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Thermal Analysis by Structural Characterization - TASC

- The Basic TASC Algorithm for Glass Transitions and Melting
- 3D TASC
- Thermal Dissolution Analysis
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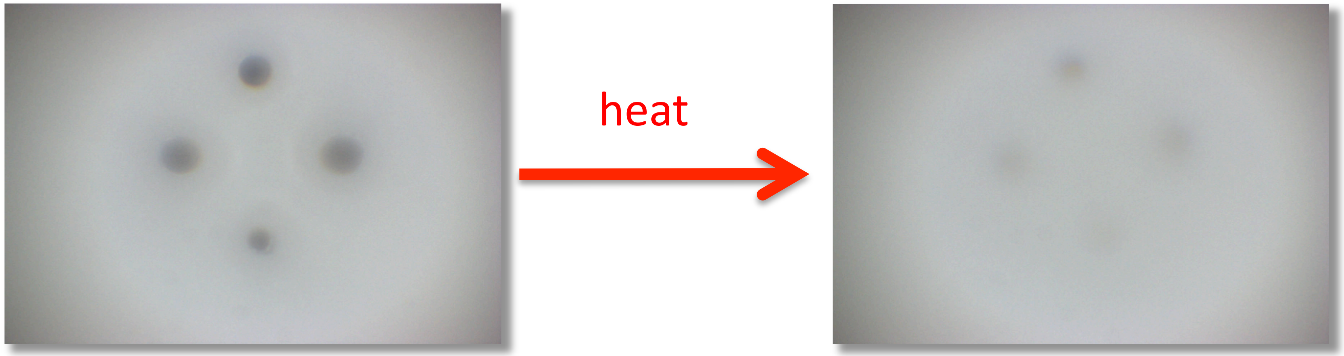
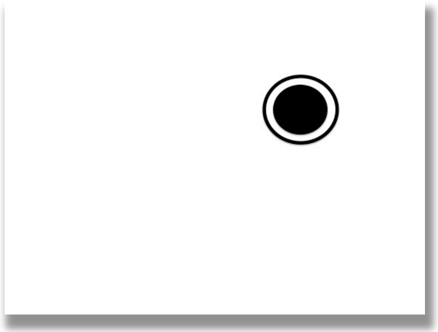
The Basic TASC Algorithm for Glass Transitions and Melting

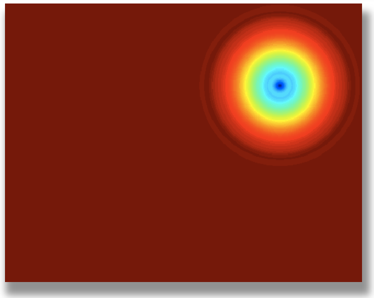
TASC consists of imposing a pattern on the surface of a sample or exploiting pre-existing structure, then characterizing how that pattern changes as the sample is heated; in this case Optical Microscopy was used but it can be applied to other forms of microscopy such as electron microscopy and Atomic Force Microscopy.

Patterns can be of many different types, in the example below we have used four indentations in a sample of filled polystyrene. Having more than one indentation allows multiple measurements to be made at the same time thus averaging is possible. Also there are indentations of different sizes thus the effect of the size of the feature can be examined. In this example, as the sample is heated above its glass transition the indentations disappear and this is detected by the TASC algorithm.

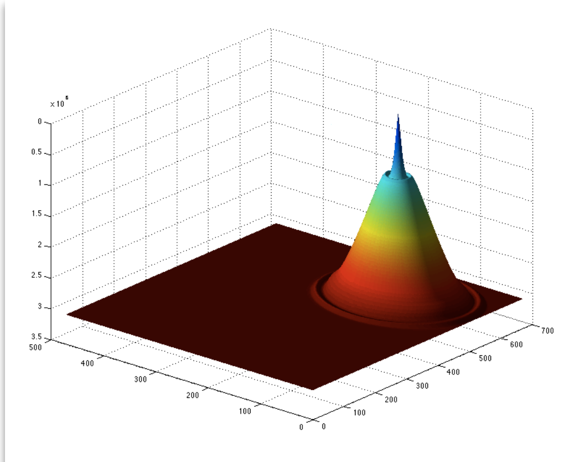


The TASC algorithm scans an area and tries to identify whether a designated structure exists and where it is located. **Top right** there is a schematic of an indentation, underneath this is the result of a TASC analysis. It locates the indentation and provides a measure of the difference between the target shape and the measured structure. In this case the difference is low, i.e. the target shape is identified, at the dark blue center of the colored circle. In this way the structure is located and its similarity to the target structure is quantified.

