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Citation: Review of Scientific Instruments 85, 043903 (2014); doi: 10.1063/1.4870798
View online: https://doi.org/10.1063/1.4870798
View Table of Contents: http://aip.scitation.org/toc/rsi/85/4
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Moissanite anvil cell design for giga-pascal nuclear magnetic resonance

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(Received 14 March 2014; accepted 27 March 2014; published online 14 April 2014)

A new design of a non-magnetic high-pressure anvil cell for nuclear magnetic resonance (NMR) experiments at Giga-Pascal pressures is presented, which uses a micro-coil inside the pressurized region for high-sensitivity NMR. The comparably small cell has a length of 22 mm and a diameter of 18 mm, so it can be used with most NMR magnets. The performance of the cell is demonstrated with external-force vs. internal-pressure experiments, and the cell is shown to perform well at pressures up to 23.5 GPa using 800 μm 6H-SiC large cone Boehler-type anvils. 1H, 23Na, 27Al, 69Ga, and 71Ga NMR test measurements are presented, which show a resolution of better than 4.5 ppm, and an almost maximum possible signal-to-noise ratio. © 2014 AIP Publishing LLC. [http://dx.doi.org/10.1063/1.4870798]

I. INTRODUCTION

Since the density of bond energy in solids is rather high,1–3 pressures well into the Giga-Pascal (GPa) range are needed to study changes in bonding, or the electronic properties of modern materials. Therefore, high hydrostatic pressures accessible only with diamond or moissanite anvil cells are of great interest in solid state physics.

Many experimental methods are available for high-pressure studies, but nuclear magnetic resonance (NMR) spectroscopy is inherently difficult to perform with anvil cells due to the small sample size and a low filling factor of the resonating radio frequency (RF) coil, yielding signals often below the thermal noise. Early approaches that used single-turn3,4 or split-pair coils outside the high pressure regions at the anvil flanks,5 suffered from a low signal-to-noise ratio (SNR), rendering a broader application of such cells with NMR almost impossible.

Recently, a new approach to GPa NMR was introduced,6 demonstrating the possibility of NMR in anvil cells using a multi-turn resonating RF micro-coil inside the sample cavity, which increased the coil’s filling factor by several orders of magnitude. First experiments7,8 showed how powerful NMR at such pressures can be, and that this set-up could even be used with low-gamma nuclei at pressures routinely above 8 GPa. These first applications were based on an anvil cell design of the Cavendish Laboratory at Cambridge University, a design widely used for de Haas-van Alphen measurements.9

Meanwhile, we have designed, tested, and performed experiments with various new homebuilt anvil cells. Herein, we introduce one of our new designs which we label “LAC-TM1,” a miniature anvil cell produced in Leipzig, see Fig. 1. The main component of the chassis is Titanium-64 grade 23 (titanium alloyed with 6 wt.% aluminum and 4 wt.% vanadium with an extra low interstitial level), a non-magnetic titanium alloy known for its high mechanical strength and low thermal expansion coefficient,10 as well as its low magnetic susceptibility.11

Typical materials for high pressure anvil cells are steel or inconel alloys.12,13 Despite their extraordinarily high mechanical strengths these alloys are strongly ferromagnetic, and thus cannot be employed for sensitive measurements of nuclear or even electronic magnetic moments, e.g., in SQUID or NMR experiments. An established alternative is hardened beryllium copper (Cu-Be alloy 25 from MATERION Brush), which is known for its excellent mechanical strength as well as its non-magnetic character, see Table I. However, at cryogenic temperatures this alloy may cause changes in the dimensions of the cell components by several micrometers. This decreases work stability due to possible pressure leakage, or in the worst case leading to total material failure due to the relatively high thermal expansion coefficient.

