Preliminary Tests of Improved 900 MHz MAS Cold Gas Delivery System

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Introduction
An application of the 900 MHz 3.2 mm MAS NMR probe for material science is to perform extended duration experiments at sample temperatures reaching 123 ± 0.2 K [1]. Low probe temperatures are achieved by directing into the stator a N₂ gas stream which has been cooled using a liquid nitrogen (LN₂) cryostat-enclosed heat exchanger (HX). The existing cold gas delivery system was found to be incapable of reliably reaching 123 K while maintaining a stable rotor spin rate, so an undergraduate team was sponsored from the FAMU-FSU Department of Mechanical Engineering senior design capstone course, and tasked to improve the performance of this system.

Experimental
The original HX consists of two loops of coiled copper tubing submerged in a bath of LN₂. Room temperature N₂ gas flows through the tubing and cools proportionally to flow. Tests on the original HX showed flow rates of up to 3340 L/h necessary to reach temperatures of 120 K measured by the probe thermocouple, located near the stator. The MAS stator routes the cold gas exhaust through the annular gaps between the rotor and two gas bearings, with a total gap cross sectional area of 0.77 mm². It was hypothesized that turbulence of the cold gas exiting the stator through the gaps at this flow rate led to rotor spin instability. Two design modifications were devised to correct the problem. First, exhaust ports were added to the stator which increased the cross sectional area of the exiting gas stream to 8.7 mm². Second, a replacement set of HX coils with 22 loops was fabricated in place of the original two loop coil. Tests with the replacement coils reached 111 K with the rotor spinning at 5 kHz and a gas flow of 1700 L/h, approximately half the flow rate required to reach 120 K with the original coils.

The temperature-dependent chemical shift of ²⁰⁷Pb in Pb(NO₃)₂ was measured at various gas flow rates and target temperatures using the spectrometer’s variable temperature regulation system. The sample was spun at a rate of 5000 ± 20 Hz, and the recorded chemical shifts were converted to temperature using the linear fit slope from Bielecki [2] of 0.753 ppm/K, presented in Figure 1.

Conclusions
The difference in temperature between the probe thermocouple and sample rotor averaged 3 K and stable spin rates were maintained to 115 K, so initial modifications to the system appear to be successful. Future modifications in Spring of 2009 will include lowering thermal losses in the probe cryostat and adding an automatic LN₂ refill capability to the HX cryostat.

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References