QuantERA Funded Projects Mid-term Strategic Conference
Granada, 13-14 November 2019
NanoSpin

This project has received funding from the European Union’s Horizon 2020 research and innovation programme under grant agreement No 731473.
The NanoSpin vision

- Using color centers to push the sensitivity limit of NMR spectroscopy to single molecules with spectral resolutions sufficient to resolve J-couplings
- Integrating color center hyperpolarization technology with ESR-on-a-chip, NMR-on-a-chip and lab-on-a-chip technology (HyperNMR-on-a-chip) to into low-cost portable units that open new markets for spin-based analytics such as personalized medicine
- Integrating color center hyperpolarization into commercial high-end NMR spectrometers to reduce 1’000 to 10’000-fold the measurement time in standard NMR experiments
The NanoSpin Approach

- Color center dynamic nuclear polarization (DNP)
- Today’s NMR and ESR experiments largely limited by their poor sensitivity
- current approaches require extreme experimental conditions
- excessively long polarization times due to slow electron spin thermalization
- NV centers in diamond color centers in silicon carbide (SiC) can dynamically produce very high polarization levels (>95 %) by means of laser irradiation even at room temperature and low $B_0$
- color center hyperpolarization is not limited to thermal equilibrium Boltzmann polarization levels and thermalization rates
- 1’000 to 10’000-fold enhancement factors become achievable
The NanoSpin Concept

- Using color center hyperpolarization in combination with advanced integrated circuit technology and advanced materials science concepts for compact hyperpolarization devices that can revolutionize many disciplines including NMR.

### Comparison Table

<table>
<thead>
<tr>
<th>NMR spectrometer</th>
<th>Sensitivity</th>
<th>Size</th>
<th>Cost</th>
</tr>
</thead>
<tbody>
<tr>
<td>22 T high resolution NMR spectrometer at NIMS</td>
<td>100-fold higher than 1T benchtop spectrometer</td>
<td>3-story building</td>
<td>&gt; €10 Mio.</td>
</tr>
<tr>
<td>1 T portable combined ESR/DNP-enhanced spectrometer with on-chip polarizer</td>
<td>1'000 to 10'000-fold higher than 1T benchtop spectrometer with 0.4% polarization</td>
<td>Portable (10 cm)³</td>
<td>&lt; €10k for spectrometer including on-chip diamond DNP</td>
</tr>
</tbody>
</table>

Using improved materials and electronics concepts to turn color center DNP into a widely used method.

The ESR- and NMR-on-a-chip concepts allow for miniaturized $B_1$ field sources with immense frequency agility and complete NMR transceivers on $1 \text{ mm}^2$. 
The NanoSpin Consortium

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Selected results

All WPs progressed according to plan 😊
Highly efficient and robust pulse sequences for polarization transfer have been developed.

An Atomic Scale Fridge

Converting Light to Nuclear Spin Polarisation

- Microwave
- Laser

300 K

1.2 K

$10^{-8} \text{ eV}$

$3 \times 10^{-4} \text{ eV}$

Nuclear spins

Electron spins

Photons

Flow of entropy

PulsePol

NanoSpin Results: WP2 NV center theory
NanoSpin Results: WP3 Material’s optimizing...

- New large volume production process for nanodiamonds
- Thin layers of diamond with outstanding coherence
- Nanostructured diamond surfaces with excellent wetting properties
- Optical and ODMR properties verified

Pyruvic acid wetting nanostructured surface
Diamond stacks increase the available polarization volume

Efficient polarization requires correcting for small deviations in diamond crystallographic orientation due to preparation of substrates (miscut / tilt of 100 orientation)

Custom stacking instrument developed with live ODMR monitoring yields (111) axis alignment to within +/- 1 degree

NanoSpin Results: WP3 diamond stacks (1/3)
NanoSpin Results: WP3 diamond stacks (2/3)

- Stacking system + alignment system = optimized stack
The optimized diamond stack shows both excellent alignment and high optical polarization.