It is known that moissanite anvils provide a cost-effective alternative to those made from diamond up to pressures of about 60 GPa.14–16 The anvils we use with our LAC-TM1 are of “Boehler-type” geometry.17 Such anvils are cut with a conical table area (despite the more commonly used so-called “Drukker-type” anvils18 which are cut with a flat table area) and were manufactured by Charles and Colvard Inc. To maximize working stability, the anvils are designed with a bigger conical table area compared to the normally used Boehler-type anvils for optical or X-ray measurements where big apertures are mandatory, leading to overall anvil dimensions of 4 mm girdle diameter, 3.4 mm total height, and 1.8 mm cone height. The table area for this particular anvil geometry is about twice the area of Drukker-type anvils with the same girdle diameter, leading to twice the maximum applicable force before failure in the anvils or the titanium seats is anticipated. The anvils’ culet faces are beveled to 15° to ensure a safe feed-through of the electrical leads as well as a homogeneous pressure distribution along the culet face.19

We will demonstrate that our Boehler-type anvil design with our LAC-TM1 is capable of reaching high pressures without material failure, and we will show with 1H, 23Na, 27Al, 69Ga, and 71Ga NMR test measurements that a magnetic field homogeneity of better than 4.5 ppm and an almost maximum signal-to-noise ratio can be achieved with these cells. This makes them an excellent tool for the study of condensed matter problems with most commercial NMR spectrometers.
FIG. 1. The LAC-TM1 design: (left) photograph, at the bottom is a millimeter scale; (right) cut-away cross-sectional drawing. Due to its straightforward build, the main focus was on minimising the cell dimensions, leading to overall dimensions of 22 mm in height and 18 mm in diameter. Labelling: (a) hot wire, (b) grounding wire, (c) Ti screws, (d) piston, (e) brass guide pins, (f) Be-Cu gasket, (g) RF micro-coil, (h) 6H-SiC anvils, and (i) x-y alignment.

II. EXPERIMENTAL

The hardened beryllium copper gaskets of an initial thickness of 500 μm were pre-indentated to 100 μm and 125 μm using large-cone 6H-SiC anvils (800 μm and 1000 μm culet diameter). Finely crushed ruby powder in the sample cavity served as pressure sensors using the pressure dependence of the ruby sharp-line luminescence, which was observed via a commercially available optical spectrometer system. Liquid paraffin oil was inserted to ensure hydrostatic conditions at pressures up to 8 GPa.

In order to monitor the overall working stability of the LAC-TM1, the pressure evolution of the ruby luminescence was monitored. The applied forces were controlled using a hydraulic press equipped with a manometer. The internal pressures were calculated using the two Lorentzian peak approach of the ruby sharp line luminescence, which was observed via a commercially available optical spectrometer system. Liquid paraffin oil was inserted to ensure hydrostatic conditions at pressures up to 8 GPa.

All NMR experiments used micro-coils (solenoids) wound (4–6 turns) from 18 μm insulated copper wire (plus a 5 μm polyurethane insulation) from GoodFellow. In order to avoid short-circuiting of the fragile copper coil with the Cu-Be gasket two channels were carved by a scalpel into the gasket accommodating the coil’s leads that were glued with an epoxy resin to the metal (this is a straightforward procedure, however, it requires substantial technical skills and training).

Fig. 2 shows the complete set-up of the high pressure region of the anvil cell. The completely assembled anvil cells were mounted on home-built NMR probes (with the axis of the solenoid micro-coil perpendicular to the external magnetic field \(B_0\)) for further tuning and matching according to the various frequencies of the nuclei under investigation. The experiments were performed at magnetic field strengths of \(B_0 = 7.05\) T and \(B_0 = 11.74\) T.

III. RESULTS

A. Working stability and pressure performance

Potential material failures under load are expected for different parts of the anvil cells: (a) breakage of the moissanite anvils; (b) a bursting of the titanium screws (which should support loads of 0.5 tons each); (c) direct material ruptures of the cell chassis.

The results of two pressure runs to investigate the accessible pressure limits of our LAC-TM1 with 1 mm and a 0.8 mm culet 6H-SiC anvils are shown in Fig. 3. In the initial loading phase at lower applied external forces, the pressure evolution shows a parabolic behavior caused by plastic deformation of the beryllium copper gaskets. Above approximately 1.2 kN and 0.75 kN for 1000 μm and a 800 μm anvils, respectively, the Be-Cu gasket becomes “locked” and the 6H-SiC anvil pressure faces progressively flatten with increasing load. In this region, the pressure increases linearly under load. Due to cupping of the anvil’s pressure faces when the flattening is complete, the pressure in the sample cavity for both large-cone moissanite anvils reached plateaus at 8.5 GPa and 23.6 GPa. In both cases, a further increase in load resulted in considerable tension of the titanium screws and material failure in the cell chassis, whereas the 6H-SiC anvils, as well as their supporting plates, were found to be undamaged.

