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INSTRUMENTATION FOR THERMOSONIMETRY

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ABSTRACT

The construction of an apparatus for thermosonimetry is discussed. The detection system is a silica glass waveguide and a PZT piezoelectric transducer. Associated equipment for furnace control and electronic signal processing is described.

INTRODUCTION

Thermosonimetry (TS*) has been defined[1] as "that technique in which the sound emitted by a substance is measured as a function of temperature whilst the substance is subjected to a controlled temperature programme". The technique therefore detects thermally induced acoustic emission, a phenomenon well-known in many fields. For example, in the metallurgical field it has long been recorded[2] that audible "clicks" can be heard during shear transformations of austinitic phases to martensitic phases[3–5] and, in the field of brittle materials, catastrophic failure by thermal stress fracturing (thermal shock) is accompanied by noise emission and has long been a problem in the industrial production of glasses[6, 7] and ceramics[8–10].

Thermally induced acoustic emission can arise either from purely mechanical sources or from physicochemical sources. In the first case, the application of a thermal stress on a solid substance induces mechanical strain within that substance. The release of this strain is normally achieved by the Griffith mechanism of microcrack growth[11] and is accompanied by high frequency acoustic emission[7, 9]. If the applied stress is sufficiently great, catastrophic propagation of Griffith cracks leading to complete fracture may occur accompanied by the audible acoustic emissions which are characteristic of thermally shocked brittle materials. In the second case, the mechanical strain is created by a dynamic process or thermal event, for example a phase transformation[12–15] or decomposition[16], release of fluid inclusions or radiation damage[17, 18]. Such events may lead to thermal fracture but normally do not and are thus not accompanied by audible noise except in exceptional cases such as "martensitic click".

As a thermal analysis technique (as opposed to a thermal shock technique), TS

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* Although not ICTA recognised, the acronym TS will be used throughout this article.

is concerned with the detection and meaning of the various acoustic emissions occurring prior to, during and after thermal events and thereby can offer a contribution to the elucidation of the thermal behaviour of solid materials and to the understanding of the dynamic processes of the solid state. In this article is described a simple, readily built and inexpensive apparatus with which meaningful TS data may be obtained. A second article will deal with temperature calibration, the influence of experimental and instrumental variables and comparative relationships between TS and other thermoanalytical techniques.

APPARATUS

Introduction

The initial development of an apparatus capable of producing a TS curve can be accredited to Smith and Peach[19, 20] who devised a method of recording the rates of decrepitation of crystalline minerals and rocks, such as garnets[21], micas[22] and granites[23]. In this apparatus[18], the sample was heated in an inconel holder attached to one end of a heavy metal sounding tube. The detector was a crystal microphone located at the other end of the tube. The microphone output was amplified and fed to a ratemeter whose output was recorded as a function of time or temperature. Alternatively, the ratemeter output was passed to a decade scaler and accumulated to obtain a smoothed "decrepigraph" or decrepitation rate curve. In addition to detecting emissions during phase transformations and decompositions, it was possible with this

