



Real time dielectric relaxation studies of dynamic polymeric systems

Richard A. Pethrick*, David Hayward

*Department of Pure and Applied Chemistry, University of Strathclyde, Thomas Graham Building, 295 Cathedral Street,
Glasgow G1 1XL, UK*

Received 5 November 2001; revised 24 April 2002; accepted 27 April 2002

Abstract

The modern dielectric spectrometer is capable of real time observation of changes in the dipole activity of molecular systems. This article discusses the issues involved in such measurements and their application to a range of problems. Data are presented on the use of dielectric spectroscopy to monitor the cure of thermosetting resin systems, water absorption in polymers, physical ageing in thermoplastics and film formation and coalescence in polymer latexes. The dielectric technique is coming of age and its application to a range of interesting kinetic problems is now possible. © 2002 Elsevier Science Ltd All rights reserved.

Keywords: Dielectric spectroscopy; Epoxy resins; Rubber toughened resin systems; Acrylic latexes; Coalescence; Film formation; Physical ageing; Free volume relaxation; Water absorption; Bound water; Free water

Contents

1. Introduction	1984
2. Development of the modern dielectric spectrometer	1984
2.1. Studies of the cure of reactive resin systems	1986
2.2. Cure chemistry	1987
2.3. Time–temperature transformation diagram	1987
2.4. Optimum conditions for cure	1988
3. Cure monitoring	1989
3.1. Dielectric and electrical measurements of the cure process	1990
3.2. Thermally stimulated discharge (TSD) measurements	1993
4. A molecular interpretation of the dielectric analysis data	1995
4.1. Phase separation in a rubber modified thermoset system	1996
5. Water absorption by polymer materials	2001
6. Physical ageing of amorphous thermoplastics	2005

* Corresponding author. Tel.: +44-141-548-2260; fax: +41-141-548-4822.

E-mail address: r.a.pethrick@strath.ac.uk (R.A. Pethrick).

7. Characterisation of film formation and coalescence in polymer latexes	2006
7.1. Modelling of the dielectric behaviour during coalescence	2008
7.1.1. Ionic conductivity	2008
7.1.2. Interfacial polarisation and blocking electrodes	2009
7.1.3. Dielectric processes associated with conducting inclusions	2009
7.1.4. Dipole relaxation behaviour	2010
8. Conclusions	2011
Acknowledgements	2012
References	2012

1. Introduction

Prior to the early 1980s frequency dependent measurements of the dielectric properties of molecular materials were performed using a range of manually balanced resistance–capacitance and capacitance–inductance bridges [1]. It was not unusual to use three or four different instruments to cover the frequency range 10^{-2} to 10^6 Hz and as a consequence, measurement of the dielectric properties of a polar material over a wide temperature range could often take many weeks. It was therefore impossible to attempt to follow the real time changes in the dielectric properties of materials. Self-balancing bridges operating at a single frequency (typically 1 kHz) were available in the mid 1970s and were used to monitor the dielectric properties of gases and liquids in production environments. The demands of the telecommunications industry in the late 1970s and early 1980s led to the development of the vector network analyser. These computer-controlled instruments were designed to measure the transfer functions over a large frequency range. Various attempts have been made to match the characteristics of these instruments to the requirements of dielectric measurements.

2. Development of the modern dielectric spectrometer

The most accurate method of determining the dielectric property of a material at low frequency (1–100 kHz) is to use a three terminal cell [1] with a transformer ratio arm bridge. In this configuration, a disc of material is sandwiched between two electrodes, one of which is a solid metal disc and the other is composed of a metal disc with a surrounding metal ring. The gap between the disc and the ring will be an air gap or an insulating ceramic or polymer. A sinusoidal voltage is applied to the top electrode and the outer ring of the lower electrode is grounded. The central of the lower electrode is taken to earth through a detector. The balancing of the transformer ratio arm bridge requires a considerable amount of patience and careful calibration at every frequency measured.

All bridge circuits derive the complex admittance of the unknown sample in terms of set of standard admittances, in this case a set of capacitors and resistors. Whether done manually or automatically the process of choosing the correct set of standards requires many cycles of the applied sinewave and leads to prohibitive measuring times at low frequencies. The vector network analyser allows measurement of the unknown without the requirement to balance a bridge circuit. In a low frequency form the simplest direct measurement [2,3] is as shown in Fig. 1.

The analyser supplies a voltage V_X which is applied across the unknown and a sensing resistor R_D . The voltage V_D generated across the sensing resistor is fed back to the analyser which computes the

Download English Version:

<https://daneshyari.com/en/article/5209221>

Download Persian Version:

<https://daneshyari.com/article/5209221>

[Daneshyari.com](https://daneshyari.com)