolometers made with materials so far excluded.

I - INTRODUCTION

In the last years the use of ruthenium oxide thick film resistors (TFR) as low temperature thermometers was successfully introduced. (1,2,3).

The merits of these components and the relative easiness and simplicity of construction of ruthenium oxide resistors on a variety of different materials as substratum suggested to investigate the performance of devices of this kind as bolometers for particle detection.

The study of the pulse behavior of a bolometer made of a material that, due to nuclear quadrupole interaction of ruthenium nuclei has, at low temperature, a very high static specific heat is not a secondary reason of interest.

If, due to the long relaxation time, the effective thermal capacity in pulse regime is insensitive to the huge nuclear term, it is possible to devise experiments with bolometers made of materials like aluminum, indium or rhenium that have each a peculiar interest. At the moment we are interested in studying at high resolution the beta decay of Re^{187}, that has the lowest Q value in Nature.

Preliminary test made using commercial TFRs deposited on an alumina substratum as sensors for small composite bolometers gave encouraging results in the 100-700 mK temperature range (4).

Recently, a composite sapphire- RuO_2 bolometer was studied by Chapellier et al (5)

To investigate further the performance limits of this kind of device, expecially with reference to the modest pulse size observed, Ruthenium oxide resistors 1x1 mm^2 wide, 15mm thick, were built on a 3X4x.5 mm^3 sapphire substratum; evaporated Aluminum pads were provided for electrical connections.

The components used to produce the TFRs were:
Du Pont QP 873 (1 kΩ/❑), 874 (10k Ω/❑), 875 (100kΩ/❑) and 876 (1MΩ/❑) to obtain, after a standard thermal process resistors of 1k, 10 kΩ, 100 kΩ, 330 k Ω(*). For sensors comparison a reference device was built by substituting the TFR sensor with a glued germanium thermistor (Cryo Cal.Inc mod. CR30E) on one sapphire crystal.

In section II results of the investigations on the I-V vs T relationships and on thermometric sensitivity are reported. In section III the pulse response to alpha particles is analyzed and thermal capacities and thermal impedances are deduced.

II - THERMOMETRIC SENSITIVITY

The resistance temperature relationship of Ruthenium oxide resistors on a sapphire substratum made with xxx Du Pont QP 873 874 875 and 876 was determined at the zero power limit in the range between 50 and 600 mk.

The data, reported in Fig. 1 were obtained as follows:

The samples were kept in tight thermal contact with the bulky copper sample-holder of an Oxford TL 200 dilution cryostat; the thermal conductance of the link was higher then 10^6W/K.

The sample-holder base temperature was measured with a calibrated carbon resistor that was periodically checked by Co^{60} Nuclear orientation Thermometry.

A .5 hertz square wave generator and a 5210 Eg&g lock-in amplifier were used to measure at each temperature point the current for 20 increasing values of the applied voltage. Power dissipation never exceeded 10^{-13} Watts.

The zero power resistance R^o vs T, extrapolated by a least square fit is reported in Fig. (1).

A reasonable good fit for $R^o(T)$ is obtained with the law:

1) $R^{o}(T) = R^{oo} * \exp (T^{o}/T)^{1/2}$

We have T^{o}=13.4 K. for 10kΩ/❑ material T^{o}=35.5 K. for 100kΩ/❑ materials

Sensitivities of lower resistivity material (1kΩ/❑ (QP873) are unsatisfactory and resistances of 1MΩ/❑ are too high; accordingly these samples were discarded.

The thermal responsivity $A^{o} = -d \log R^{o}/d \log T$ resulting from the zero power data for the 10 K samples and for the 100 K samples are reported in Fig. (2) and compared with the responsivity of a germanium thermistor ( ). However, deviations from the ohm law due to field effects are evident in the lnR vs Vb plot reported in Fig. (3).

Such an effect, that reduces the thermal responsivity of the device at normal operating conditions, is expected on the basis of hopping conduction models (6,7).
A reasonable fit to the data, for low values of Vb and T> 50mK, is given by:

$$R(V,T) = R^{o}(T) * \exp(-a*Vb/T) \qquad (a)$$

and the thermal responsivity of the device is reduced according to the formula:

$$A = -d\log R^{o}/d\log T + a*Vb/T.$$

At 80 mK we have a = 3.5 10^{2}K/Volt.

