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DVS Application Note 10

Direct Visualisation of Moisture-Induced Morphological Tranformations using the DVS Video Microscope

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DVS is now established as a useful analytical tool in many R&D laboratories throughout the world. This application note describes the integration of DVS with a colour video microscope for the in-situ visualisation of moisture induced morphological transformations, which may be correlated with the gravimetric data.

Introduction

Figure 1 shows a schematic of the essential video interface hardware in the DVS video microscope instrument developed by SMS for the simultaneous measurement of both gravimetric and visual moisture induced changes using a DVS-Advantage instrument.

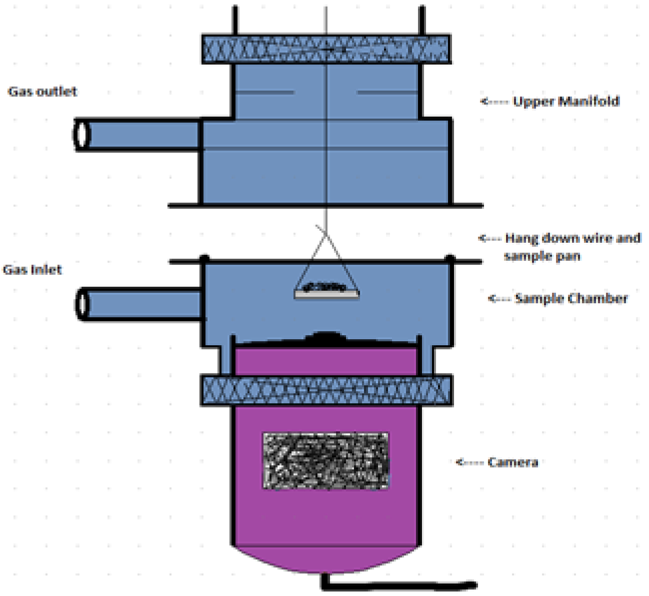

Figure 1. Schematic of the DVS Video Microscope.

The basic principal of the design is similar to a standard DVS gravimetric analyser, with both the sample and reference sides being symmetrical in design. The major difference in this case is that a colour digital video microscope is mounted directly below a flat-bottomed quartz sample pan. The video microscope is interfaced to the microcomputer controlling the instrument with a USB port connection. This allows images of the sample to be taken manually or automatically via the instrument software. Colour images with a resolution of 1280 x 1024 may be obtained with a magnification as high as 200X. Images may be acquired at fixed time intervals or at the end of each programmed step in the vapour method being used.

The vapour methods provide a check box that is selected if an image is desired for a particular step. These images may be saved in a variety of formats, including JPEG, TIF, MPEG, BMP and others. It is also possible to use these images to create a time-lapse movie of the experiment. Additionally, the images may be used with freely available third party software to enhance features using false colour and filters. A grid pattern may be super-imposed on the images for detailed measurements when calibrated with the provided reticle.

Method

Dynamic vapour sorption (DVS) is a well-established method for the determination of vapour sorption isotherms. The DVS-Advantage 1 instrument used for these studies measures the uptake and loss of vapour gravimetrically using a SMS UltraBalance with a mass resolution of ±0.1 µg. The vapour partial pressure around the sample is controlled by mixing saturated and dry carrier gas streams using electronic mass flow controllers. The temperature is maintained constant at 25 ±0.1 °C.

Lactose was used as a model pharmaceutical material. Amorphous lactose was obtained by spray drying. The amorphous lactose was stored over desiccant at 6 °C, to limit any spontaneous recrystallization.

Results

The instability of amorphous spray dried lactose at high humidities is well documented and has been characterised by both DVS and differential scanning calorimetric (DSC) techniques [1]. Figure 2 shows humidity ramping data for 100% spray dried amorphous lactose at 25°C from 0% RH to 90% RH at a rate of 2% RH/hour. Figure 3 shows DVS video microscope images of 100% spray dried amorphous lactose taken during the same ramping experiment at the relative humidities indicated by the corresponding letters on the gravimetric data.

