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TEMPERATURE REGULATION FOR THERMAL ANALYSIS UNDER MICROWAVE RADIATION

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Abstract

In a microwave study a program was designed for thermal regulation. This software allows different types of regulations: P, PI, PD or PID (proportional integration derivation). Results obtained with PID and PI regulation for different previous linear heating schedules (1 deg·s^{-1} and 0.15 deg·s^{-1}) imposed on a polymer sample (DGEBA/3DCM) up to 280°C and more are reported. Mathematical resolution of thermal laws applied to the sample permits the regulation constants (T_i, T_d and G_a) to be linked to the physical features of the polymer. A method used to calculate: the reflexion constant of the wave on the polymer, ρ and the diminution factor, α is presented.

Keywords: diminution factor α of the wave, microwave, PID regulation, reflexion constant ρ

I. Thermal regulation

Introduction

The designed program permits imposition of an order of temperature on a polymer sample by controlling a microwave generator. Orders may be a linear increase of temperature or a single step selected by the operator.

The polymer subjected to microwave radiation is an epoxy resin: DGEBA/3DCM, cured by microwave heating at 2.45 GHz [1].

Equipment

The program runs under Turbo Pascal 6.0 [2]. The hardware consists of a 386 microprocessor which runs at a speed of 16 MHz and a 80386SX coprocessor. Data acquisition is performed by an Analog Device RTI 815 electronic

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card. A Chromel Alumel thermocouple set in the sample core via a bore in it is used for temperature measurements. Thus, the acquired temperature is the inside temperature of the sample. The microwave generator designed by Sairem delivers an adjustable power between 0 and 1200 W at a frequency of 2.45 GHz. We used a cavity which gives a resonating standing wave with a coupling iris and a short circuit at the end of the cavity.

Samples

Samples used were taken from an epoxy resin DGEBA/3DCM. They were cylindrical or parallelepipedic in shape, measuring Ø 6×25 mm or 16×8×8 mm, with a bore of Ø 2 mm to set the thermocouple.

The regulation program

The program designed in the laboratory to perform these experiments is very easy to use, thanks to various messages to help the operator during the acquisition of the experimental parameters to stop or retrieve an experiment already done. The data acquisition is performed 5 times per second, and stored in a file of results. The different types of regulation systems shown are YN (Yes, the power is switched on; No, the power is switched off), PI and PID [3].

Results

a) YN regulation

For this regulation, the operator sets the level of incident power used during the experiment and the rate of heating. This regulation software is designed with two proportional strips, the first at 10 K below the ordered slope of temperature to set the microwave power at 20% of the specified power (P_{max}) and other at 5 K below the order to set 5% of P_{max} (P_{max} is the maximum incident power used during the experiment).

Figures 1–3 show results obtained for 200 s of experiment at an ordered heating rate of 1 deg·s^{-1} for various P_{max} values on a parallelepipedic sample.

In each case, the regulator exhibits good behaviour. After a short preliminary period of about 30 s, the order is followed without any gap. However, a difference is observed between the order and the sample temperature, with an increase in the preliminary period at weaker P_{max} (Table 1).