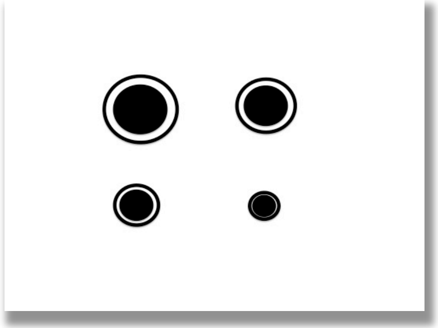


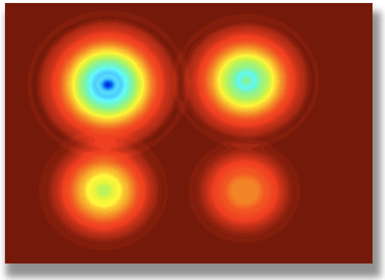


Right is a 3D representation of the output of the TASC analysis. The apex of the cone provides the location of the feature

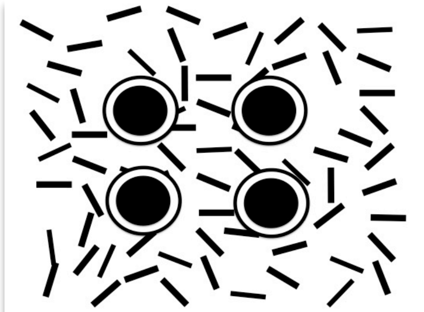


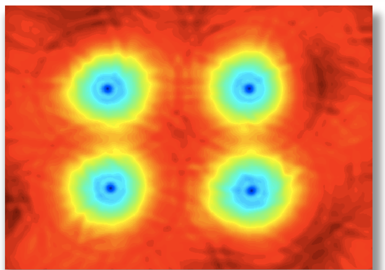
Above right is shown a series of schematic indentations of decreasing size. The degree of recognition by the TASC algorithm decreases as the size of the indentation decreases i.e. the difference between the target structure and the background becomes smaller. In this way the target is both located and its change in size is quantified.



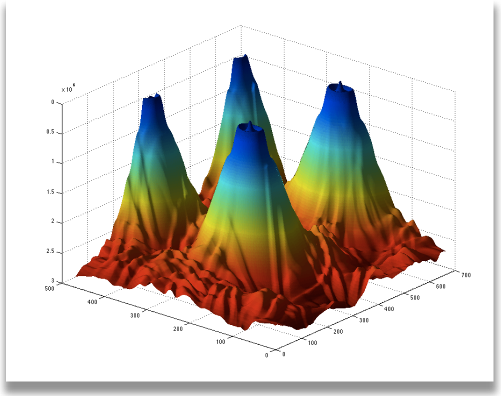


It is important that the algorithm is robust because it must deal with non-ideal samples. **Above right** there is a schematic of 4 indentations in a 'noisy' background. Below this is the result of a TASC analysis; it can still identify and locate the indentations.

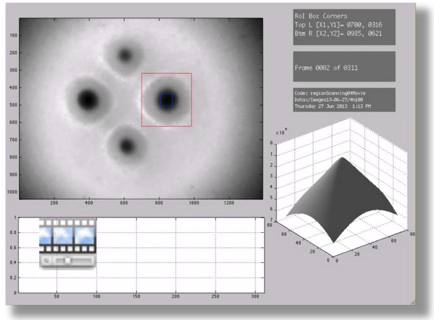




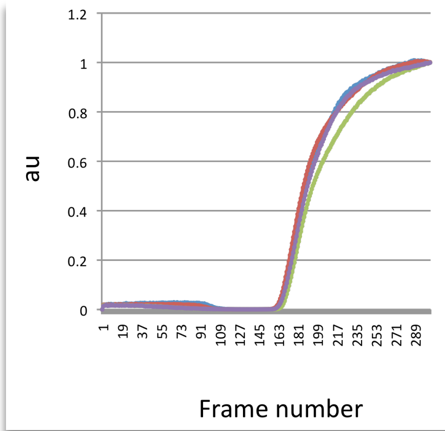
Right is a 3D representation of the output of the TASC analysis with the noisy background. The apexes of the cones provide the locations of the target structure.