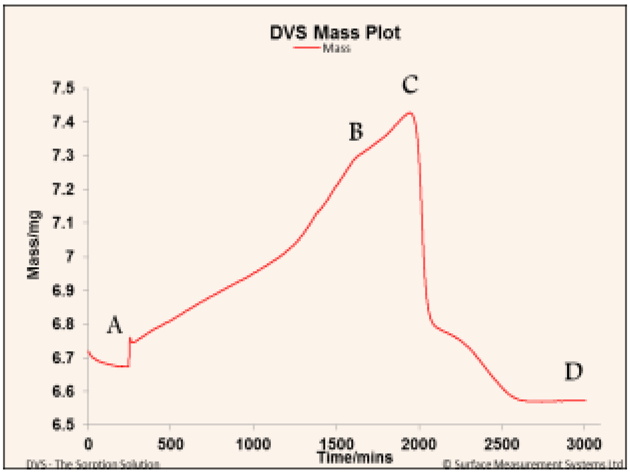

Figure 2. Schematic of the DVS Video Microscope.

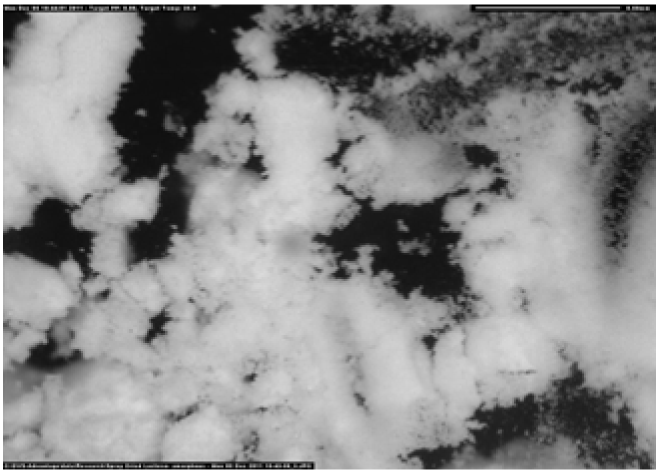

Figure 3a

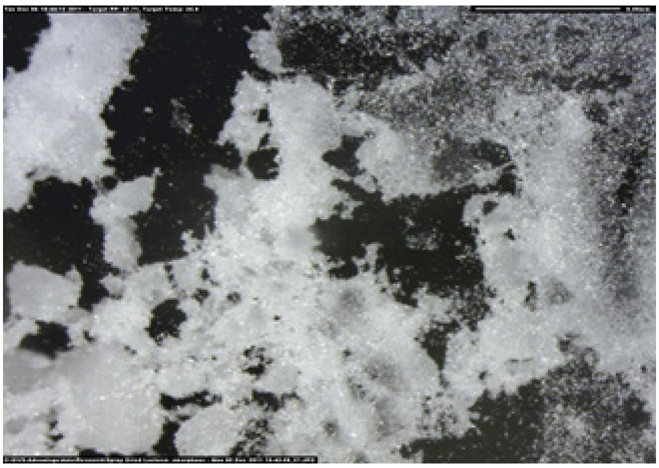

Figure 3b

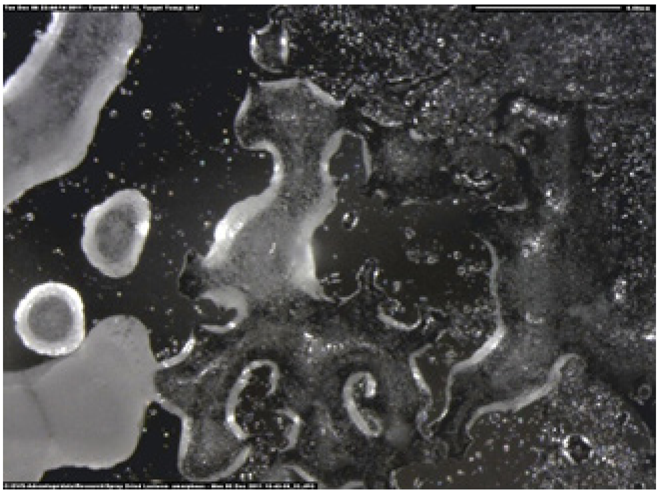

Figure3c

Figure 3d

Figure 3. DVS images for 100% spray dried amorphous lactose during humidity ramping at(a) 0% RH, (b) 50% RH, (c) 60% RH and (d) 90% RH.

