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DEFORMATION - CALORIMETRY WITH LOW RESOLVING TIME BY USING ONE THERMOCOUPLE

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Abstract

A method is described to measure very small quantities of heat, produced in a muscle by active or passive deformation, with aid of one thermocouple inside the sample. The temporal resolution of the equipment is 160 ms and can be decreased to about 20 ms by mathematical methods employing the theory of linear response. The sensitivity threshold for heat measurements corresponds to a temperature change of $5 \cdot 10^{-5}$ K. Validity and linearity of the output response are verified by calibration and numerical solution of the heat conduction law. The method holds even for measurments of the deformation dynamics on polymers and other solids.

1. Introduction

Measuring the heat of deformation of various solids requires an appropriate calorimetric device, which permits to determine the heat of the total deformation process in long term measurements as well as the production of heat during the stretching experiment.

Thus the calorimeter should be quick enough for a high temporal resolution and it should be precise enough for a good accuracy. Unfortunately it is impossible to fullfill both requirements of accuracy and high temporal resolution by one device. Additionally calorimetric measurements of muscle often make spatial resolution of the distribution of heat sources desirable, because

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the muscles are not homogeneous (fibres, tendon).

So we have developed the following method:

In stretching experiments the total heat of deformation is measured by a heat - flux calorimeter (Schlipf and Höhne, 1981, Höhne et al., 1982) whereas the production of heat is determine by measuring the temperature change as a function of time insid the sample with a small thermocouple.

The change of temperature observed by the thermocouple is related to the heat of deformation by $Q = m \cdot c \cdot \Delta T$, if the condition are adiabatic. The latter is fullfilled if the deformation process is quick, the time constant of the thermocouple is low (i. its heat capacity is small) and the cooling of the sample is re latively slow. The cooling of the sample after having stretched is a complicated function of its heat capacity, its conductivit its geometrical form and of the surrounding medium.

Quick measurements by one thermocouple require an amplifying de vice, which is very quick and which has a great gain and a smal noise. Furthermore a periodical perturbation by signals generated by stimulating muscles via nerve, had to be dropped.

2. Experimental Equipment

These requirements lead to the application of Lock - in - ampli fication, thus periodical signals (the frequency and phase of which are known) were needed.

As the temperature observed by the thermocouple cannot be modulated , the electrical signal produced by the thermocouple is chopped and amplified by a Lock-in, synchronized by the chopper frequency. In order to eliminate statistical errors, the stretching experiment is carried out about ten times, the results ar stored in a microcomputer and the average is recorded.

The experimental equipment is shown in fig. 1, the technical dates can be seen in the following table.

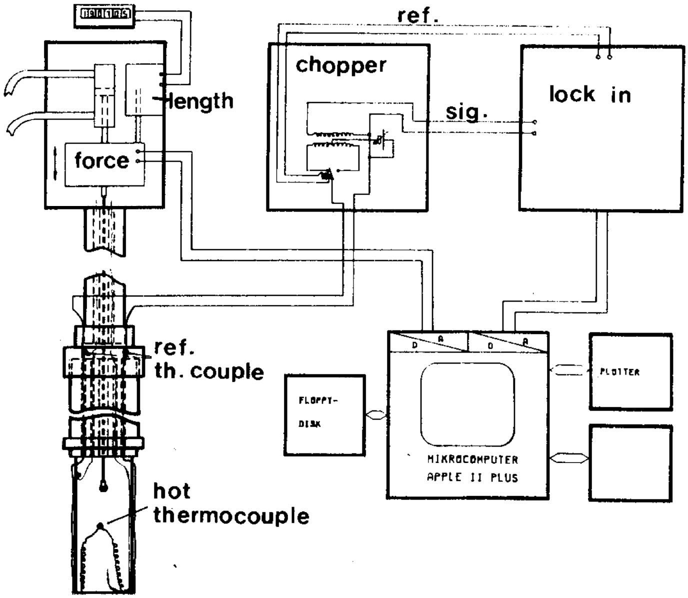


Fig. 1: Experimental equipment for measuring work and heat changes inside of samples, during deformation. The measuring cell (left part of the figure) is placed inside a heat-flux calorimeter.