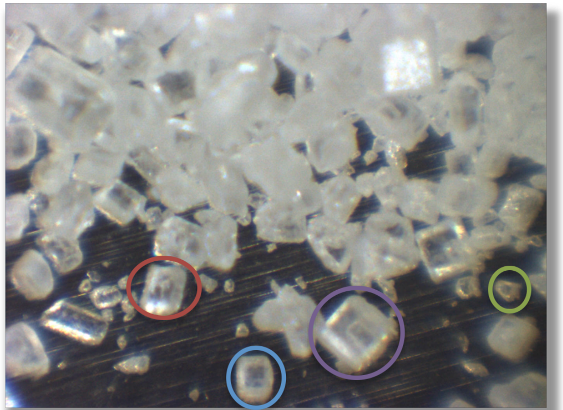
Press on the image **right** to see a video of the TASC analysis on the Cyversa website www.cyversa.com



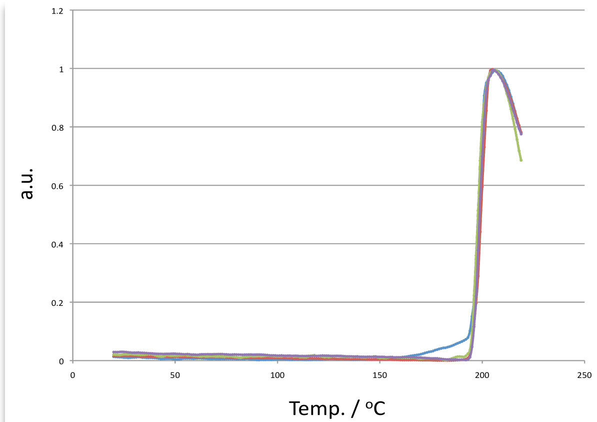
Right is shown the results for the four indentations shown above in filled polystyrene. The repeatability is good and the onset is the same for all four measurements.



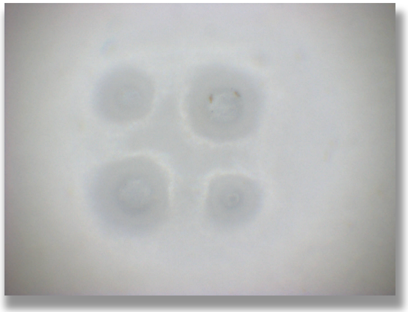
In the example given above a featureless sample has a pattern imposed on it and it is this structure the TASC algorithm uses. It is also possible to use intrinsic structure. In the image **right,** a sucrose sample is shown. The sample was heated and the crystals surrounded by the colored circles were analyzed with the TASC method.



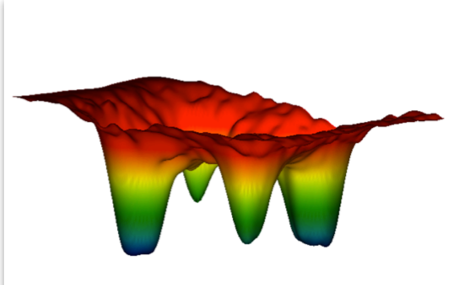
Right are the results. Again good repeatability is achieved with the same onset in all cases.