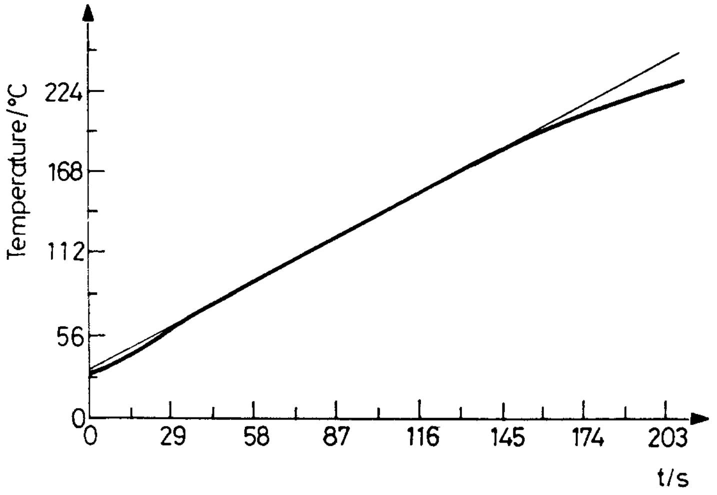

Fig. 1

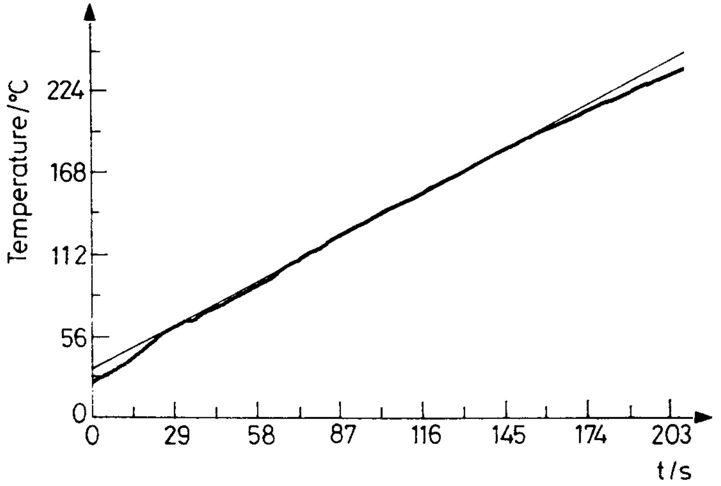

Fig. 2

Conclusion

A good result was obtained with a specified power of 840 W for a linear heating rate order of 1 deg·s^{-1} with a gap just after 242°C. For this temperature, the thermal radiance loss is more important than the microwave power dissipated in the polymer sample.

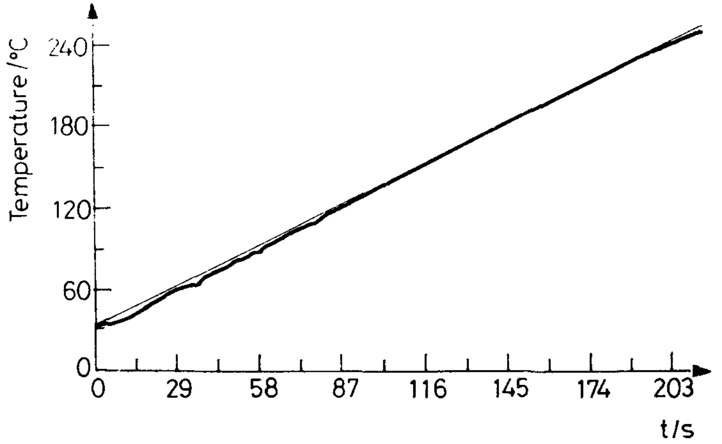

Fig. 3

Table 1

Figure	Power (P_{max}) / W	Preliminary period / s	Beginning of gap / °C
1	660	36	169
2	720	25	180
3	840	20	240

The regulator is not responsible for this gap, because at that temperature the maximum power was delivered to the sample. The problem was that the heating ordered slope was too high for this material.

b) PID and PI regulators

For these regulation systems, the operator specifies the heating rate order. Nevertheless, it is necessary to make an experiment with the regulator switched off to calculate its constants. The Broïda [3] method was used. A step of microwave power (200 W) was imposed on the polymer after 90 s. The polymer reaction in this step is shown in Fig. 4 (a parallelepipedic sample).

The following values were obtained from the Broïda formula:

$$\tau = 90.6;\ \theta = 262.06;\ G_s = 0.88$$

$$\theta = 5.5(t_1 - t_2)$$

$$\tau = 2.8t_1 - 1.8t_2)$$

$$G_s = \frac{\textit{Value of power step}}{\textit{Value of highest temperature reached}}$$

From the curve obtained, t_1 and t_2 are respectively the abscissa of $s_1 = 0.28 \cdot S_f$ and $s_2 = 0.40 \cdot S_f$. S_f is the maximum value reached by the response curve, in our case $S_f \approx 190°\text{C}$.