Table:

temperature resolution	$5 \cdot 10^{-5}$ K
risetime	62 ms (50 % of signal)
	165 ms (90 % of signal)
chopper frequency	96 cps
calibration constant	$1,3 \cdot 10^{-5}$ k/mm plot $\pm$ 10%

An original measurement of tension and heat versus time, of stretching resting muscle, is drawn in fig. 2.

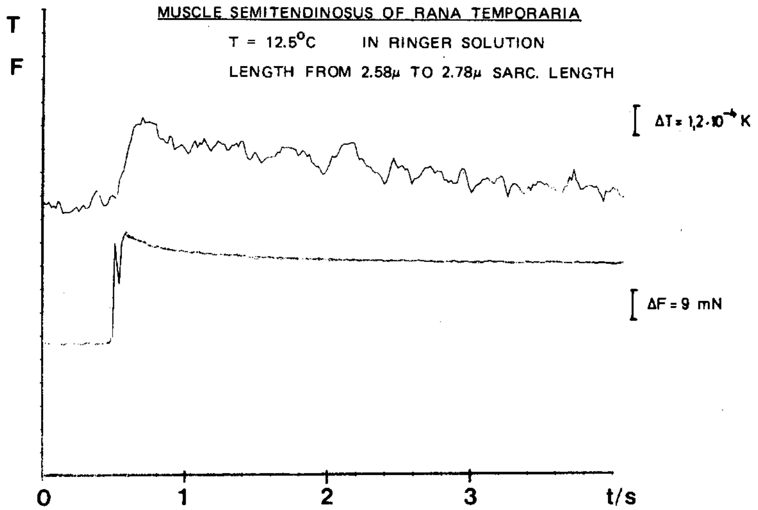


Fig. 2: Temperature change and tension measurement in a stretching experiment with a muscle, placed in Ringersolution.
Plotted functions are the average of 5 measurements.

2.1 Calibration and Temporal Resolution

There are two ways to calibrate the system. The first one is based upon a calibration of the electrical part of the device, where the thermocouple is replaced by a known voltage source. This voltage source produces rectangular pulses of known height and width, which are nearly the same as given by the thermocouple under experimental conditions. The response is recorded by the equipment.

It can be described by the parameters t_{50} and t_{90}, denoting that time, after which the response has reached 50 % and 90 % of its maximum value, respectively. Fig. 3 demonstrates the result of the "electrical" calibration.

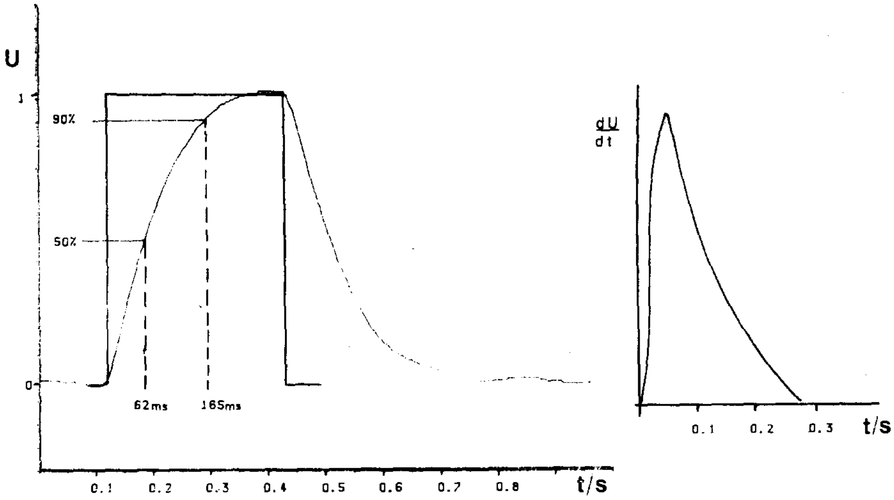


Fig. 3: a) Response of the electrical equipment to a rectangular signal the risetime for 50 % of the signal is 62 ms, for 90 % of the signal 165 ms.

b) Differentiated left part of the response signal, the "apparatusfunction" of the electrical equipment.