Anyway, for the highest value of Vb applied, this approximation is not valid and direct measures are unreliable because heating effects cannot be excluded.

Data were then numerically corrected assuming, according to the model of B. Abels, p. Sheng, M.D. Coutts, Y.Arie (6,7) for granular metal film conduction:

$$\frac{R(V,T)}{R^{o}(T)} = \exp\left(1/\varepsilon^{*}\right) \int_{-1/\varepsilon^{*}}^{\infty} \frac{z \exp(-z)\, dz}{\left[1 - \exp\left(-z/\left(z+1/\varepsilon^{*}\right)*\varepsilon^{*}/T^{*}\right)\right]} \qquad b)$$

where

$$\varepsilon^{*} = \varepsilon/\varepsilon_{c} \qquad T^{*} = kT/c_{o} \qquad R_{o}(T) = R_{oo} \exp\left[2\,(c_{o}/kT)\right]$$

that in the weak field limit coincides with (a).

The constant C and ε where determined by the low power dissipation experimental data and the weak field expression.
For the 10K samples it results: ε* + 2.2 volt/cm and C^{o} = .32 meV.

III - SINGLE PARTICLE RESPONSE

The pulses produced by the absorption of alpha particles in the bolometer with a loose thermal link to the heat sink where recorded with a 2430 Tektronix digital scope and are reported in Fig. 4 a,b,c.

Digital averaging was applied to reduce the effects of electronic noise.

The alpha flux, from a mixed nuclides source with three main alpha lines at 5.15, 5.48, 5.81 MeV was reduced to less then 1 alpha/sec.

A number of measures was made with a constant voltage biasing configuration (Fig 5), that, eliminating the integrating effects of parasitic capacitance of the input connections, allows us to examine the fast time structure of the signals with ambient temperature electronics.

A 200 μsec risetime was measured, almost independent on temperature that was attributed to a thermal impedance between the sapphire and the ruthenium oxide thermal capacities.

However at the lowest temperatures and with loose thermal links the constant voltage configuration cannot be applied because of instabilities due to thermal feedback.

For this reason biasing through a load resistor Rl was more extensively used.
Measurements were made with three different setups, a, b, c, for the thermal link to the heat sink; the thermal impedance is mainly due to Kapitza resistance between the bolometer and his mechanical support.

The three setups are reported schematically in Fig. (6a,b,c) together with the corresponding concentrated constant models.

A reasonable good fit of the pulse decay is obtained with a single decay time constants of about 2 msec when the thermal link is connected directly to the sapphire (setup a).

When the device is supported, with a different holder, through the ruthenium oxide resistor (setup b) the slope is well approximated by

$$V(t)=V^{\circ}[A\exp(-t/\tau_1)+B\exp(-t/\tau_2)]$$

as expected for model b; suspending the bolometer with the superconducting Aluminum wires of the electrical connections, time constants of the order of one second were obtained (setup c).

The equivalent thermal conductance Geq =dW/dT between the bolometer and the heat sink was determined in each case from the static V-I load curves corrected from the field effect by eqz(b).

Setup a and setup b Pulse height distributions were recorded with a multichannel analyzer.and an RC-RC filter with 200 sec time constants.

IV - DATA ANALYSIS

The averaged pulse shape obtained from alpha particles with setup a and b were compared with the impulse response computed, by means of a standard simulation program for electric circuits, for the corresponding concentrated constant models of Fig. 6 a, b.

The evaluation of the Debye thermal capacity of the sapphire C_s and, for setup b of the glue mass C_g was based on literature data (8).

The impedance toward the heat sink is identified by the measured values of Geq.

The total thermal capacity of the bolometer Cb, that fits the pulse decay times is then determined with an estimated accuracy of about 20% for the lowest values.

The values of Cb so determined are independent on the efficiency of fast thermalization processes of the incoming energy and results obtained for setup a and setup b coincide within errors, if the added thermal capacity of glue is taken into account. In Fig. (7) the data referred to setup a and c are shown.

