LASER-HEATED NMR PROBE SYSTEM FOR IN SITU CATALYSIS

LASER-KUUMUTUSEGA IN SITU KATALÜÜSI TMR MÕÕTEPEA SÜSTEEM

MASTER THESIS

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SUMMARY

This thesis work was inspired by international demand on specialized NMR MAS probes for the study of catalytic processes as they are used in oil refineries. From a study of available probe options, it became clear that they invoke serious compromises in modelling actual reaction conditions, also designs tend to make the experiment complicated.

We introduced a fiber-optic laser-heating system, which is relatively convenient and safe to use in every laboratory. The probe was designed, constructed and partly optimized. We were also able to test it in the most critical function - heating. Even with a limited optimization, temperature performance exceeded our expectations - aimed 350 °C was achieved with only about 40% of heating power and tested top temperature was 433 °C. In addition to a novel heating system, the probe provides also rapid reduction of the temperature for reaction freezing, first of a kind ever made. Novel is also the non-contact rotor temperature measurement in high magnetic fields. Full functionality of the probe, combining RF performance, spinning, feedstock flow, reaction product collection, heating and cooling, is currently being tested.

The work here also a cornerstone for future automation developments, in which the sample temperature (laser powers) can be manipulated by a computer, via dedicated software interface. The control software should ease the selection of proper gradient distribution and temperature modulation, indicate all the probes critical thermal parameters on single screen, for better overview.