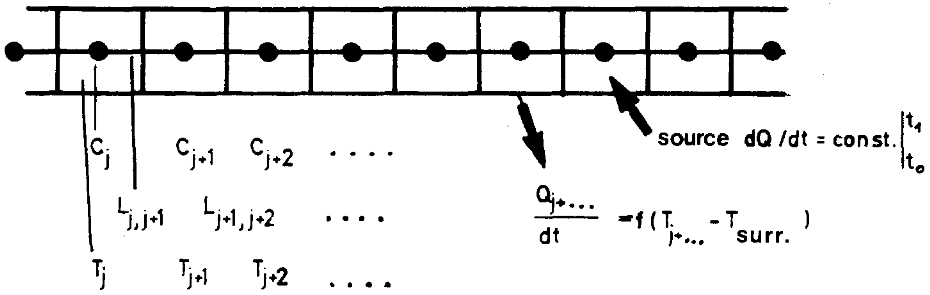
The calibration constant of the complete system describing the relation between temperature and the recorded signal is finally calculated from the known thermoelectric power of the thermocouple.

So the "electrical" calibration constant of $1{,}2 \cdot 10^{-5}$ K ± 5 % /mm plot is yielded by assuming a thermoelectric power of 60 μV/K. This way of calibration seems to be rather theoretical, however, since the thermoelectric power of the thermocouple could be influenced by the deformation of the thermocouple itself. This deformation cannot be avoided, because it is fixed in the sample which is stretched. So a second more "experimental" way giving additional information with respect to calibration has been applied, where a sample of well known thermoelastic properties is measured. We used natural rubber, the properties of which ha been determined by Kilian et al. (1983). The samples were fixed just like the muscles in the calorimeter and had a similar stre strain behaviour. The observed relation between the change of temperature and strain, multiplied by the heat capacity of the sample is compared with the dates of literature. By this method a calibration constant of 1,4 · 10^{-5} K/mm plot ± 10 % is obtain which agrees with the value given by the first method. This result, however, cannot be applied to the stretching experiment o muscles, because the surroundings of the muscles consists of Ringer solution, which has nearly the same thermal properties a water, whereas the natural rubber is surrounded by air. The cal bration by stretching natural rubber cannot be carried out in water, because this would change the thermoelastic properties - thus the dates of literature do not hold for this case. In orde to apply the calibration to the measurement of muscles, we calculate the transport of heat in the various parts of the system consisting of the sample, the thermocouple and the surroundings

2.2. Calculation of Transport of Heat

Transport of heat in the system has been calculated numerically by the relaxation method (Schneider, 1957; Sucec, 1975), in one dimensional approximation. Fig. 4 shows the arrangement of cell the properties of which are permitted to differ from each other i.e. the system is allowed to be inhomogenous.

The relaxation method leads to an recursive algorithm see (Hoff 1983) determining the temperatures of each cell at any time fro given boundary and initial conditions



$$T_j = T_{Aj} + \left[\Delta t \left\{ L_j \left(T_{Aj-1} - T_{Aj}\right) - L_{j+1} \left(T_{Aj} - T_{Aj+1}\right) \right\} + Q_j + Q_q \right] / C_j$$