The quite low values of the thermal capacity obtained with setup a and the 2msec thermal decay time are compatible with the computed Debye and electronic contribution (lower line in Fig. (7)).

Data taken with setup c, with about 1 sec. decay time, give much higher values of C_b indicating the presence of large contributions whit long relaxation times.

They can be attributed mainly to the large nuclear quadrupole specific heat of Al_2O_3 (5) and to contribution from the RuO_2 (9). (Upper line in Fig. 7). The central line in Fig. 7 indicates the static thermal capacitance of the RuO_2 with the static nuclear quadrupole interaction included.

The impedance Rz between sapphire and thermistor, that is estimated from the risetime measured with the constant voltage configuration, is 10^7 K/Watt around 150 mK and is at least one order of magnitude smaller than the impedance between the bolometer and the thermal sink.

The pulse width is smaller than expected on for an ideal bolometer with the effective thermal capacity Cb measured by the pulse decay time, the zero frequency responsivity of the thermistor and complete instant termalization of the alpha particle energy in the sapphire.

The ratio ε between measured and expected pulse width for the ideal bolometer vs temperature is shown in Fig. (8) for setup a and b.

The relative difference in pulse height obtained with setup a and setup b is fully explained by the different shunting effect of the thermal impedance Rz in the two cases. The above consideration are still valid if distributed thermal impedance and capacity is assumed for the Ruthenium oxide thick film.

The size of the pulse height reduction cannot be quantitatively attributed to microscopic energy trapping in metastable levels during the thermalization process. Indeed, pulse width measurements with a sapphire-germanium reference bolometer, built by substituting the thick film on the sapphire crystal with a glued germanium thermistor, and mounted with a thermal link and time constant very similar to setup a, indicate that the fraction of energy instantaneously thermalized in the sapphire is, at least, three times higher.

Thermal gradients between the sapphire and the thick film require inconsistent assumption to produce the required corrections

An error of one order of magnitude in determining Cb from pulse decay times seems implausible and so the inefficient pulse response has to be attributed to a reduced responsivity of the thick film sensor in pulse conditions. The fact that for setup c and time constant of about 1 sec. ε results about 20%, not far from the values obtained with the sapphire-germanium detector, support this hypothesis.

The temperature dependence of ε suggests to attribute the effect to a low thermal diffusivity in the non homogeneous thick fil, that is, to the high Kapitza thermal impedance between the metallic grains and the vitreous matrix of the TFR at the lowest temperatures.

The best alpha particle spectra obtained by pulse height analysis with the RuO_2 bolometer and the mixed nuclides alpha source have an energy resolution of 4% FWHM, mainly due to noise from the thermistor.

The energy resolution, obtained in identical conditions with the germanium-sapphire reference bolometer, that has electrical and thermal characteristics very similar to that of the RuO_2 devices, is .7% FWHM, for 5.5 MeV alpha particles.

The noise level limiting the resolution of the RuO_2 bolometer is in fair agreement with shot noise of the bias current in the thermistor.

The higher pulse responsivity of the germanium-sapphire system could explain the better resolution.

Conclusions

It is confirmed that, below 100mK, in pulse conditions the effective thermal capacity of RuO_2-Al_2O_3 composite bolometers is only due to Debye and electronic contributions and the large nuclear terms do not contribute unless very long time constant are used.

The modest pulse height observed in single particle detection is due to a pulse responsivity reduced with respect to the zero frequency value, and the effect may be attributed to the low thermal diffusivity in the inhomogeneus structure of the ruthenium oxide thick film resistors at very low temperatures.

The results is a positive indication about the possibility of building low thermal capacity bolometers also with materials with high nuclear moments.

The performance of ruthenium oxide thick film resistors under 100 mK in pulse conditions is not satisfactory for the realization of high quality composite bolometers.