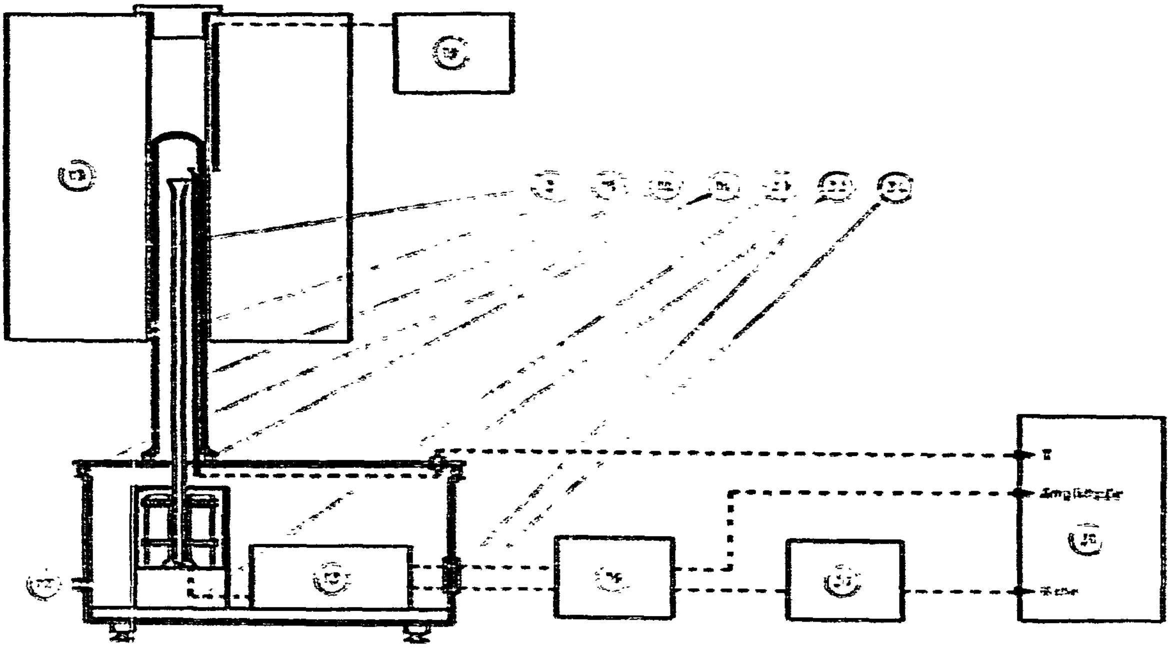

Fig. 1. General layout of thermosonimeter. 1–11, See Fig. 2; 12, preamplifier; 13, furnace; 14, silica sheath; 15, thermocouple (Pt/Pt.Rh); 16, silicone rubber vacuum seal rings; 17, vacuum/gas port; 18, temperature programmer; 19, main amplifier; 20, rate meter; 21, recorder; 22, vacuum lead-throughs; 23, acoustic foam; 24, vibration mounts.

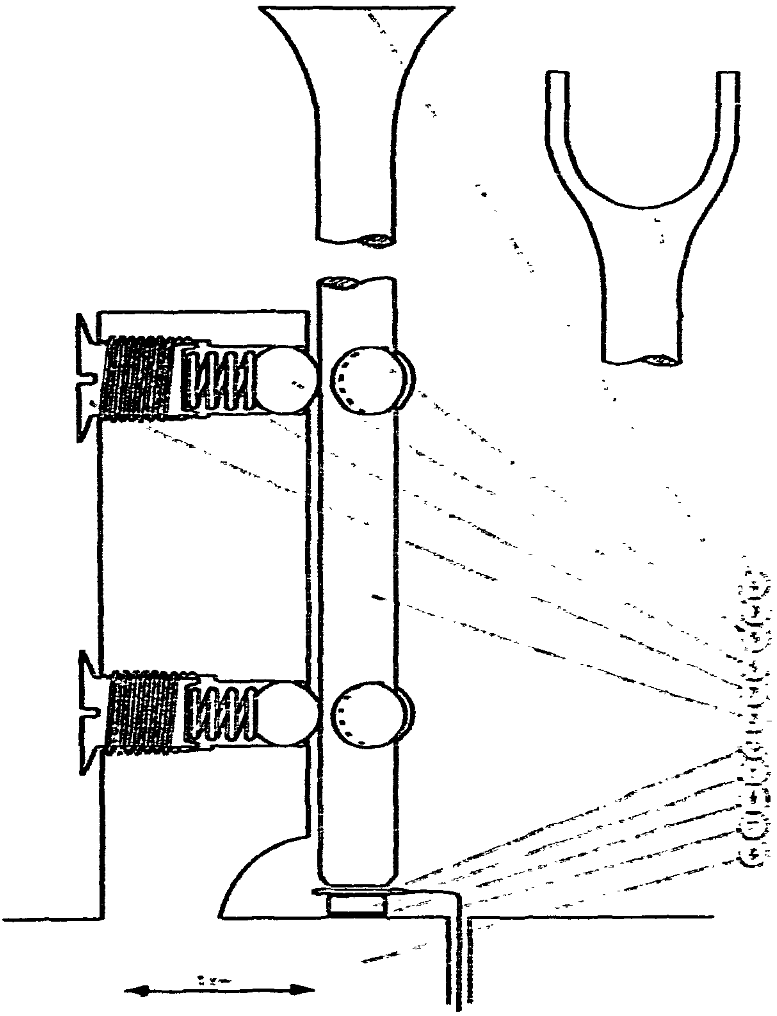

Fig. 2. Elevation of waveguide assembly. 1, PZT transducer crystal; 2, silica waveguide; 3, sample platform; 4, sample cup (alternative to 3); 5, steel ballbearings; 6, tensioning spring (shown for 1 ballbearing only); 7, adjustment screw (shown for 1 ballbearing only); 8, silvered resin; 9, steel base (recoil mass); 10, duralumin foil; 11, lead to preamplifier.

