APPLICATION OF NEW INSTRUMENTATION AND MEASUREMENT TECHNIQUES TO THERMAL ANALYSIS OF MATERIALS

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Principles and operation of the infrared gold image furnace are discussed and its advantages over conventional resistance furnaces for studies of advanced materials listed. Using the gold image furnace in combination with a standard microscope allows continuous monitoring of materials to very high temperatures. New approaches to measurement of heat capacity and thermal diffusivity/conductivity are also described.

Keyword: infrared gold image furnace, semiconductors, superconductors

Introduction

The explosion in materials development has produced substantial quantities of new and improved, but often expensive, products. In many cases the materials have to be produced in special forms and combinations having optimum properties based on specific purities, inhomogenities, heat treatments etc., each requiring that fabrication and characterization be undertaken under especially clean environments and at faster heating and cooling rates than those used in more conventional thermoanalysis.

This has stimulated the development of new thermal technology instrumentation for fabrication and qualitative analysis of materials together with new and modified techniques for determining quantitative thermal properties and performance. In the latter case this has required that measurement be made more rapidly often on special forms and under conditions that cannot be satisfied using appropriate classical methods.

Historically, thermoanalysis and thermal parameter determination were considered distinct and separate tools in development and characterization of materials. The former has been considered primarily as the means to obtain essen-
tial qualitative information on structure and behaviour during development, and
the latter provides necessary quantitative performance values for a material
and/or its applications. Classical thermal analysis techniques used ‘small’ test
specimens and were undertaken rapidly under transient conditions, whereas quan-
titative methods, generally required ‘large’ specimens and were time consuming.

More recently the critical issue became the numbers and types of materials re-
quiring investigation. Results were required on smaller specimens and with high
precision. This was the major stimulus for development of new or modified
methods based on transient techniques for transport properties. As a result of
these stimuli it has been found that some of these newer quantitative techniques
can also be used for qualitative purposes.

The present paper describes two tools developed especially for use in fabrica-
tion and analysis of many new materials. In addition, it includes the essential
principles of several newer methods and gives examples of their use as ther-
moanalytical tools.

Techniques, instrumentation and applications

Infrared gold image furnace

Many ‘new’ materials such as semiconductors, superconductors and com-
posites require fabrication in very clean high-vacuum or gaseous environments.
In some cases particular, precisely controlled temperatures and/or heating, cool-
ing or cycling rates are required. In addition, various applications such as re-entry
from space, and rapid thermal annealing can require that relevant thermal
analysis studies be made at heating rates that are well in excess of those attainable
with conventional resistively-heated furnaces.

These requirements are met by the infrared gold image furnace. This radiation
furnace, combined with a specially designed temperature controller, consists es-
sentially of one or more high-power-density tungsten filament argon of halogen
sealed quartz tubes together with one or more very high reflective, water-cooled,
elliptical, parabolic, or planar gold surface reflectors. These surround an ap-
propriate size quartz tube containing the artifact to be heated in the required con-
taminant-free environment. The result is a compact, low-mass, energy efficient,
maintenance-free system that allows different types, and forms of specimen to be
heated and cooled at heating rates from the conventional 1 to 100 deg min⁻¹ up to
many hundreds of degrees per minute.

A comparison of the features of the gold image furnace with those of conven-
tional resistance furnaces is shown in Table 1. These individual furnaces and
larger systems based on them have found very wide use for many applications.