Fig. 4: One dimensional model for numerically calculation of heat transfer

C_j = heat capacity of one cell

$L_{j, j+1}$ = coefficient of heat transfer between neighboured cells

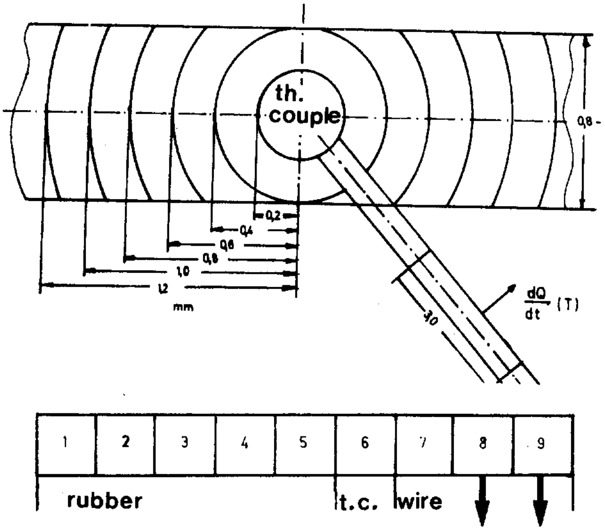
T_j = actual temperature of one cell,

T_{Aj} = temperature before

T_{surr} = temperature of surroundings

This onedimensional arrangement is considered to represent the threedimensional arrangement of a model of the real system (fig.5). As the coordinate of the onedimensional arrangement corresponds to the radius of the threedimensional model, each cell represents one shell of an arrangement of concentric spheres. The heat capacities and the coefficients of conductivity depend on the radius. They have been calculated for each cell (fig. 5).

The numerical calculation of heat transfer has been carried out for a heat pulse leading to a stepchange of temperature distributed



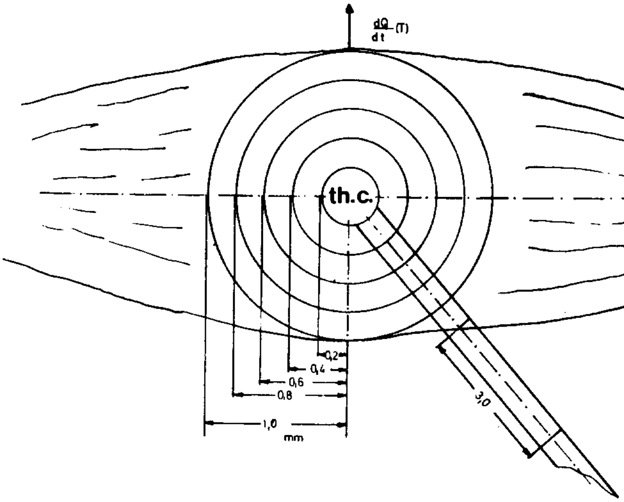
	Cell	C_j (mJ/K)	$L_{j,j+1}$ (mW/K)
Rubber	1	1,77	4,0
	2	1,44	3,2
	3	1,12	2,4
	4	0,8	1,6
	5	0,37	0,79
Th. couple	6	0,125	0,23
Wire	7	0,146	0,13
	8	0,146	0,13
	9	0,146	0,13

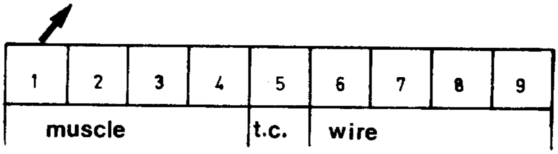
Heat loss: Cell 8 and 9, 20 W/m^2
Surface: Cell 8 and 9, 1,7 mm^2

Fig. 5: Quasi onedimensional model of heat transfer

a) in the case of a sample of natural rubber surrounded by air

$c_{p,rub}$=1,75 J/gK, $\lambda_{th.c.}$ 30 W/mK, λ_{rub} = 0,16 W/mK





	Cell	C_j (mJ/K)	$L_{j,j+1}$ (mW/K)
Muscle	1	7,14	24,5
	2	4,34	13,8
	3	2,24	6,12
	4	0,8	2,94
Th. couple	5	0,125	0,23
Wire	6	0,146	0,13
	7	0,146	0,13
	8	0,146	0,13
	9	0,146	0,13

Heat loss: Cell 1, 1000 W/m^2
Surface: Cell 1, 10 mm^2

Fig. 5 b) in the case of a frog muscle surrounded by Ringer solution

$c_{p,mu} = 3{,}5$ J/gK, $\lambda_{H_2O} = 0{,}6$ W/mK, $\lambda_{mu} \triangleq \lambda_{H_2O}$

homogeneously all over the sample. After the pulse the heat flows into the cell simulating the thermocouple and into the surroundings. In fig. 6 the temperature of the thermocouple is plotted versus time for natural rubber surrounded by air and for a muscle surrounded by water. The wires of the thermocouple are in contact with the surroundings, hence a transport of heat occurs trough them, because of their relatively large thermal conductivity. This has been taken into account by considering them as a heat sink, whereas the sample is thought adiabaticall isolated. In the case of the muscle the thermal conductivity is much greater than that of rubber, additionally the Ringer solut corresponds by no mean to an adiabatical isolation. As the trans port into Ringer solution across the surface of the sample is great compared to that through the wire (the surface between th sample and the wire is small), the latter is neglected
The result reveals a plausible behaviour: the maximum value is hardly influenced, whereas the rate of cooling depends on the strength of the heat sink standing for heat transfer into the surroundings.

