Technology Update

The Second Generation Modulated 3D Technology

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Introduction

In 2000 LS Instruments revolutionized the world of light scattering by introducing the 3D LS Spectrometer, the first commercial implementation of the 3D Cross-Correlation spectroscopy technology. This allowed SLS/DLS practitioners for the first time to measure samples without worrying about artefacts arising from multiple scattering effects. In 2010 we further improved the 3D LS Spectrometer by developing the patented Modulated 3D Technology. Building on 3D Cross-Correlation, it improves performance in terms of signal to noise ratio and maximum measurable sample concentrations. We are now proud to announce the implementation of a second generation Modulated 3D Technology whose benefits are illustrated below.

The 3D Cross-Correlation and Modulated 3D Technologies

Simultaneous Static (SLS) and Dynamic Light Scattering (DLS) [1] represent the technique of choice for rapid in-situ characterization of submicron dispersions. In its standard implementation the SLS/DLS technique allows for the determination of the radius of gyration, the molar mass, the second virial coefficient, the angle dependent hydrodynamic radius and the diffusion interaction coefficient of the sample under investigation as long as no multiple scattering is present. This means that the scientist must perform a time consuming set of measurements on a dilution series of the original sample, in order to ensure the absence of multiple scattering and the validity of the final results of the measurements.

The 3D Cross-Correlation technology [2,3], as implemented in the 3D LS Spectrometer, consists in simultaneously performing two light scattering experiments in a specific geometrical arrangement, a so-called 3D geometry. This results in the suppression of multiple scattering and assures the validity of the measured quantities as long as some singly scattered light is still present. This comes however at the cost of a fourfold reduction of the signal to noise ratio of the measurement.

The Modulated 3D technology [4], offered as an option for the 3D LS Spectrometer, solves the problem of signal to noise ratio reduction by performing the two light scattering experiments alternately at a very high frequency. In its technically challenging implementation the technology mainly consists of an electronic modulation board that drives two laser modulators to alternately switch on and off the input laser beams. At the same time, it synchronously and alternately rejects the photon signals detected by two photon detectors before being fed to the correlator.

The main challenges posed by the Modulated 3D technology can be summarized as follows:

- strict timing stability requirements of the modulation board
- synchronization between the electronics board and the correlator used to compute the correlation function
- finite switching time of the laser modulators

The last point has the main impact on the performance of the 3D Spectrometer. The switching time poses a lower limit to the modulation period, which also represents the shortest lag time of the measured correlation function. As a result, slow switching times limit the fastest colloidal dynamics effectively measureable. Furthermore, slow switching times result in slower transitions between the two light scattering experiments during which detected photons must be rejected. Hence the more the modulation half period approaches the switching times, the larger the undesirable reduction of the measured scattered intensity.

Second Generation Modulated 3D: Performance Improvements

In its first implementation, the modulation board was operating at a fixed modulation period of $\tau_M = 3200\ \mathrm{ns}$, and no synchronization was possible with the third party OEM correlator adopted in the 3D LS Spectrometer. After an intense development phase LS Instruments completely redesigned the modulation board and developed its own correlator called the LSI Correlator [5]. This allowed LS Instruments to achieve the synchronous operation of the two boards and the possibility to select the modulation period, thus addressing the main limitation of the first generation implementation. To illustrate the resulting performance improvement, we conducted intensity measurements as a function of the modulation period. In Figure 1 the solid blue circles represent the measured intensities for the 2nd generation modulation system, whereas the black solid circle represents the value measured for the 1st generation unit obtained in the same experimental conditions. We noticed that for the modulation period used in the 1st generation implementation (vertical yellow line) we almost doubled the measured scattered intensity. On the other hand, in order to obtain the same scattered intensity as in the 1st

generation implementation (horizontal yellow line), we can modulate five times faster in the 2nd generation unit than in the 1st generation.

To cast these improvements into performance figures closer to a real world application, we use the setting of a DLS particle sizing task as an illustrating example. In order to achieve a given statistical accuracy in a measurement, an increase of the scattered intensity results in a reduction of the measurement time by the same factor. On the other hand, by decreasing the modulation period we can reduce the smallest measurable hydrodynamic radius by the same factor.