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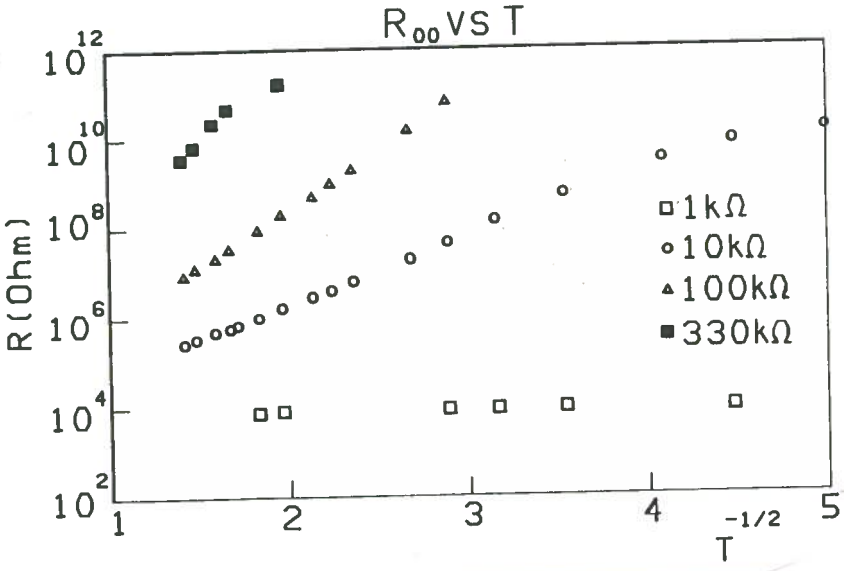

FIG. 1 - Zero power resistance R^o vs T dependence

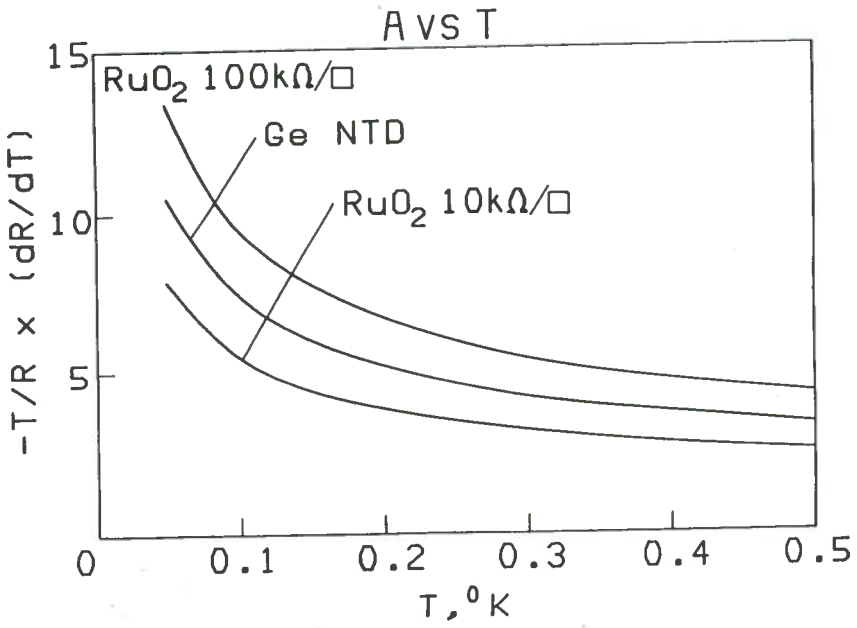

FIG. 2 - Zero power responsivity A= dlog R^o/dlogT vs T for 10 kΩ/□ and 100kΩ/□. For comparison the responsivity of a germanium NTD is reported.

