Journal of Neutron Research 19 (2017) 23–26
DOI 10.3233/JNR-170037
IOS Press

Dynamic infrared sample controlled (DISCO) temperature for the tumbler cells for ultra small angle neutron scattering (USANS)

J.B. Leão [a,*], R.P. Murphy [b], N.J. Wagner [b] and M. Bleuel [a,c]
[a] *NIST Center for Neutron Research, Stop 8562, Gaithersburg, MD 20899, USA*
[b] *Department of Chemical and Biomolecular Engineering, University of Delaware, Newark, Delaware 19716, USA*
[c] *Department of Materials Science and Engineering, James Clark School of Engineering, University of Maryland, College Park, MD 20742, USA*

Abstract. In this paper a method to control the temperature for a rotating Ultra Small Angle Neutron Scattering (USANS) sample holder is introduced. The temperature is controlled via halogen light bulbs and an infra-red (IR) sensor to avoid interference with the rotation.

Keywords: USANS, rotating sample holder, temperature control, colloidal suspensions, thermoresponsive

1. Introduction

As USANS experiments target structures in the micrometer regime, sedimentation during the measurement can be an issue for liquid samples. In some cases, it is possible to compensate the density difference between the particles and the solvent in order to avoid sedimentation, for example by changing the H_2O/D_2O-ratio, but often this option will lead to an unwanted reduction of the scattering contrast of the sample. Moreover, colloidal suspensions, gels, and flocs that contain large particles, networks, or aggregated structures often require sample rotation throughout a scattering measurement to prevent gravitational settling when the solvent and particle densities differ significantly. Furthermore, the microscopic and macroscopic properties of colloidal suspensions can depend strongly on temperature.

Addressing the sedimentation issue of colloidal suspensions, Anders Olsson et al. [3] developed a sample holder which fits standard SANS cells used at the NIST Center for Neutron Research (NCNR) and tumbles/rotates at variable speed continuously around the beam axis. A sample changer with four rotating sample positions (4R) were each position is capable of independent rotational speeds. Currently one is on loan from the Upsala University in Sweden and available for users at the NCNR. The original design includes a temperature control with Peltier elements, which works very well in the temperature range starting at about 15°C up to 40°C. Nevertheless, the uncertainty of the actual sample temperature due to the location of the sensor and the rotation of the sample holder needs to be addressed.

The Dynamic Infrared Sample Controlled (DISCO) temperature environment presented here (see Fig. 1) extends the temperature range towards higher temperatures with a maximum temperature of currently about 80°C. This new

*Corresponding author. E-mail: juscelino.leao@nist.gov.

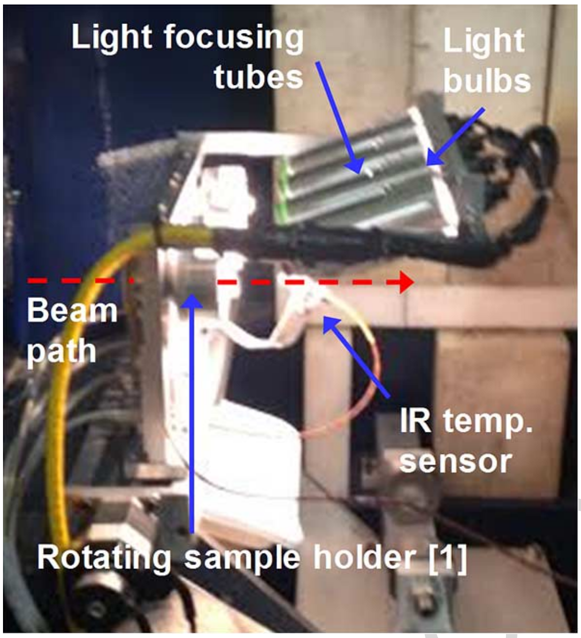

Fig. 1. Photo of the 4R sample holder with the DISCO temperature environment installed at the USANS beamline [1] at the NCNR.

setup also reads the temperature directly at the sample position. The Infrared (IR)-sensor for this readout shows a bias at low temperatures, but once the sample temperature is above 30°C the accuracy of the temperature controller (and thus the temperature control) is better than 0.5°C. Thus, scattering measurements with the DISCO–USANS environment acts to limit sedimentation by continuous sample rotation and acts to control sample temperature by infrared heating with feedback control.

2. Technical setup

Four 75 W, 120 V halogen incandescent light bulbs driven by a silicon controlled rectifier (SCR) power controller were mounted in a variable angle frame and each fitted with cylindrical aluminum tubes. The cylindrical tubes focus the light and allow for effective illumination of the entire sample holder as mounted onto the rotating stage. Once properly aligned, the infrared heat from the halogen bulbs impinges onto one of the faces of the sample holder thus heating the entire cross section of the sample holder. Temperature homogeneity across the sample surface area is ensured by the conduction heating path along the neutron beam direction.