The total signal of the equipment is obtained as a convolution integral of the numerical solution, concerning the temperature change of the thermocouple with the "apparatus function", describing the dynamical behaviour of the electronic device with respect to a pulse shaped signal (see fig. 3). This convolution integral is plotted in fig. 7 for those times, where the signal increases. The cooling process is not taken into account in these diagramms, since the determination of the heat by the relation $Q = m \cdot c \cdot \Delta T$. implies adiabatic conditions. The integrat curves of fig. 7 can be compared with the heat of deformation o served in a real stretching experiment - the result is demonstr

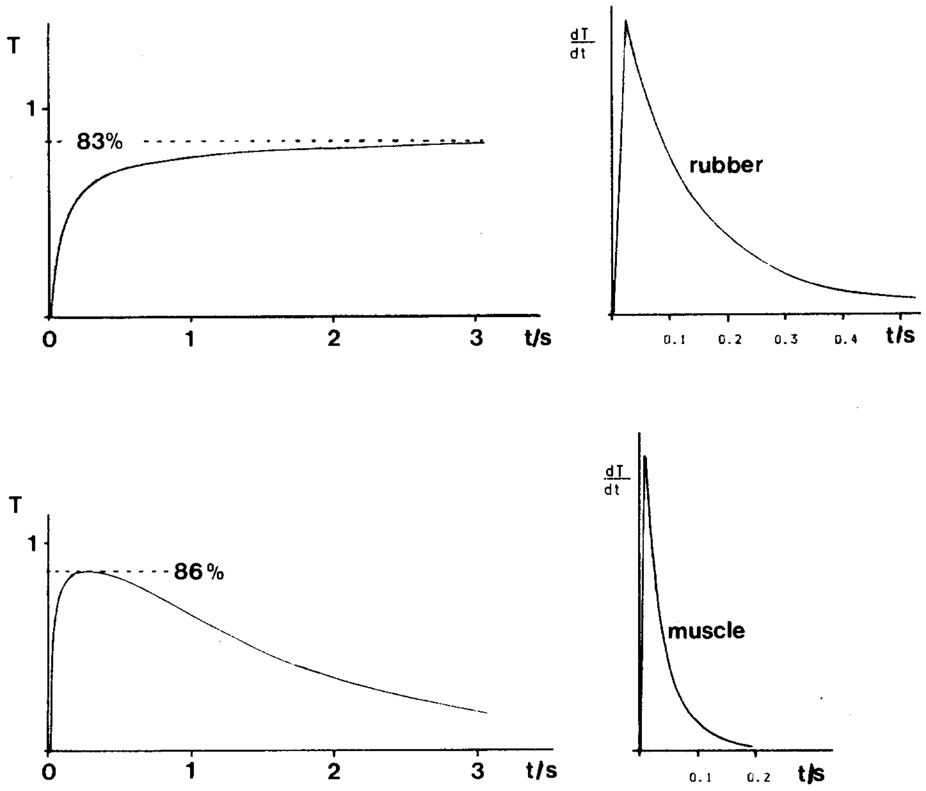


Fig. 6: Calculated change of temperature of the model from fig. 5 in case of a) rubber and b) muscle
Right hand side of the figure:
Differentiated left part of the curves a) and b) of figure 5

ted in fig. 8. As can be seen the calculated and the measured curve coincide thus giving evidence both for the correctness of the one dimensional model computation and the linearity of the device. As a result the "apparatus function" (fig. 3 b) holds for to "desmear" the measured signal to get the original temporal heat production of the sample with a resolution of 20 ms.