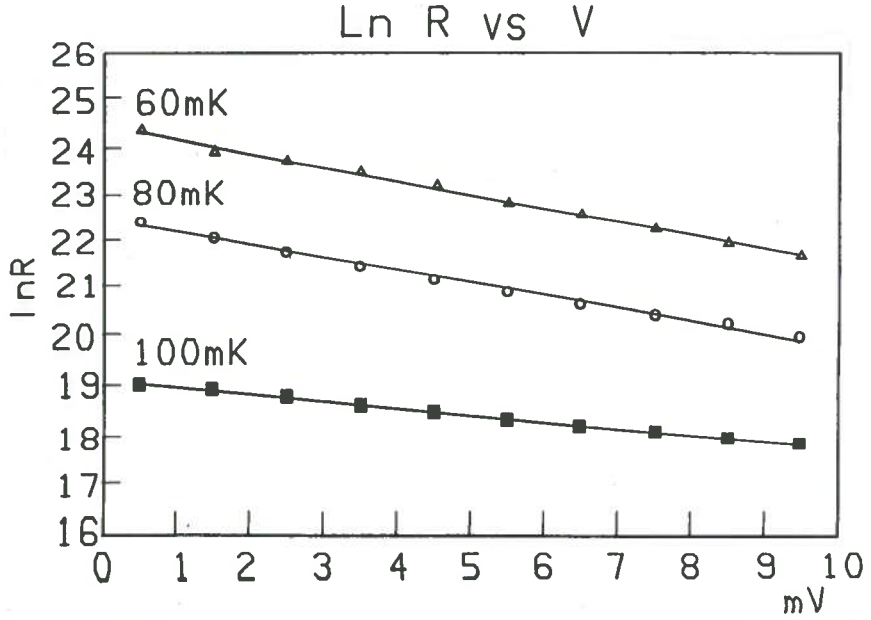

FIG. 3 - Log R vs V_b dependence of 10 kΩ/□ showing the field effect. The measurements are made with the TFR in tight thermal link with the heat sink.

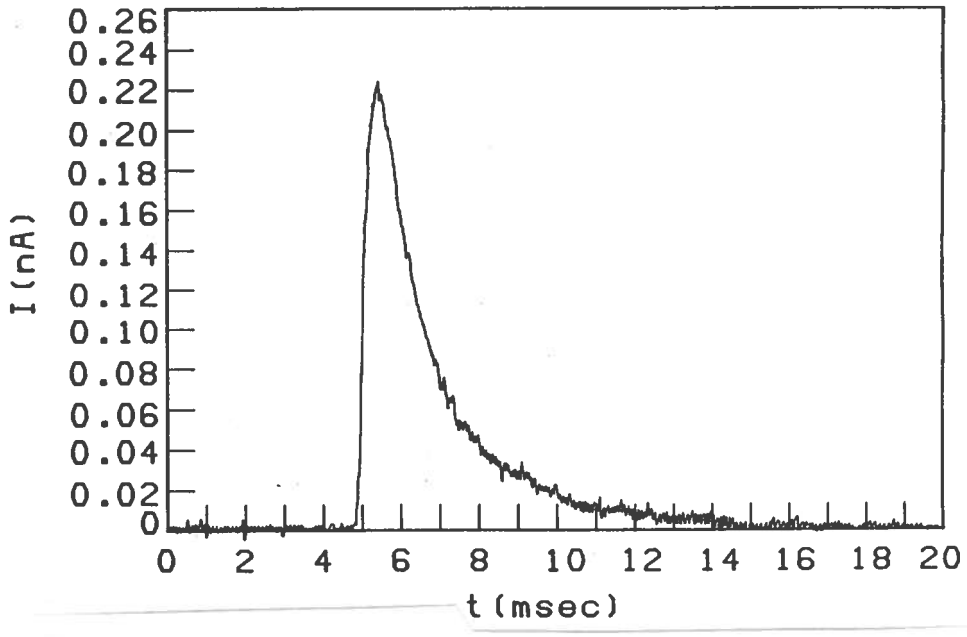

FIG. 4 - a) Setup a: Averaged pulse shape due to alpha particles. The full line represents the fit with the concentrated constants model.