An infrared thermocouple mounted below the rotating stage is used for temperature control. The thermocouple used was emissivity calibrated by using a platinum sensor mounted inside the sample holder and contained within a viscous medium with thermal conductivity of 4.1 W/m K and thermal impedance of 0.81 (°C) cm^2/W. The temperature offset between the two sensors were well within 0.5°C, though the accuracy of the platinum sensor is better than 0.3°C and the accuracy of the infrared sensor (±2% of nominal range).

3. Results

An example of the DISCO–USANS utility is shown in Fig. 2 for a temperature-sensitive colloidal suspension that is susceptible to sedimentation on the timescale of several hours [2]. The sample is composed of coated silica rods suspended in tetradecane, with approximate particle dimensions of 0.3×2 μm as shown by the scanning

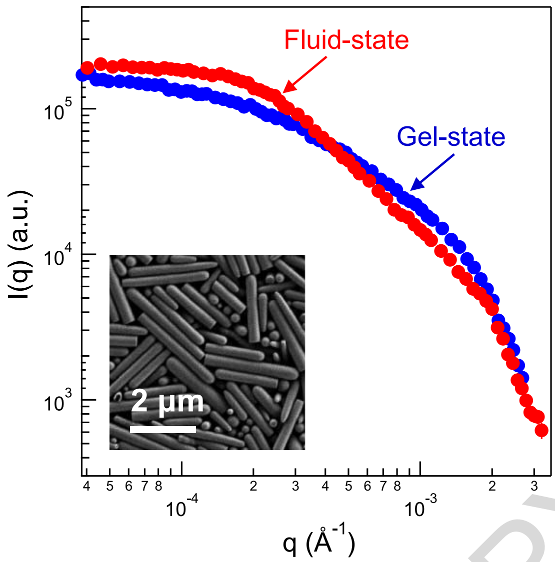

Fig. 2. DISCO–USANS data for a thermoreversible gel composed of coated silica rods in tetradecane in a fluid-state at 40°C (red) with infrared heating and in a gel-state at 27°C (blue) with no infrared heating. The tumbler cell slowly rotates the sample to limit gravitational settling and collapse of the fluid or gel microstructure. Inset SEM image shows the dried colloidal silica rods with approximate dimensions 0.3×2 μm.

electron microscopy (SEM) measurement in the inset of Fig. 2. The suspension undergoes a thermoreversible transition from a fluid-like state at higher temperatures (40°C) to a gel-like or solid-like state at lower temperatures (27°C). The DISCO–USANS measurements show distinct changes in the microstructure at different temperatures as a result of reversible, short-range, interparticle attractions in the fluid-state and gel-state.

4. Closing remarks

The controlled temperature and tumbling capabilities of DISCO–USANS is uniquely suited for samples that experience both gravitational settling and temperature sensitivity within the range of 30°C to 80°C. However, future improvements could incorporate convective air-cooling to decrease the low-temperature limit, or to improve the infra-red focusing, intensity, or thermal insulation and usage of 125 W light bulbs to increase the high-temperature limit. The current setup is fully compatible with the initial temperature control using Peltier elements for temperatures between 15°C and 40°C.

As an alternative to IR, ultraviolet light could be incorporated to study the microstructural changes of ultra violet-responsive samples, such as UV curing of paints, coatings, adhesives, or melts. If the samples show ordering like lipid membranes in solution, the rotational stage allows for tracking the directional changes induced by the UV, in the case that the neutrons are collected in "event-mode" triggered by the rotation of the cells.

Acknowledgements

The authors would like to thank the organizers of the 9th International Workshop on Sample Environment at Scattering Facilities for the opportunity to disseminate these results. This work utilized facilities supported in part by the National Science Foundation under Agreement No. DMR-1508249. The authors acknowledge financial support of this work from the University of Delaware and the National Institute of Standards and Technology under the cooperative agreements 70NANB10H256 and 70NANB12H239.

References

[1] J.G. Barker, C.J. Glinka, J.J. Moyer, M.H. Kim, A.R. Drews and M. Agamalian, Design and performance of a thermal-neutron double-crystal diffractometer for USANS at NIST, *J Appl. Cryst.* **38**(6) (2005), 1004–1011. doi:10.1107/S0021889805032103.

[2] R.P. Murphy, K. Hong and N.J. Wagner, Thermoreversible gels composed of colloidal silica rods with short-range attractions, *Langmuir* **32** (2016), 8424–8435. doi:10.1021/acs.langmuir.6b02107.

[3] A. Olsson, M.S. Hellsing and A.R. Rennie, A holder to rotate sample cells to avoid sedimentation in small-angle neutron scattering and ultra small-angle neutron scattering experiments, *Meas. Sci. Technol.* **24**(10) (2013), 105901. doi:10.1088/0957-0233/24/10/105901.