DYNAMIC MEASUREMENTS OF COMPLEX PERMITTIVITY AND TEMPERATURE DURING MICROWAVE HEATING

Michel R. OLLIVON*

Organisation Moleculaire et Macromoleculaire, C.N.R.S.-BP 28, THIAIS-94320, France, *Present adress: L.T.B./N.C.I., N.I.H., Bethesda, MD 20205.

ABSTRACT

A simple, fast and fully automated method for the measurement of complex permittivities and temperature of solid or liquid samples heated by microwave absorption at 2.43 GHz, using the small perturbation technique, is described. First results are discussed.

INTRODUCTION

The microwave complex permittivity of a dielectric is temperature dependent and the developement of many applications needs the knowledge of this dependence. Since there has been speculation about differences in the permittivities (ε' and ε'') of materials heated with microwave power, compared to the permittivities of the same materials heated by conventionnal methods, it is interesting to measure these permittivities during microwave heating (1,2).

A simple automated method for the accurate measurement of complex permittivities of samples, heated by microwave absorption at 2.43 GHz, is described. This technique allows both the monitoring of heating kinetics, and permittivity measurements, simultaneously.

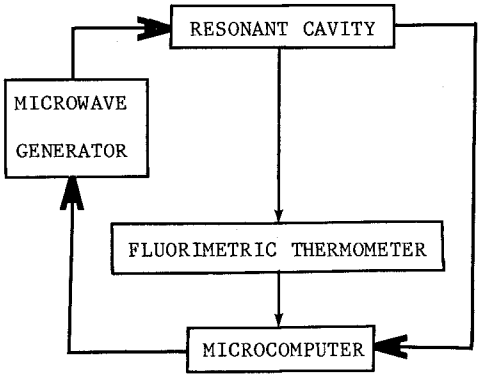

Fig.1 Basic closed loop circuit used to monitor the cavity resonance

METHOD AND APPARATUS

The small perturbation technique (3-5) is used, but the cavity resonant frequency tracking is obtained by frequency tuning instead of the mechanical adjustement generally used. Moreover, both permittivity measurements and heating are performed with the same microwave source.

In the system, a microcomputer continuously monitors a resonant cavity containing the sample, via a frequency controlled generator, in a closed loop circuit (fig. 1). The frequency controlled generator (THOMSON) which contains a voltage and temperature controlled Xtal oscillator and multiplier-amplifier stages, can be precisely tuned to a selected frequency. It delivers microwave power up to about 6W to a monomode TE 013 rectangular cavity (fig. 2).

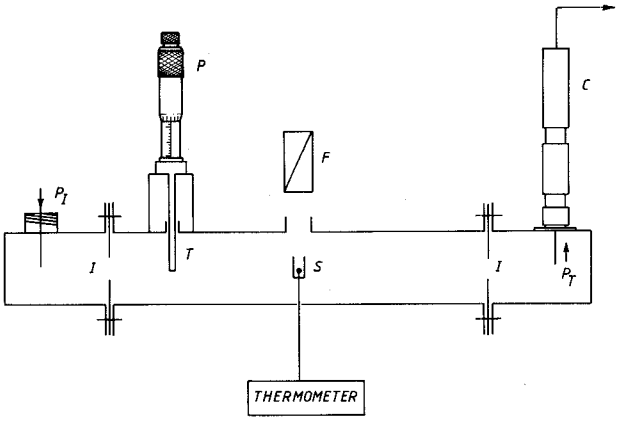

Fig.2 Schematic drawing of the cavity (S=Sample, C=Crystal detector, I=Input power, F=Small electric furnace, P=Piston used to remove the silica tube T when large sample has to be measured).

The sample is positioned at an electric field maxima and perturbes the cavity resonance. The microcomputer periodically measures the cavity's Q and the resonance frequency perturbation, via a crystal detector, using a fast frequency sweep (typically 50 msec.).