The DVS video microscope images clearly show the transformation of the amorphous spray dried powder (a) into a glassy form at 50% RH (b) due to a moisture induced lowering of the glass transition temperature (Tg) of the amorphous phase [1]. At 60% RH, the sample mass reaches a maximum at which point it is clear that the sample has sorbed substantially and has taken on an slightly opaque appearance (c). The former observation is due to the formation of a highly mobile glassy state whereas the latter is probably due to initial precipitation of minute lactose crystals. At 90% RH precipitation of solid lactose is evident by an increase in the opacity of the image (d) and corresponds with the large moisture loss associated with the formation of crystalline α-lactose monohydrate in the gravimetric data.

Below are shown some other examples of images taken with the DVS Video Microscope. JPEG format was used to reduce the size of the document. Higher resolution formats are available however. In Figure 4 is shown an image of Maltodextrin crystals at 80% RH and 200X magnification. Several images of this sample were taken until the sample deliquesced. The images were combined into a 'movie' which shows the progression of water sorption and crystal solvation. This movie is available upon request.

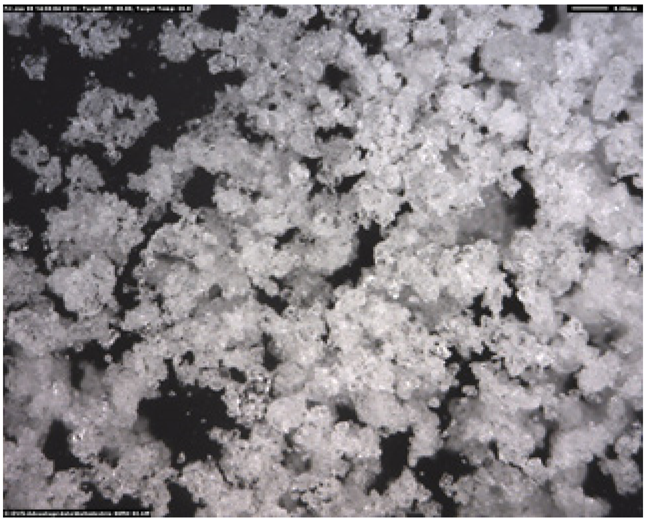

Figure 4 – Maltodextrin crystals at 200X, 80% RH.

Sucrose crystals were imaged at 95% RH as shown in Figure 5. Here too several images were taken as the crystals adsorb moisture. The dark areas on the crystal facets show water condensation between the crystal and the sample holder.

Carbon fibers are imaged in Figure 6 at 200X manification and 0% RH. A grid pattern is overlayed on the image. The software allows for calibration of this grid. In this case the grid squares are 0.5 mm on each side.

In Figure 7 we see a sample of MgCl2 recrystallized after deliquescense. The false color in the image is added using third party software which is freely available on the internet. Crystal orientation is highlighted by the false color.

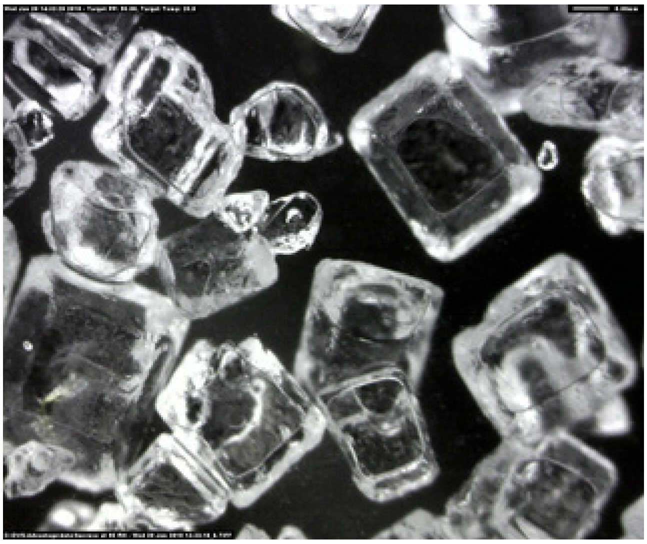

Figure 5 – Sucrose crystals at 200X, 95% RH.

