# Revealing the micro-thermal properties of materials

By Duncan M. Price*

Micro-thermal analysis combines the imaging capabilities of atomic force microscopy (AFM) with the characterisation ability of thermal analysis. AFM is a technique in which a sharp tip is scanned over a surface to build up an image of the surface topography. For micro-thermal analysis the conventional probe is replaced by an ultra-miniature resistive heater (see Figure 1). This also serves as a means of measuring temperature. The tip. when used in conjunction with a reference probe. acts as a differential scanning calorimeter (DSC) cell. Various images of the surface may be generated: the conventional topographic image and images whose contrast is determined by the thermal properties of the surface. The latter are generated from the power required to maintain the tip at a constant temperature and related to the surface thermal conductivity (DC thermal image). Introducing a high frequency temperature modulation gives an image related to the thermal diffusivity of the surface (AC thermal image) whose depth of view into the sample depends on the wavelength of the modulation.

Having imaged the sample. any point in the surface can be selected and the probe tip placed upon it. The temperature of the tip can then be changed in exactly the same way as conventional thermal analysis to obtain calorimetric measurements of transitions.

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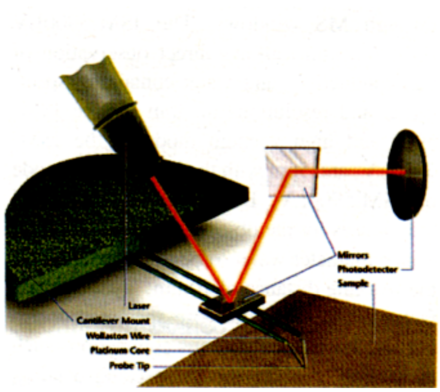


*Fig. 1: Schematic of probe set-up*

In addition. when the tip is placed on a selected point. a carefully controlled force is applied to it. This measurement is closely analogous to thermomechanical analysis (TMA). If the sample should soften as the temperature is increased the probe will indent into the surface. Both the calorimetric and mechanical property measurements are made simultaneously affecting an area only a few microns square. Because of this, high heating rates (in excess of 1000°C/min) can be used.

Figure 1 (left-right) shows the topography, DC and AC (@ 30 kHz) thermal images of a light emitting diode. The scratches in the centre of each image are due to the removal of a contact wire. The two thermal images show marked differences in contrast between the centre of the image and the outside. These differences were investigated by performing localiscd TMA measurements at five different positions on the sample (Fig. 3). The sequence of scans took a few minutes. These show the difference in thermal expansion coefficients

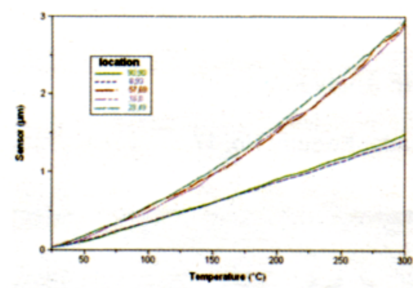


***Fig. 3: Localised TMA measurements from positions on the sample in Fig. 2***

of the component parts of the diode. Interestingly when a second LED was examined no thermal image contrast was observed, although a similar topography was obtained. Localised TMA confirmed that the surface was homogenous and further investigation discovered that this component had been coated with a protective layer. Thus it is possible to image a material and then carry out spatially-resolved thermal analysis in order to characterise it locally.

In addition to the electronics industry. micro-thermal analysis is finding wide applications in the analysis of polymers, pharmaceuticals and biological materials.

More information on the instrument described here can be obtained by email at info@tainst.com, website: www.tainst.com or

Reader Enquiry No. 16.

***Fig 2: Topography, DC and AC images obtained at 30 kHz of a light emitting diode***