3D TASC

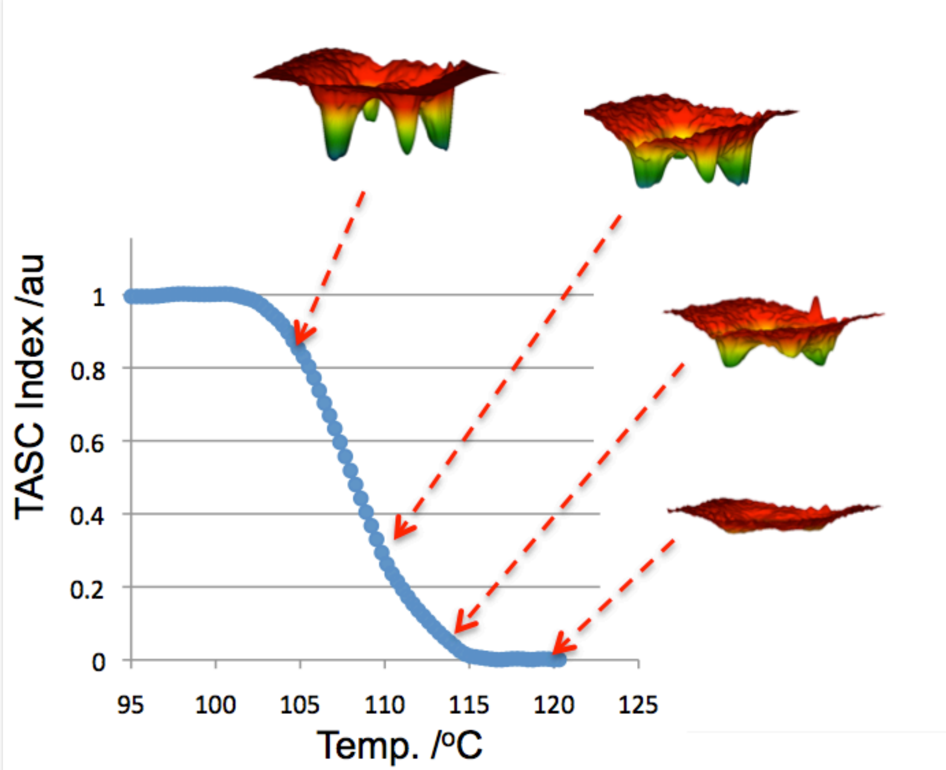


Left is the optical image of a pattern of four indentations in a sample of filled polystyrene, the image has extended depth of focus derived from multiple images with different focal planes, a technique commonly referred to as z-stacking. **Right** is the 3D image created from the same images using suitable mathematical processing.



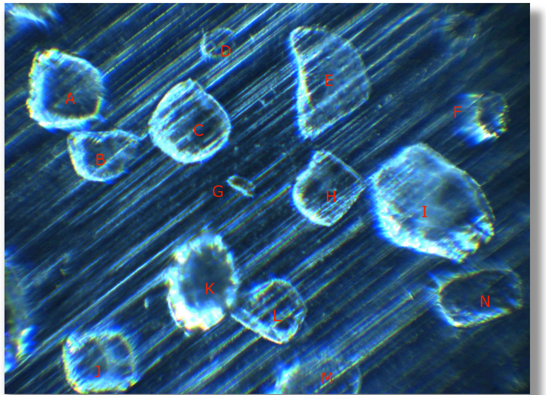
Right is a plot of normalized volume change along with the 3D images used to calculate this quantity.

The TASC algorithm can be applied to 3D images when sufficient are obtained during a single experiment. Alternatively, the 3D data can be used to calibrate the 2D TASC. In both cases a more quantitative measurement of flow properties can be obtained.

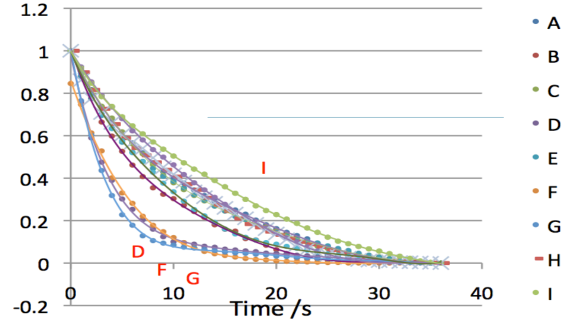


Thermal Dissolution Analysis

Right is an image of a collection of sugar crystals in water in a DSC crucible. The TASC algorithm can follow their disappearance as they dissolve; a video of this process can be seen by clicking on the image on the Cyversa website www.cyversa.com. Plots of the dissolution of these crystals are shown **below right.**



In the graph shown **right**; it can be seen that small crystals D, F and G disappear much faster than the large crystal I. The temperature is clearly a crucial parameter in determining dissolution rate and this is carefully controlled by the Hot Stage. **This is called Thermal Dissolution Analysis or TDA**.