apparatus to distinguish decrepitation due to explosion of fluid inclusions, misfit of solid inclusions, intergrain splitting and radiation damage recovery and to relate the temperatures of these events to other geothermometric and geobarometric measurements[18].

The TS apparatus described in this article is based on a design by Lonvik[24] who has applied the technique in studies of the thermal behaviour of quartzites in relation to ferrosilicon manufacture[25] and to phase diagram studies[26]. A schematic diagram of the basic instrument which has been developed and satisfactorily operated in this laboratory over a period of years is given in Fig. 1. The principal components are (a) the sample holder and waveguide assembly, (b) the piezoelectric transducer detector, (c) the furnace and associated control equipment and (d) the electronic signal processing equipment. Significant features of each are briefly discussed below. A detailed elevation of (a) and (b) is given in Fig. 2.

The waveguide assembly

The signal detection system is based on the use of a sensitive mechanical/electrical (piezoelectric) transducer crystal (1 in Fig. 2). Readily available piezoelectric materials, e.g. $Pb(Zr, Ti)O_3$ (PZT), $LiNbO_3$, $BaTiO_3$ and quartz, mostly have Curie temperatures below 500 °C so that a measuring system which uses direct coupling between the sample and the transducer crystal is applicable only at lower temperatures. Also, the piezoelectric properties of a crystal vary with temperature and recovery of the crystal is not always possible, especially if a thermosetting coupling agent is used. For these reasons, it is preferable to adopt a waveguiding system to transmit acoustic emissions from the heated sample to a transducer crystal at a constant ambient temperature. The couplings between the sample and the waveguide and between the waveguide and the transducer crystal may be permanent (see, for example, the signal detection systems described by Fate[27] and by Hums and Jax[28] in which high temperature cements are used), but non-permanent couplings have operational advantages. Good non-permanent couplings may be achieved by polishing each contact surface and using a thin film of silicone oil to aid signal transfer to the transducer crystal.

The waveguide, 2, requires to be constructed from a material which (i) has a high transmission quality (Q factor), (ii) is readily shaped and polished, and (iii) is thermally stable over the required temperature range. For normal thermal analysis work using conventional furnace technology for the range ambient–1000 °C, silica glass is an ideal material although special ceramics and noble metals have superior long term stability to thermal cycling and are necessary for work much over 1000 °C. Although it is possible to calculate ideal dimensions for a waveguide, in practice these are governed by the nature of the samples to be investigated, the dimensions of the furnace to be used and the design of the waveguide support system. A convenient size is 18–20 cm long, 3–4 mm diameter and smoothly shaped at one end either into a 1 cm diameter polished platform, 3, for sectioned samples or single crystals or into a 1 cm diameter bell-shaped cup, 4, to contain powdered samples.

To ensure efficient transfer of signals from the waveguide to the transducer crystal, the waveguide support system must permit the waveguide to be accurately and repeatably positioned vertically over the crystal. It should also be adjustable (to accommodate different waveguides) and should make minimal contact with the waveguide (to minimise mechanical damping). A proven design uses two sets of stainless steel ballbearings, 5, each set consisting of three ballbearings arranged coplanarly and contacting the waveguide at 120° around its circumference. The two sets hold the waveguide approximately 1 cm and 3 cm from the waveguide/transducer crystal interface. The firmness of contact between each ballbearing and the waveguide is adjusted by means of a small spring, 6, whose tension is controlled by an adjustment screw, 7.

The transducer crystal

The waveguide in its support assembly acts as a damped harmonic oscillator and the dimensions of the transducer crystal should be such as to permit it to resonate at the fundamental frequency of the assembly. Suitable transducer crystals are PZT discs approximately 1 mm × 3 mm diameter. In association with the waveguide assembly described above, an audio range resonance frequency occurs at approximately 1 kHz. Lonvik[24] similarly has found resonance to occur around this frequency.