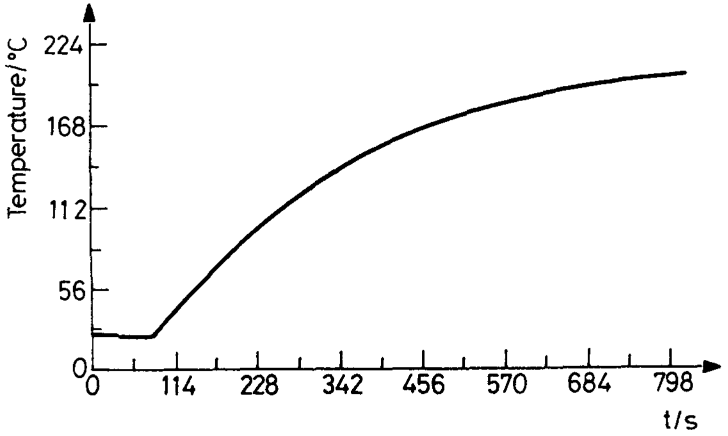

Fig. 4 Increase of temperature due to a power step of 200 W

These constants allow calculation of the different factors of the regulator.

$$\text{PI: } Y(p) = 2.79\left(1 + \frac{1}{262.06p}\right)\varepsilon(p)$$

$$\text{PID series: } Y(p) = 2.79\left(1 + \frac{1}{262.06p}\right)(1 + 36.24p)\ \varepsilon(p)$$

$$\text{PID parallel: } Y(p) = \left[3.21 + \frac{1}{106.3p} + 104.23p\right]\varepsilon(p)$$

$$\text{PID mixed: } Y(p) = 3.12\left[1 + \frac{1}{298.3p} + 31.8p\right]\varepsilon(p)$$

Y is the control signal imposed to the generator
ε is the gap between the order and the real temperature
p is the Laplace transformation of the input signal.

The PID relations after transformation of p into a temporal variable were the same. We arbitrarily used the PID series relation in our program. Results obtained for a heating order of 1 deg$\cdot$s^{-1} with PI and PID regulation are shown in Figs 5–9.

These results demonstrate the great efficiency of the PI and PID regulators.

Figure 5 depicts regulation obtained with a PI regulator. The time of reaction is 10 s, and the gap between order and real temperature is zero up to 230°C for the same slope as the order.

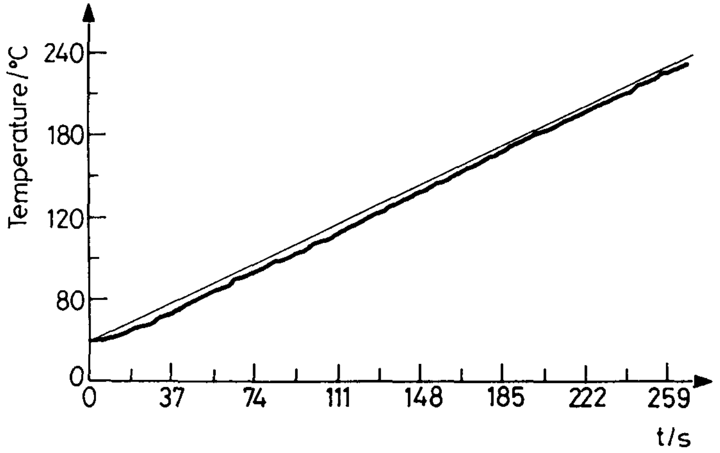

Fig. 5 PI regulator, parallelepipedic sample, 1 deg·s^{-1}

Figure 6 shows the result given by a PID series regulation. In this case, the derived action creates some instability, but reduces the gap between order and measured temperature to zero. This behaviour was forecast by Broïda [3]. He recommended the use of a PI regulation or a PID with weak derivative action for this type of system (1st order) in order to have absolute stability during the regulation. The microwave power was stopped before the end of the experiment, to avoid sample destruction.

