Optimization of dynamic nuclear polarization system for contrast variation in small angle neutron scattering

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The significant difference of neutron scattering length between proton and deuteron has been utilized in many aspects of neutron spectroscopy. As one of the examples, contrast variation technique in small-angle neutron scattering (SANS) is essential for studying nanometer-scale structure of materials composed of multi-components and multi-phases. On the other hand, a polarized neutron's scattering length against a polarized proton is largely dependent on polarization degree and relative direction of them. This can also be utilized for contrast variation.

Our group have applied this technique to polymer [1] and rubber [2] systems which are not easily deuterated. By applying vapor sorption of free radical TEMPO, which serves as an electron spin source necessary for dynamic nuclear polarization (DNP), we have for the first time succeeded to polarize silica-filled SBR rubber, which is widely used for fuel-efficient tire. From the observed SANS profiles against a polarized neutron beam with various proton spin polarization (P) values, the partial scattering function of silica was successfully separated, whose spatial distribution is a key factor for fuel-efficiency [2].

After the last SANS experiment, we have improved the DNP system. As for sample handling, we searched a optimum condition for radical doping and minimized a sample's contact with atmospheric oxygen, which tends to relax surrounding proton spins faster. As for the instrument, in order to enhance the power of microwave irradiation, which stimulates polarization transfer from electron to proton spins in DNP process, we introduced a high-power microwave amplifier and reduced the loss of microwave through the transmission pathway. As a result, we have polarized TEMPO doped polystyrene up to 50.8 % at 1.2 K and 3.35 Tesla. Consequently, the proton's scattering length against fully polarized neutron can be controlled from -1.114×10^{-12} cm to 0.366×10^{-12} cm. The achieved variation width is 1.42 times of the difference between proton and deuteron.

References

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