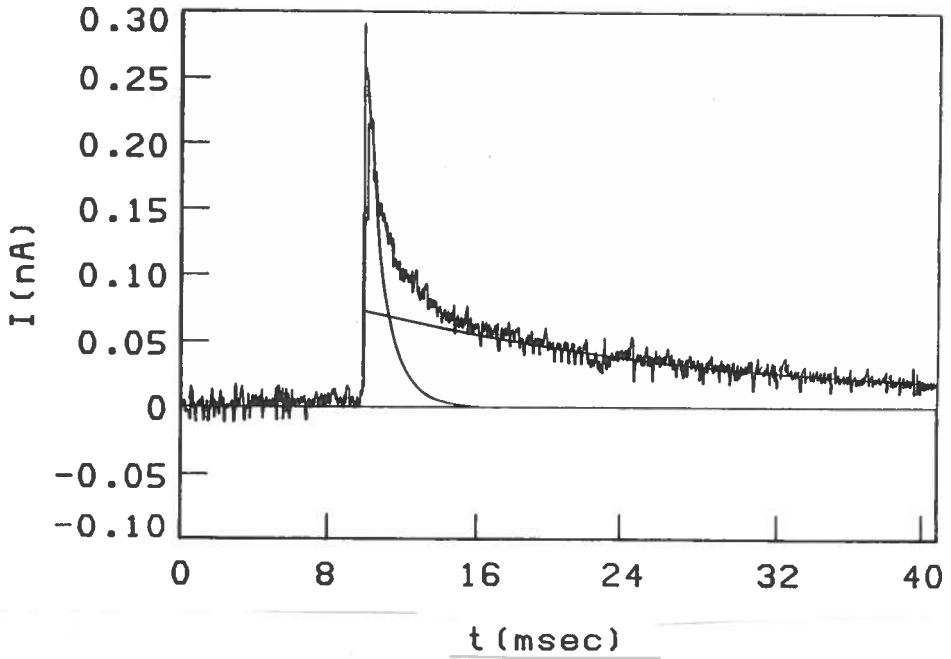

FIG. 4 - b) Setup c: Selected pulse due to an alpha particles

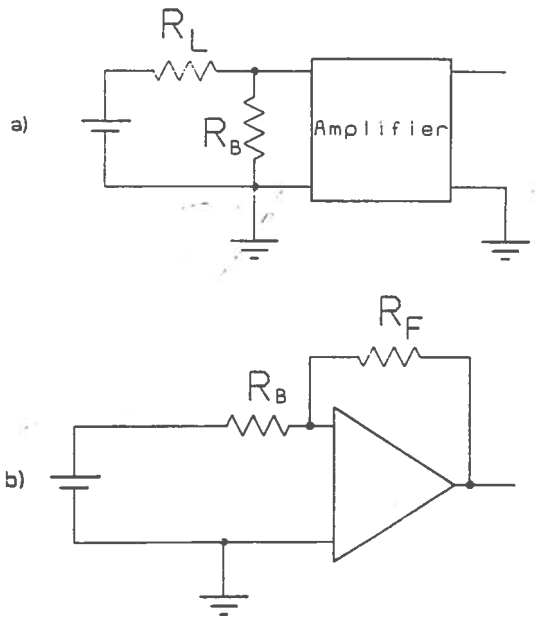

FIG. 5 -Biasing configurations-
a) Constant voltage biasing: The system, thanks to the reduced effect of the parasitic capacitance, allows risetime measurements also for high impedance bolometers, but is subject to thermal instabilities at high responsivity.
b) Load resistor biasing: The system is intrinsically stable, but for high values of the thermistor resistance the time response is slow