After both pressure runs, the thickness of the pre-indent Be-Cu gaskets was reduced from 125 μm and 100 μm to 50 μm and 35 μm, respectively. The inset of Fig. 3 shows that the glass transition of the applied paraffin oil occurred at pressures above 9 GPa, leading to a considerable loss in hydrostaticity.

TABLE I. Summary of properties of materials (for references see text). \(R_{\text{me}}\) is the yield strength, \(\kappa\) is the thermal conductivity, \(\alpha_V\) is the coefficient of thermal expansion, and \(\chi\) is the magnetic susceptibility; (*) denotes hardened state values. Ti64 grade 5 is Titanium-Al6wt.%-V4wt.%, Ti64 grade 23 is the extra-low interstitial version of grade 5 and Cu-Be is Copper-beryllium alloy 25.

<table>
<thead>
<tr>
<th>Material</th>
<th>(R_{\text{me}}) MPa</th>
<th>(\kappa) Wm(^{-1})K(^{-1})</th>
<th>(\alpha_V) (10^{-6}) K(^{-1})</th>
<th>(\chi) ppm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti64 grade 5</td>
<td>896</td>
<td>6.6</td>
<td>9.5</td>
<td>≈3</td>
</tr>
<tr>
<td>(1200)*</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ti64 grade 23</td>
<td>862</td>
<td>7.3</td>
<td>9.5</td>
<td>≈3</td>
</tr>
<tr>
<td>(1150)*</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cu-Be alloy 25</td>
<td>400</td>
<td>105</td>
<td>17.5</td>
<td>≈5</td>
</tr>
<tr>
<td>(1500)*</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Steel 316/316L</td>
<td>1200</td>
<td>16.2</td>
<td>9.7</td>
<td>&gt;1000</td>
</tr>
</tbody>
</table>

FIG. 2. (Left) schematic drawing (side view) of the high-pressure NMR sample cavity. (Right) photograph (top view) of a prepared gasket with a 4-turn copper micro-coil using a large-cone 6H-SiC anvil with a 800 μm culet.
FIG. 3. Pressure performance test of the LAC-TM1 using two large-diameter (1.0 and 0.8 mm) culets. The applied forces were measured using a liquidus manometer. In both cases, the pressure reached a maximum at 8.5 GPa and 23.5 GPa at external forces of 3.2 and 4.5 kN, respectively. (Inset) ruby luminescence spectra from within the sample cavity. Note the loss of hydrostaticity at about 9 GPa due to the glass transition of the pressure transmitting paraffin oil.

B. NMR performance

By now, the LAC-TM1 has been used routinely (above 9 GPa) with various NMR experiments in our laboratory. Here, we focus on the investigation of the resolution and sensitivity with test measurements performed on four different materials.

Our experiments show (see below) that the field homogeneity across the sample region is better than 4.5 ppm in our LAC-TM1. This result was achieved by placing the cells in our commercial superconducting, wide-bore magnets without employing any room-temperature shimming. Since this homogeneity is better than we demand, and certainly allows for a large number of NMR applications, we did not investigate this issue any further. Rather, we focussed on testing the sensitivity with four different materials, which is ultimately the limiting factor for applications.

A summary of parameters needed for measuring the NMR performance is given in Table II. Typical spectra are presented in Fig. 4.