The piezoelectric disc operates in a thickness expander mode and has its flat faces electroded. One pole is earthed by bonding it with a thermosetting silvered resin, 8, to the steel base, 9, of the waveguide assembly. To the other pole is similarly bonded a thin (0.03–0.10 mm) polished metal foil, 10, on to which the shielded electrical lead, 11, to the preamplifier, 12, is attached. The polished narrow end of the waveguide can then be accurately adjusted by means of the sprung ballbearing support system to rest squarely on the top polished face of the foil. Any metal or alloy with good conduction properties makes a suitable foil but soft metals, e.g. Al and Cu, although readily obtained as thin foils and readily polished tend to become scratched after prolonged use. A thin film of a silicone oil at this interface eases this problem and aids signal transfer, but the use of a harder alloy, e.g. duralumin, provides a better long-term coupling.

The furnace

For work up to 1000°C, a 15–20 cm long nichrome or platinum wound vertical tube furnace, 13, with a bore of about 2.5 cm is satisfactory for use with the 18–20 cm waveguides described above. This permits a 2 cm diameter protective silica sheath, 14, to be inserted between the furnace and the waveguide. A mechanical system for lifting and accurately locating the furnace centrally over the sample is desirable. Thermocouples cannot be placed within the sample because they vibrate, thus monitoring of sample temperature must be performed with a thermocouple, 15, located as close as possible to the sample. A refinement is to build in alongside the TS sample a DTA head and to use the DTA reference thermocouple for temperature measurement. Effecting a seal, 16, at the base of the protective sheath allows gas atmosphere and vacuum work to be performed, 17.

In TS, the furnace operates in close proximity to a transducer crystal and care must be taken in selecting a suitable temperature controller/programmer, 18, since furnaces controlled with conventional on/off controllers can induce switching noise even in a well-earthed and shielded crystal. A fast-cycling zero-switched type of programmer is preferred. A similar range of heating/cooling rates as are used in conventional thermoanalytical techniques is desirable although for thermal shock studies, rates of at least 100 K min^{-1} are required. For normal TS work, a fast-cycling Stanton Redcroft LVP with programme rates up to 40 K min^{-1} is used in this laboratory.

Signal processing

To avoid losses and interferences, signals from the transducer crystal pass along a short well-shielded lead to a charge-sensitive preamplifier, 12, built on to the steel base of the waveguide support assembly. If a vacuum facility is included, a preamplifier such as the Ortec 124/5 is capable of vacuum operation.

Subsequent signal processing depends on the type of measurement and mode of presentation which is required an number of configurations have been described for various acoustic emission studies[4, 5, 15, 24, 29–31]. In general, any of three parameters relating to the emitted noise may be measured as a function of temperature, viz. rate, amplitude (energy) and frequency. Measurements of the frequencies of the emitted noises require complex multichannel analysers and a knowledge of the transfer functions of the sample/waveguide and waveguide/transducer interfaces. However, rudimentary data can be obtained with a transient recorder and the use of simple filters permits frequency ranges to be estimated. The average amplitudes of the emissions are easily measured by passing the preamplifier output (or the transducer crystal output itself) directly to a conventional amplifier and a recorder. Although less easily obtained, rate measurements generally provide more useful information and can be regarded as the normal TS curve. For this type of measurement, the preamplifier output is passed to a main pulse amplifier, 19, and subsequently to a rate meter, 20, supplied with a recorder output. Various modifications are possible. For example, the rate meter may be a scaler or single channel analyser which can provide cumulative rate curves or integrated curves more directly comparable with conventional TG and DTA curves. In this laboratory, the main amplifier is an Ortec 485 and the ratemeter is an Ortec 449 log/lin. A log facility on the ratemeter is especially useful for initial surveys to establish best amplifier gains and ratemeter full-scale ranges and time constants. *X/Y* or *X/T* recording is ideal for TS rate curves but if other data is to be simultaneously recorded, e.g. amplitude, *ΔT*, cumulative rate etc., multipen *X/t* recording is necessary. In this laboratory, a Watanabe Multicorder, 21, is normally used.

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