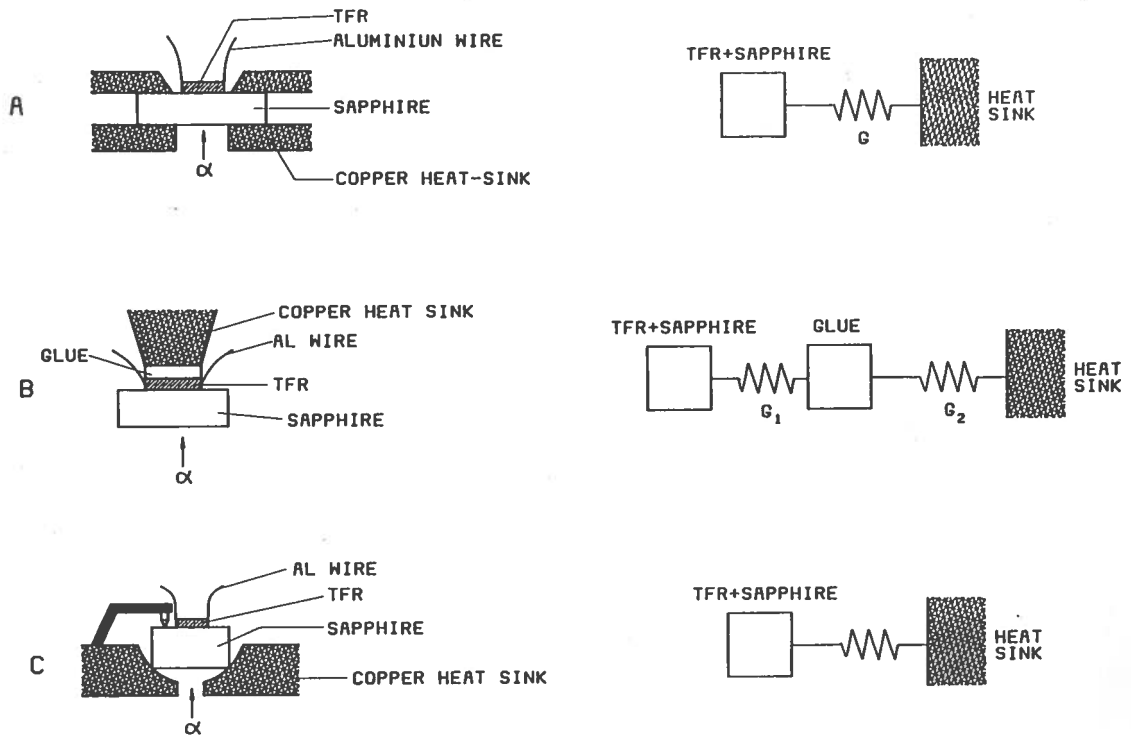

FIG. 6 - The three experimental setup and the corresponding concentrated constant models.Setup **a:** the thermal link to the heat sink is connected to the sapphire.
The thermal time constant is about 2 msec.
Setup **b**: the thermal link is connected to the TFR through the glue mass of thermal capacitance C_g. The main thermal time constant is about 20 msec.
Setup c: The thermal link is realized with the superconducting aluminum electrical connections to the TFR. The time constant is of the order of one second.

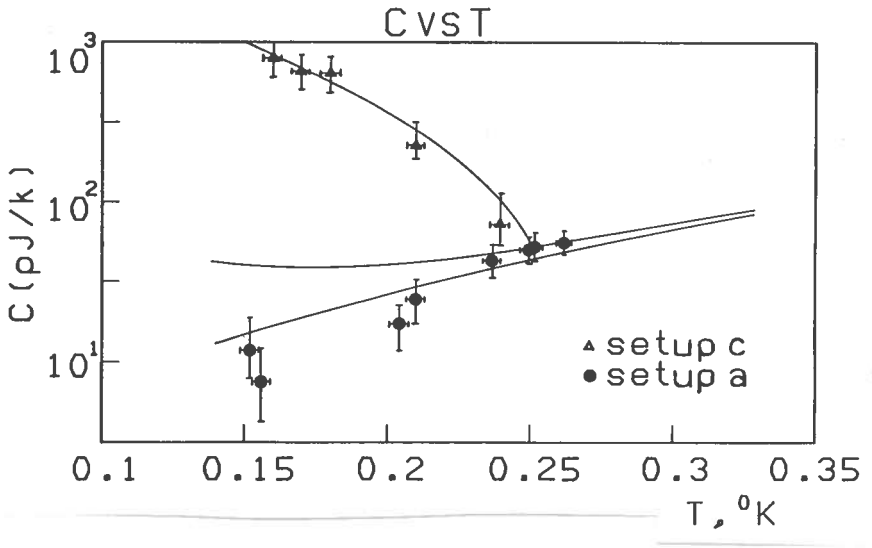

FIG. 7 - Thermal capacity of the RuO_2-Sapphire bolometer vs temperature. Lower line: Thermal capacitance computed with only the Debye and electronic contribution. Upper line: total thermal capacitance computed including the contributions from the nuclear quadrupole specific heat of Al_2O_3 (5) and from the RuO_2 (9). The central line indicates the static thermal capacitance of the RuO_2 with the static nuclear quadrupole interaction included.