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The detector voltage is send to the microcomputer via an optically isolated instrumentation amplifier, then a 12 bit analog-digital converter. During the time intervals between sweeps (typically 5 sec.), the microcomputer controls the heating by tuning the frequency of the generator, via a digital-analog converter, to the resonance maximum of the cavity. These perturbations are compared to standards (aliphatic alcohols or more convenient solid standards) in order to determine precisely the ε' and ε" of the sample.

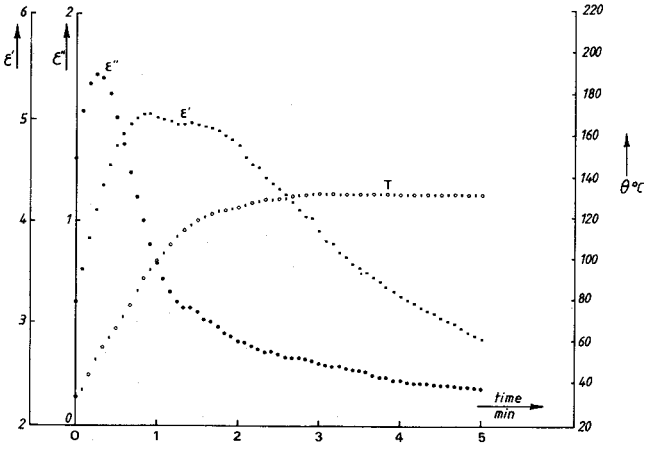

Fig. 3 Heating of 250 microliters of hexanol as displayed on the computer screen in real time: ε', ε" Dielectric constants and temperature in ^{o}C are recorded as a function of time in minutes.

The delicate measurement of the sample temperature (1,2,6) in the resonant cavity during heating has been solved by using a nonperturbing fluorimetric thermometer (LUXTRON) with a silica fiber as temperature probe. This fiber alone supports the sample and measures its center temperature. This method is expected to be more accurate than the classic method which uses an external thermocouple, especially during rapid heatings.

RESULTS

The sample temperature and both dielectric constants are displayed on the computer screen during the analysis as a function of time (fig. 3) and recorded. From these data, dielectric constants may be plotted as a function of temperature (fig. 4). The dimensions (height and diameter) and the weight of the cylindrical sample must be chosen to keep the small perturbation conditions valid. Samples having higher losses require a larger height, smaller diameter and weight. For example, low-loss samples of glass up to 1500mg (h=25mm,d=6mm) have been used, compared to high loss samples of wet paper (roller of about 80 mg.).

For materials with very weak losses, a small electrical furnace heats the sample to a temperature from which permittivities are measured during cooling. Results, such as polymerization kinetics of epoxy resin, will be discussed elsewhere.

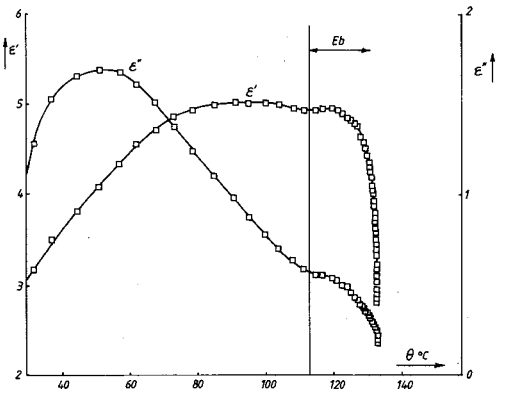

Fig. 4 ε' and ε" Dielectric constants are plotted as a function of temperature - over about $110^{o}C$ boiling of the sample occurs modifying the shape of the sample.

It should be first noted that with the fluorimetric thermometer, the upper limit of temperature is $220^{o}C$. However its replacement by an optical pyrometer should permit higher temperature measurements. Secondly, studies at others frequencies are possible, simply by adding (or removing) multiplier stages to the microwave source and using others cavities with the same basic apparatus.

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