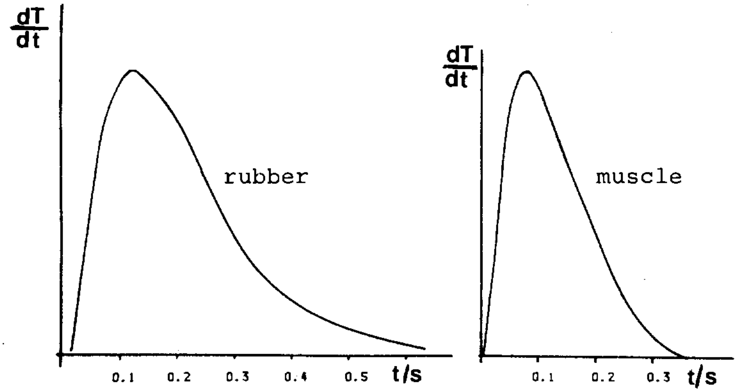


Fig. 7: Differentiated curves from fig. 6 convoluted with "apparatus function" from fig. 3 b

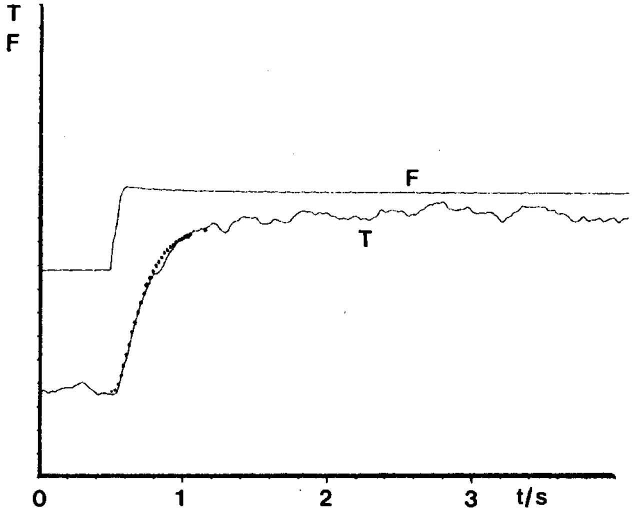


Fig. 8 a: Averaged temperature change of rubber in air
Solid lines: As measured after 10 % stretch
Dotted lines: As calculated by integration of curves in fig. 7.

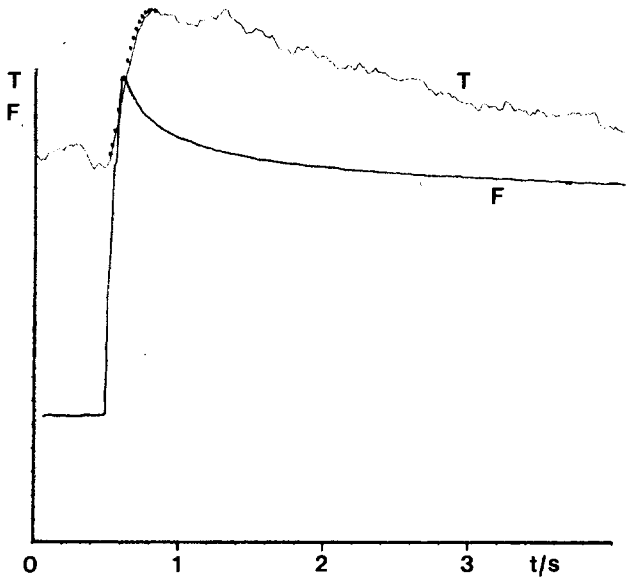


Fig. 8 b: Averaged temperature change of frog muscle in Ringer solution.

Solid lines, dotted lines: See fig. 8 a

3. Conclusions

The described method may be used, to measure heat productions in small samples, as the temperature rise is independent of the volume. To give statements about the heat produced in the sample, it is necessary to know the heat capacities of the sample and the thermocouple. For measurements of the total heat produced of a sample, the assumption have to be made, that the sample is homogenous, or the measured region is representative for the whole sample. As a result it is possible to carry out caloric measurements with higher temporal resolution and precision particularly in small samples, as by heat flux calorimeters. The advantages of the method, enables the possibility of further applications in dynamical calorimetry, e.g. in muscles, but also in polymeres and other solids.

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