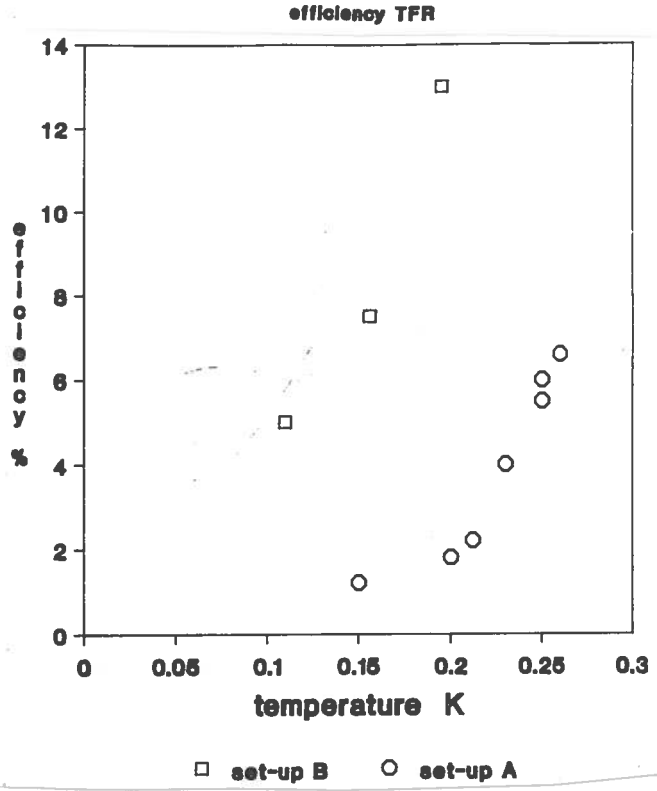

FIG. 8 - ε vs T plot for setup a (circles) and b (squares).ε is the ratio of the measured pulse width to the pulse width expected for an ideal bolometer with the effective thermal capacity Cb, responsivity equal to the zero frequency responsivity of the thermistor and complete instant termalization of the alpha particle energy in the sapphire vs temperature.

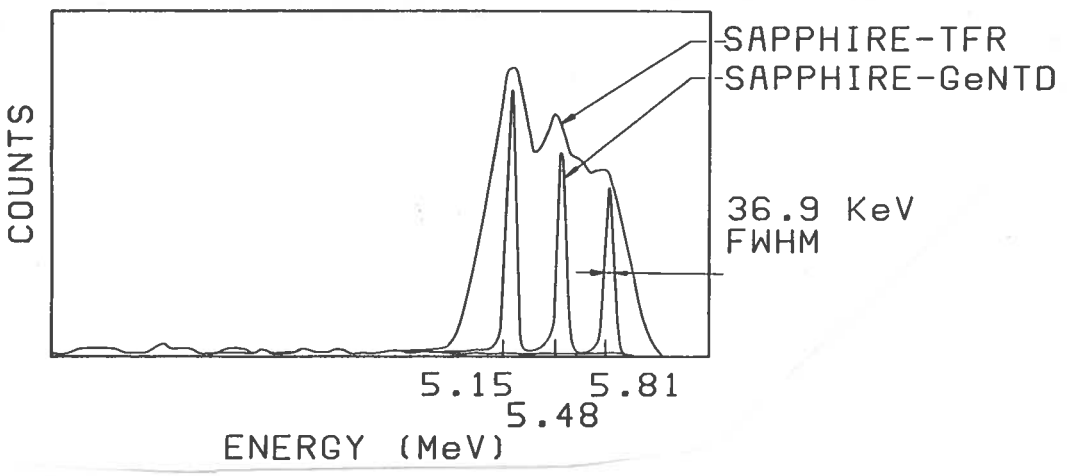

FIG. 9 - Comparison of spectra obtained with the Sapphire-RuO_2 and with the Sapphire-Ge bolometer. Mixed nuclides alpha source with main lines at 5.15 5.48 and 5.81 Mev.