Figure 7 gives the result obtained with a PID series with weak derivative action. The final temperature is more than 260°C without any differences between the order the sample temperature for an ordered heating rate of 1 deg·s^{-1}.

Figure 8 shows the curve obtained for a heating rate of 0.15 deg·s^{-1}. The regulator displays perfect action, which maintains the some heating rate throughout the experiment. Thus, this system will be used to regulate the temperature increase during thermal analysis under microwave.

Figure 9 demonstrates that in this case thermal loss is too important, even for a radiance of 900 W. This material was not able to follow such an order.

This is an extreme order, which was intended to show the maximum order that can be imposed on this material.

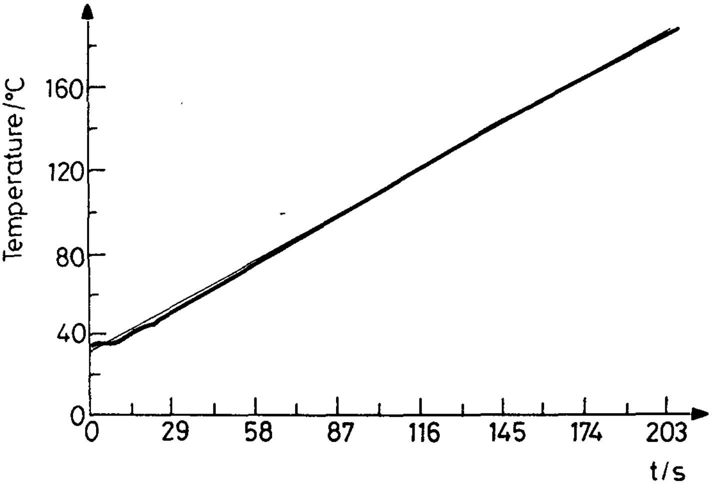

Fig. 6 PID regulation, cylindrical sample, 1 deg·s^{-1}

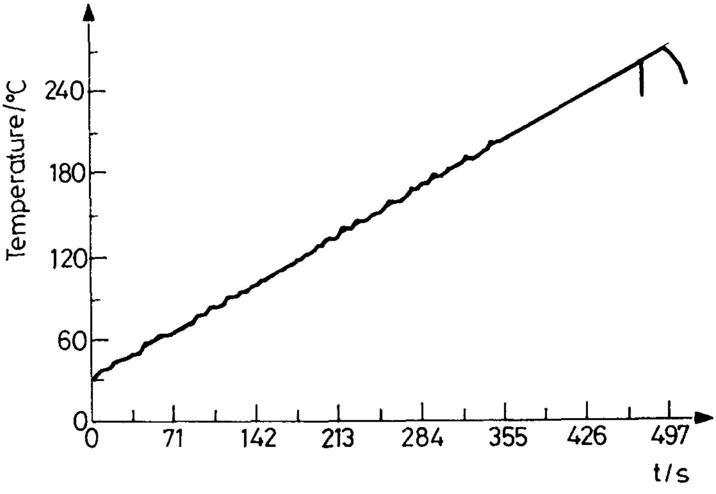

Fig. 7 PID regulation, parallelepipedic sample, 1 deg·s^{-1}

II. Physical meaning of transfer function constants

Introduction

It is interesting to link the regulation factors to specific physical features of the material. There are two different possibilities:

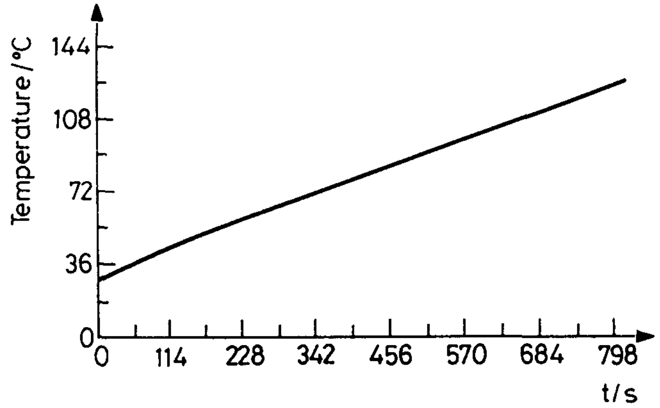

Fig. 8 PID regulator, cylindrical sample, 0.15 deg·s^{-1}

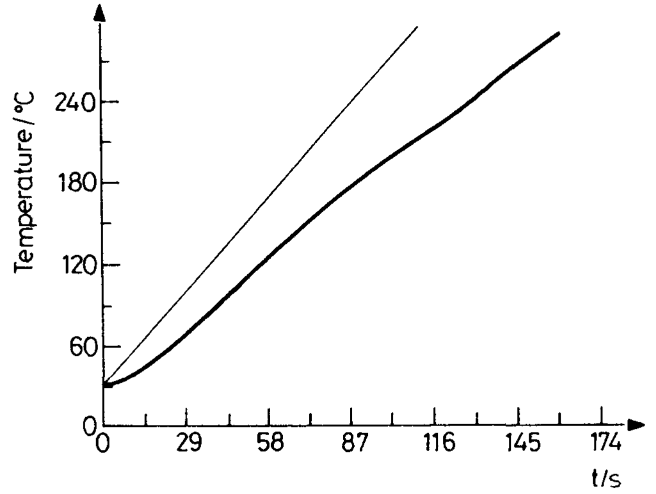

Fig. 9 PID regulator, parallelepipedic sample, 2 deg·s^{-1}

– If the material features are *well known*, then no equipment is needed to determine the PID factors. The established mathematical relations will give this factors.

– If the material features are *unknown*, then the material reaction to a power step will give the PID factors and the specific physical features of the sample graphically.

Transfer function calculation

Application of a thermal balance sheet to the sample gives the following relation [4]:

$$Q_1 = Q_2 + Q_3 \tag{1}$$

where Q_1 is the heat pile up, Q_2 is the radiance loss and Q_3 is the microwave heating.

There is no thermal loss by convection because the cavity has no opening which can create a convection flux.

This equation can also be written as follows:

$$mC_\mathrm{p}\frac{\mathrm{d}T}{\mathrm{d}t} = -\ hS(T - T_\mathrm{ext}) + W_\mathrm{i}(1 - \rho^2)\ \exp\ (-\ 2\alpha d) \tag{2}$$

C_p is the heat capacity in weight ($\mathrm{W \cdot kg^{-1} \cdot K^{-1}}$)
h is the overall heat transfer coefficient ($\mathrm{W \cdot m^{-2} \cdot K^{-1}}$)
W_i is the microwave power (W),
α is the reduction constant,
m is the sample weight (kg),
S is the sample surface ($\mathrm{m^2}$),
ρ is the reflexion constant of the electromagnetic wave on the material,
d is the sample thickness (m).

First assumption

The reflexion factor on the material, $(1-\rho^2)$, is constant between two acquisition temperatures. The slope of the function $(1-\rho^2) = f(t)$ (Fig. 10) is linear after 100°C for an epoxy resin cured under microwaves.

Thus, for an interval of less than 1 K, which corresponds to the present acquisition rate, the first assumption is justified after 110°C.

Second assumption

The variations in the reducing factor α are small compared to the sample size. By definition, we have $D_\mathrm{p} = 1/2\alpha$, which represents the penetration depth of the wave in the material. For a frequency of 2.45 GHz, $D_\mathrm{p}=25$ cm for an epoxy resin (DGEBA/3DCM) cured under microwave. Then, even if α decreases with temperature, D_p is still high enough to keep the penetration depth of the microwaves higher than the size of the sample (2 cm). Thus, the second assumption is justified.

