# Rapid Determination of Cure Rate and Direct Identification of Spatial Variations in Cross Link Density

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## Abstract

*Transition temperature microscopy (TTM) is a novel local thermal analysis technique that maps spatial variations in thermal properties on length scales from millimeters to nanometers. Traditional bulk thermal analysis provides a sample-averaged result and cannot generally supply sufficient information about complex structures or heterogeneities within polymeric systems. There currently exists a nanoscale thermal analysis (nanoTA) technique in which a nanoscale thermal probe heats a localized region on the sample surface to measure its thermal properties, including thermal transition temperatures like cure, crystalline melting points and glass transitions. TTM enables these nanoTA measurements to be carried out rapidly at a succession of points, thus creating automated high-resolution spatial maps of the thermal properties of a sample.*

## Introduction

The range of material systems undergoing nanoscale characterization is expanding. For some time Atomic Force Microscopy (AFM) has enabled high resolution surface imaging but is limited in specific property analysis. More recent innovation incorporates embedded heating elements into an AFM like probe permitting thermo-mechanical experiments to be conducted with <100nm resolution [1]. Non-AFM based platforms now utilize identical probes for rapid and efficient characterization of thermal events such as cure, melt, and glass transitions of heterogeneous material systems. Distinct advantages are experienced in throughput, surface sensitivity, and lateral resolution.

## Experiment

Nano-thermal analysis (nanoTA) is ideal for polymeric materials which undergo thermal events that are accompanied by a dimensional change or softening. The technique is analogous to a conventional thermo-mechanical apparatus (TMA) [2] where a probe tracks the expansion of a sample while it is heated in a furnace. A fundamental difference from nanoTA is that the entire sample does not need to be heated, rather the micro-machined nanoTA probe heats itself while simultaneously tracking the dimensional response of a sample point <100nm. Softening temperatures can be determined on ultra thin coatings and on sub-micron domains [3] while preserving spatial dependencies lost in bulk instruments.

Cross linking also plays an important role in polymers ranging from mechanical properties, shape memory, to absorbance characteristics. The degree of cross linking can be determined by assessing the penetration depth of a thermal probe in a thermo-mechanical experiment. NanoTA is a localized method that can be used to rapidly determine cross-link density at a multitude of points. Cross-link density is a good indication of cure. With respect to the bulk sample, nanoTA is relatively non-destructive therefore probe penetration can be tracked on the same sample at various stages. Cure may effectively be monitoring in a time resolved manner.

NanoTA also extends into an imaging mode by building a false color map from an array of individual measurements over a user defined sample region. Figure 1 depicts the variations in softening point over an ink drop on polymer substrate. Each pixel is representative of a <100nm point measurement and the color corresponds to the softening temperature at that location. Figure 2 depicts the AFM topography of a single toner particle after microtome with corresponding nanoTA curves attained from different regions within this complex system. Softening temperature is used to identify the various material components and determine where these materials finally reside.

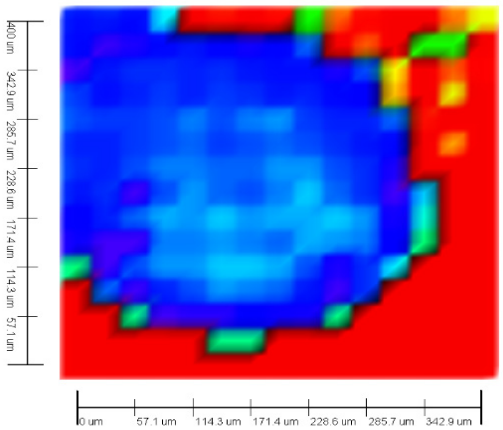


***Figure 1.** A 400x400um TTM of an ink drop on polymer substrate showing variations in Tg by measuring local differences in softening point. Higher Tg [red], lower Tg [blue].*

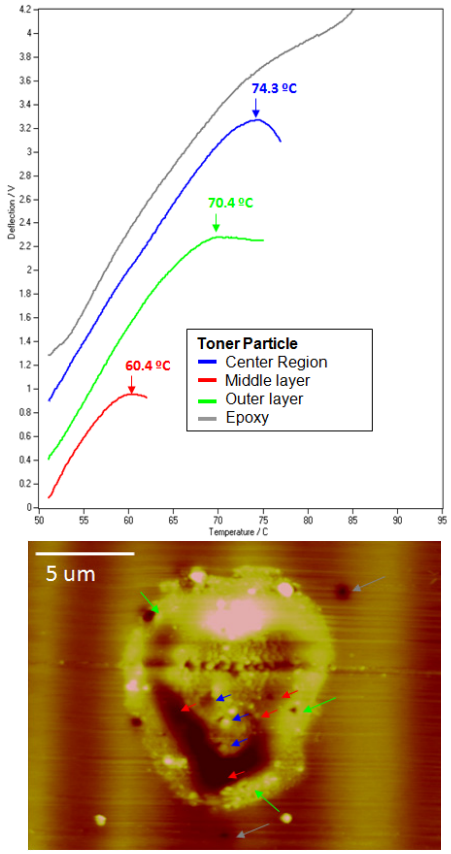


***Figure 2.*** *An AFM image of topography [upper] and corresponding nano-thermal measurements [lower] at various regions within a single toner particle*

## References

[1] W.P. King et al., *Atomic Force Microscope Cantilevers for Combined Thermomechanical Data Writing and Reading,* Applied Physics Letters, **78**, pp. 1300-1302, (2001).

[2] G. Meyers et al., "*Localized Thermal Analysis: From the Micro- to the Nanoscale"*, American Laboratory, 39, pp. 9-14 (2007)

[3] L. Germinario et al., *Nano Thermal Analysis of Polymers, Thin Films, and Coatings*, Microscopy and Microanalysis Conference, (2007).

## Author Biography

*Mr. Khoren Sahagian graduated from the University of California, Santa Barbara with a BS in mechanical engineering. He is an applications scientist at Anasys Instruments and is involved in the design, development and testing of the nanoscale thermal analysis technologies and products and currently focuses on applying the breakthrough Transition Temperature Microscopy (TTM) technique to samples in different applications. In 2008 he was awarded the R&D 100 for the 100 most significant innovation of the year.*