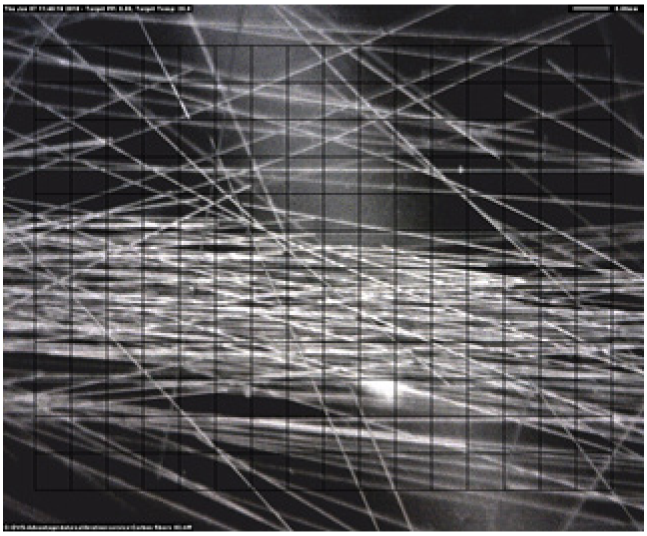

Figure 6 – Carbon fibers at 200X, with grid overlay.

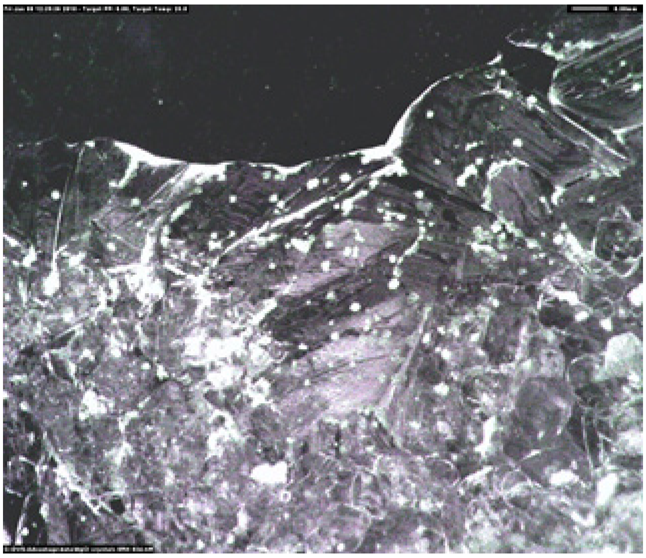

Figure 7 – MgCl2 recrystallized, false colour image highlighting crystal orientation, 200X, 0% RH.

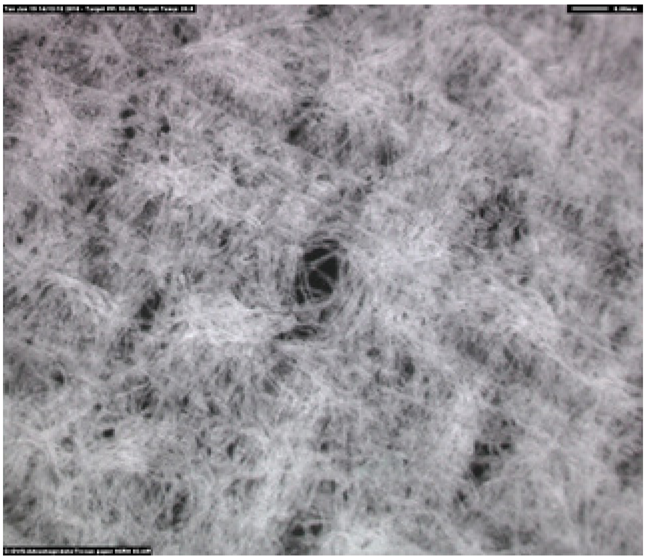

Figure 8 – Tissue paper 200X, 95% RH, showing fiber organization.