Third assumption

The external temperature (25°C) is lower than the average temperature of the experiments (130°C), and thus hST_{ext} is insignificant relative to $T(p)[mC_p\,p + hS]$.

Then, with assumptions one and two, Eq. (2) becomes,

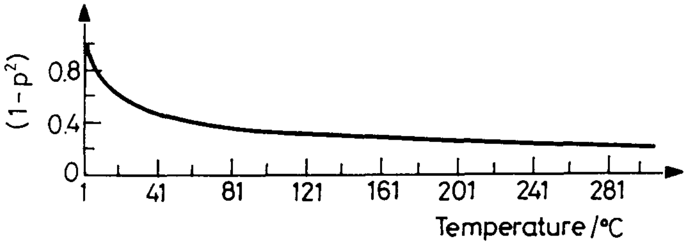

Fig. 10 Theoretical evolution of the function $(1-\rho^2) = f(t)$

$$mC_pT(p)p + hS(T(p) - T_{ext}) = W_i(1 - \rho^2)\exp(-2\alpha d) \tag{3}$$

$$T(p)(mC_p\,p + hS) - hST_{ext} = W_i(1 - \rho^2)\exp(-2\alpha d) \tag{4}$$

and with the third assumption:

$$T(p)(mC_p\,p + hS) = W_i(1 - \rho^2)\exp(-2\alpha d) \tag{5}$$

This gives the transfer function of the system:

$$H(p) = \frac{T(p)}{Wi(p)} = \frac{(1-\rho^2)\exp(-2\alpha d)}{mC_p\,p + hS} = \tag{6}$$

$$= \frac{(1-\rho^2)\exp(-2\alpha d)}{hS}\left[\frac{1}{1 + \left(\frac{mC_p}{hS}\right)p}\right]$$

This transfer function is a first-order transfer function. The overall form of these functions is

$$H(p) = \frac{G_r}{1 + \zeta p} \tag{7}$$

where G_r is the gain and ζ is a constant of time for the system:

$$G_r = \frac{(1-\rho^2)\exp(-2\alpha d)}{hS} \tag{8}$$

$$\zeta = \frac{mC_p}{hS} \tag{9}$$

The curve in Fig. 4 is exactly the same as for a first-order system, and thus Eq. (5) is right. Then, if we use two samples of the same material, but different in shape, and two different power steps, Eq. (8) will give two different values G_{r1} and G_{r2}. The ratio of these values will give α and ρ:

$$\alpha = -\frac{1}{2(d_2 - d_1)} \ln\left(\frac{G_{r1}}{G_{r2}}\right) \tag{10}$$

$$\rho = \sqrt{\left(1 - \frac{hSG_r}{\exp(-2\,\alpha\, d)}\right)} \tag{11}$$

III. General conclusion

These examples demonstrate the possibility of imposing a linear heating rate by microwave heating on a polymer with a PID regulator and to determine some physical data of the sample.

This type of heating will be of interest for thermal analysis because a material with a bad thermal conduction factor will never be heated so rapidly by conventional heating especially in the bulk.

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Zusammenfassung — Im Rahmen einer Mikrowellenuntersuchung wurde ein Programm zur thermischen Regelung entwickelt. Diese Software erlaubt verschiedene Arten von Regeltypen: P, PI, PD oder PID. Es wird über die Ergebnisse der PID- und PI-Regelung verschiedener vorangehender, auf Polymerproben (DGEBA/3DCM) bis 280°C und mehr angewendeter linearer Aufheizprogramme (1 deg/s und 0.15 deg/s) berichtet. Die Anwendung der mathematische Lösung der thermischen Gleichungen auf die Proben gestattet es, eine Verbindung zwischen den Regelkonstanten (T_i, T_d und G_s) und physikalischen Eigenschaften des Polymers herzustellen. Es wird weiterhin eine Methode zur Berechnung der Reflexionskonstante ρ der Welle auf das Polymer und des Abminderungsfaktors α gegeben.