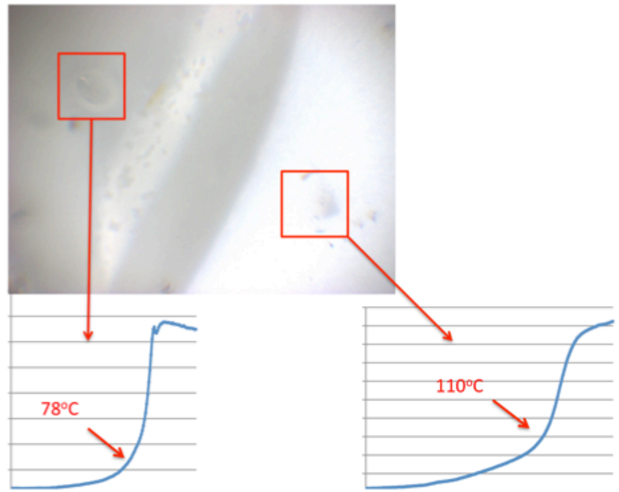


The data obtained by TDA can then be analyzed in a variety of ways, see Application to Pharmaceuticals for an example. Dissolution at different temperatures in different environments with different distributions of crystal sizes can be studied to obtain a complete picture of dissolution kinetics.

Local Thermal Analysis (LTA)

Right shows two TASC measurements made at different points on the sample shown in the image. The locations are indicated by the red squares. It can be seen that the transition temperatures at the different locations are different thereby demonstrating that these are different materials. The scale of scrutiny depends on the magnification and the size of the indentations made in the sample.

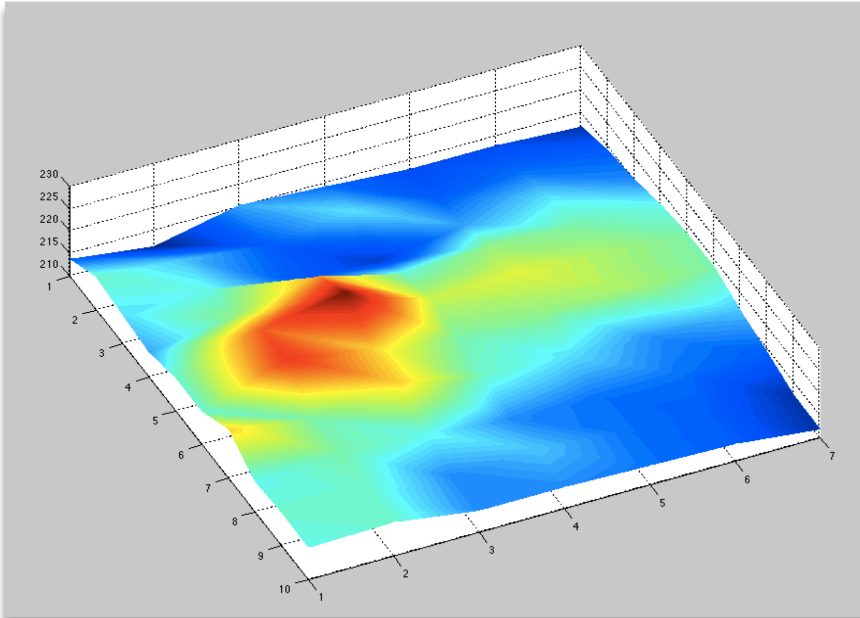
An extension of this called **T-Map** enables images to be created on the basis of transition temperatures, see below.



The TASC method for LTA uses a uniform temperature field, this means that all parts of the sample have the same temperature. This ensures that the thermal history of the sample during the experiment is known and that all temperature measurements are accurate. This is an advance over the first generation LTA approach which uses **a heated probe because this imposes a highly non-uniform temperature fiel**d on the sample. This difference means that TASC can achieve better accuracy for transition temperatures and interpretation is easier because the thermal history at each location is known.

Transition Temperature Mapping: T-Map Mode

In some cases it is possible to make a series of TASC measurements in a grid pattern to create an image based on transition temperatures. **Right** is a 3D image created by mapping transition temperatures for a sample of Zoledronic Acid; see TASC Applications for Pharmaceuticals for more details.



Transition Temperature Histograms

The T-Map data or simply a multiplicity of TASC measurements on individual crystals or regions of a sample can be used to create a histogram (**right**) of the distribution of transition temperatures that characterizes the inhomgeneity of the sample. See Applications Pharmaceuticals for more details.