A sample of tissue paper at 200X and 95% RH is shown in Figure 8. This image clearly shows the fiber orientation and the weave pattern.

Another color enhanced image is shown in Figure 9, which is a sample of Berea Sandstone. The individual crystallites are highlighted by the color in this image, also taken at 200X manification. Figure 10 is the same sample in black and white.

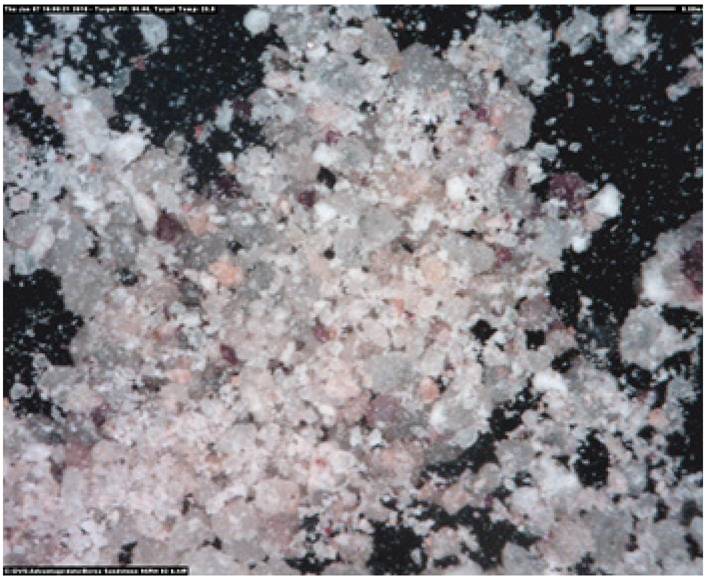

Figure 9 – Berea Sandstone 200X, 95% RH, showing individual crystals. False color enhanced.

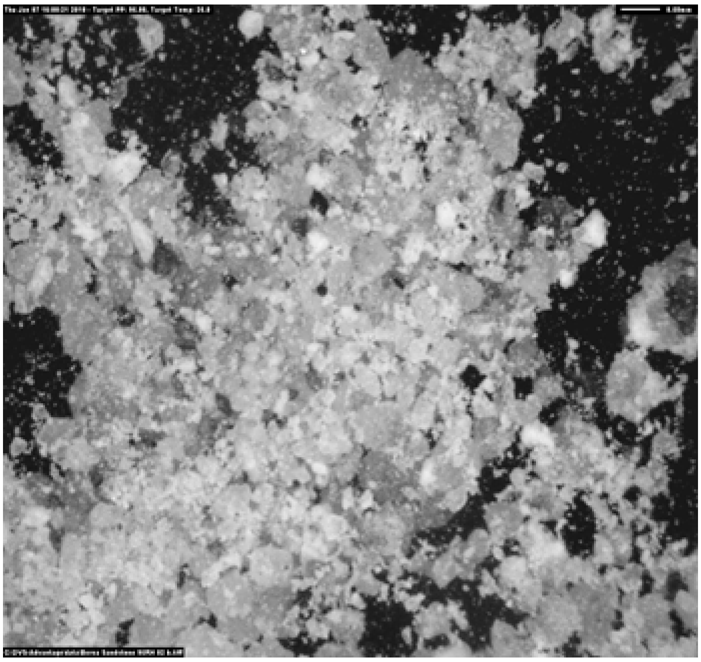

Figure 10 – Berea Sandstone 200X, 95% RH, black and white image.

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Conclusion

This application note therefore demonstrates the power of in-situ video microscopy in an automated gravimetric adsorption system for the correlation of visual morphological changes in the sample with gravimetric data.

Acknowledgement:

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References

1 Buckton G. and Darcy P., Proc 1st Wld. Mt. APGI/APV, Budapest, 1995.

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