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Thermal analysis for the 21st century

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There are three major problems with our current thermal methods. The first is a purely practical one, experiments often take too long, especially thermomechanical measurements. The second is related to sampling; frequently the sample is either too small, or too thin or buried within a larger component from which it is difficult to extract. The third is more fundamental; the information they provide is not spatially resolved.

Atomic force microscopy (AFM) is a technique in which the tip of a probe is rastered over a surface to build up an image of the surface topography. Our apparatus is based on a conventional AFM where the tip of the probe has been replaced by an ultra-miniature resistive heater. The resistance also serves as means of measuring temperature, thus the tip, when used in conjunction with a reference probe, serves as a micro modulated-temperature DSC cell. The tip is held at a constant average temperature and rastered over the sample surface in contact mode to build up an image. The data collected are the topography, as in traditional AFM, plus thermal conductivity, measured from the average (DC) signal plus thermal diffusivity, as measured from the response to the modulation (AC signal). Having imaged the sample, any point in the image can be selected and the probe tip is placed on it. The temperature of the tip can then be scanned in exactly the same way as conventional thermal analysis to obtain calorimetric measurements of transitions. In addition when the tip is placed on a selected point a carefully controlled force is applied to it. As the temperature increases the sample often softens and the probe indents further into the sample. This measurement is closely analogous to a thermomechanical analysis (TMA) measurement. Both the calorimetric and mechanical property measurements are made simultaneously at heating rates in excess of 500°C min^{-1}.

These micro-thermal analysis measurements solve all of the problems outlined in the opening paragraph. It also opens up a new range of applications for thermal methods in polymer science and potentially many other areas such as pharmaceuticals.