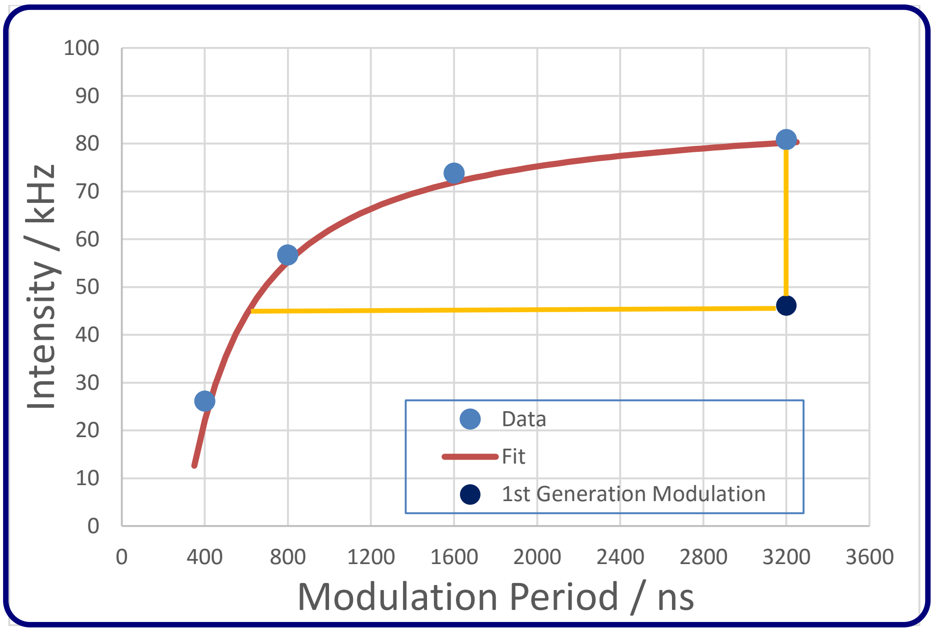

Figure 1. *Measured scattered intensity as a function of the modulation period for the 2nd generation modulation unit (solid blue circles) and for the 1st generation unit (black solid circle)*

With this example in mind, the data collected can be recast in terms of measurement time reduction versus smallest measurable size reduction as illustrated in Figure 2. Here we can appreciate how the second iteration of the Modulated 3D technology delivers a fivefold

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reduction of the smallest measurable particle size and an almost twofold reduction of the measurement time.

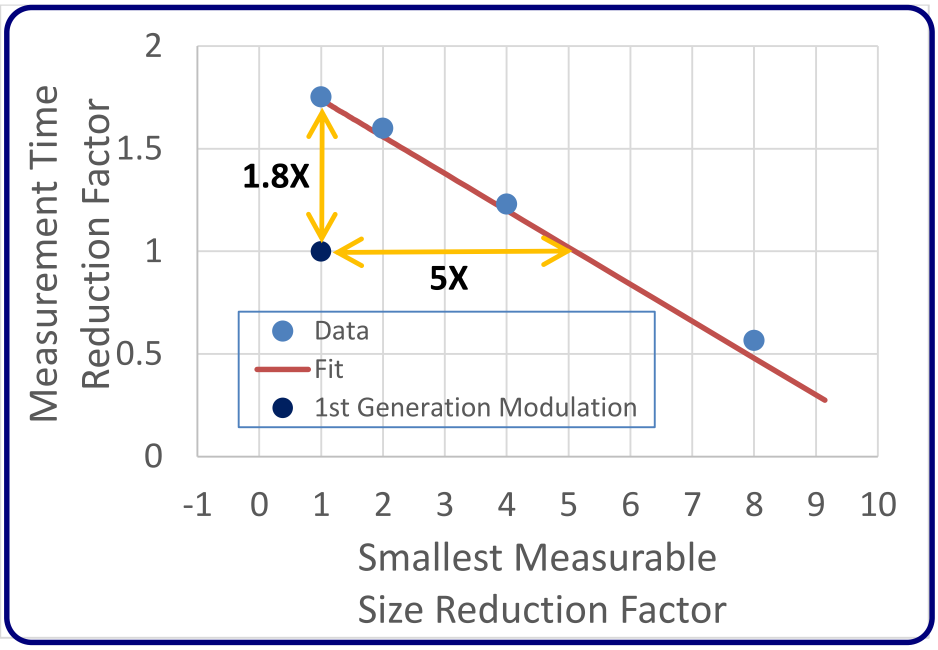

Figure 2. *Example of a possible the real word performance improvement for a DLS particle sizing task obtained by switching form the 1st generation 3D Modulation technology to the latest 2nd generation*

Conclusions

While our patented Modulated 3D Technology resulted in a significant increase of the signal to noise ratio in 3D cross-correlation experiments, it also introduced some trade-offs. The 1st generation implementation caused an increase of the smallest detectable hydrodynamic radius, and a decrease of the detected scattering intensity. These limitations were primarily due to the initial design of the modulation board and the OEM correlator previously used for the 3D LS Spectrometer. Based on a rigorous electronics redesign and the development of the LS Instruments advanced correlator board, we can now offer a 2nd generation Modulated 3D Technology ready to be used by our customers. This results in a major improvement in the size resolution of our unique suite of instruments that implement 3D cross-correlation technology at the same time reducing the measurement times.

References

[1] S. Bantle, M. Schmidt, W. Burchard, *Simultaneous static and dynamic light scattering*, Macromolecules **15**, 1604 (1982).

[2] K. Schätzel, *Suppression of Multiple Scattering by Photon Cross-correlation Techniques*, J. Mod. Opt. **38**, 1849 (1991).
[3] C. Urban and P. Schurtenberger, *Characterization of Turbid Colloidal Suspensions Using Light Scattering Techniques Combined with Cross-Correlation Methods*, J. Colloid Interface Sci. **207**, 150 (1998).
[4] I.B. Block, F. Scheffold*, Modulated 3D cross-correlation light scattering: Improving turbid sample characterization*, Rev. Sci. Instrum. **81**, 113103 (2010).
[5] The LSI Correlator is sold by LS Instruments as standalone product as well. For more information about its features please visit its internet page at this address http://www.lsinstruments.ch/products/lsi_correlator/

Scan the image below with your mobile device to get more information about the 3D LS Spectrometer:

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