Narrow ODMR line after stacking, enabling efficient polarization.

Good illumination of entire stack, resulting in very high NV optical polarization.

Stack ready for polarization experiments.
A complete NMR-on-a-chip transceiver has been manufactured and characterized

- 1 MHz – 130 MHz, max. current 1.4 A
- Input noise: $1 \text{nV}/\sqrt{\text{Hz}}$
- $^1\text{H}$ Concentration sensitivity @ 1.5 T: 1.5 mM
A complete EPR-on-a-chip transceiver has been manufactured and characterized

Very agile frequency control (> 1 GHz/μs) and full phase control (full amplitude control in next generation)

Large $B_1$ fields > 1 mT (i.e. > 28 MHz) @14 GHz
## NanoSpin Results: Completed milestones

<table>
<thead>
<tr>
<th>No.</th>
<th>Designation</th>
<th>Nature*</th>
<th>Delivery date</th>
<th>Partners involved (indicate the number and underline the lead partner)</th>
</tr>
</thead>
<tbody>
<tr>
<td>M1</td>
<td>NV centres with long coherence times</td>
<td></td>
<td>2</td>
<td>M12</td>
</tr>
<tr>
<td>M2</td>
<td>Design of a diamond-DNP probe head for commercial NMR spectrometers</td>
<td></td>
<td>2</td>
<td>M12</td>
</tr>
<tr>
<td>M3</td>
<td>First generation of monolithic ESR and NMR spectrometers available</td>
<td>Prototype</td>
<td>M18</td>
<td>M18</td>
</tr>
<tr>
<td>M4</td>
<td>Optimal hyperpolarisation sequences, experimental set-ups and signal processing available</td>
<td>Software</td>
<td>M18</td>
<td>M18</td>
</tr>
<tr>
<td>M5</td>
<td>Optimised nanodiamonds</td>
<td>Material</td>
<td>M18</td>
<td>M18</td>
</tr>
</tbody>
</table>
A milestones have been completed in time

For the second half of the project we therefore target to complete all milestones as well

<table>
<thead>
<tr>
<th>Number</th>
<th>Month</th>
<th>WP #</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>M6</td>
<td>20</td>
<td>WP5</td>
<td>DNP setup integrated in spectrometer</td>
</tr>
<tr>
<td>M7</td>
<td>24</td>
<td>WP6</td>
<td>Combined portable NMR/ESR spectrometer</td>
</tr>
<tr>
<td>M8</td>
<td>30</td>
<td>WP4</td>
<td>Monolithic NMR and ESR spectrometers with optimum pulse control</td>
</tr>
<tr>
<td>M9</td>
<td>30</td>
<td>WP2</td>
<td>Proposal for optimal diamond surface termination based on ab initio calculations</td>
</tr>
<tr>
<td>M10</td>
<td>30</td>
<td>WP5</td>
<td>Milestone experiment in commercial spectrometer with a 1’000-fold reduced measurement time compared to thermal polarisation</td>
</tr>
<tr>
<td>M11</td>
<td>30</td>
<td>WP6</td>
<td>Successful integration of hyperpolarisation into portable setup</td>
</tr>
<tr>
<td>M12</td>
<td>30</td>
<td>WP8</td>
<td>Optimized colour centres in wafer and nanocrystalline SiC</td>
</tr>
<tr>
<td>M13</td>
<td>36</td>
<td>WP7</td>
<td>Successful NV-based detection of J-couplings in nearby molecules</td>
</tr>
<tr>
<td>M14</td>
<td>36</td>
<td>WP6</td>
<td>Portable Hyper-NMR-on-a-chip system</td>
</tr>
</tbody>
</table>
Dissemination

- 19 journal and conference (4-page peer reviewed) publications
- 1 book chapter
- 1 national conference
- 4 invited talks
- 2 patent applications
- 1 popularization article
- 3 presentations at popularization conferences
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