As usual, we define the signal-to-noise ratio (SNR) per scan after a $\pi/2$-pulse by the ratio of the amplitude of the voltage induced across the terminals of the RF coil ($U_{\text{ind}}$), and the root-mean-square (rms) thermal noise voltage ($U_{\text{Noise}}$) from the resistance of the wire of the RF coil, i.e.,

$$SNR = \frac{U_{\text{ind}}}{U_{\text{noise}}}.$$  

For a magnetization $M_0$ that precesses with the Larmor frequency $\omega_0 = \gamma_n B_0$ ($\gamma_n$ is the gyromagnetic ratio of nucleus $n$) in a plane that contains the axis of a solenoid ($N_{\text{coil}}$, number of turns; $A_{\text{coil}}$, effective cross-section of the solenoid) we have approximately

$$SNR = \eta N_{\text{coil}} A_{\text{coil}} \omega_0 M_0 \frac{1}{\sqrt{4 R_{\text{coil}} k_B T \Delta f}}.$$  

(1)

TABLE II. Summary of the parameters for SNR measurements. Next to sample (numbered according to discussion in the text) and the measured nucleus we show: the culet diameters, the number of turns of the RF micro-coil ($N_{\text{coil}}$), the volume of the micro-coil ($V_{\text{coil}}$), the approximate geometrical filling factor ($\eta$), the number of resonant nuclei N, the external magnetic field strength ($B_0$), predicted RF amplitude $B_{1, \text{theo}}$, duration of $\pi/2$ pulse ($t_{\pi/2}$), measured RF amplitude ($B_{1, \text{exp}}$), the expected (SNR$_{\text{theo}}$) and measured (SNR$_{\text{exp}}$) signal-to-noise ratios. The quality factor (Q) was in the range of 18–25, and the RF pulse powers ranged between 450 and 550 mW, the noise bandwidth was 1 MHz. For liquid gallium and 4:1 methanol/ethanol mixture the $t_{\pi/2}$ pulse duration refers to non-selective excitations, whereas for all other cases only the central transition was excited.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Nucleus</th>
<th>Culet (mm)</th>
<th>$N_{\text{coil}}$ (nl)</th>
<th>$V_{\text{coil}}$ (nl)</th>
<th>$\eta$</th>
<th>N ($10^{16}$)</th>
<th>$B_0$ (T)</th>
<th>$B_{1, \text{theo}}$ (mT)</th>
<th>$t_{\pi/2}$ (μs)</th>
<th>$B_{1, \text{exp}}$ (mT)</th>
<th>SNR$_{\text{theo}}$</th>
<th>SNR$_{\text{exp}}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>(1) Na-A</td>
<td>$^{27}\text{Al}$</td>
<td>0.25</td>
<td>5</td>
<td>10</td>
<td>1</td>
<td>1.5</td>
<td>7.05</td>
<td>36</td>
<td>1</td>
<td>23</td>
<td>0.2</td>
<td>0.12</td>
</tr>
<tr>
<td>(2) Al metal</td>
<td>$^{27}\text{Al}$</td>
<td>0.8</td>
<td>4</td>
<td>3.1</td>
<td>≈0.75</td>
<td>14</td>
<td>11.74</td>
<td>43</td>
<td>0.75</td>
<td>30</td>
<td>12</td>
<td>8.2</td>
</tr>
<tr>
<td>(3) meth./eth.</td>
<td>$^1\text{H}$</td>
<td>1</td>
<td>5</td>
<td>6.4</td>
<td>1</td>
<td>2.9</td>
<td>7.05</td>
<td>23</td>
<td>3</td>
<td>19.6</td>
<td>50.3</td>
<td>25.1</td>
</tr>
<tr>
<td>(4) Ga metal</td>
<td>$^{69}\text{Ga}$</td>
<td>0.8</td>
<td>4</td>
<td>3.1</td>
<td>0.5</td>
<td>2.8</td>
<td>11.74</td>
<td>57</td>
<td>0.75</td>
<td>32</td>
<td>1.0</td>
<td>0.73</td>
</tr>
<tr>
<td></td>
<td>$^{71}\text{Ga}$</td>
<td>0.8</td>
<td>4</td>
<td>3.1</td>
<td>0.5</td>
<td>1.8</td>
<td>11.74</td>
<td>53</td>
<td>0.5</td>
<td>38</td>
<td>1.2</td>
<td>0.56</td>
</tr>
</tbody>
</table>
The macroscopic magnetisation $M_0$ is given by Curie’s high-temperature formula,

$$M_0 = \frac{N \gamma_n^2 h^2 I_n (I_n + 1) B_0}{3 \kappa B T},$$

(2)

where $N$ denotes the number of resonant nuclei in the volume $V$, and $I_n$ is the spin of nucleus $n$.

(1) Na-A zeolite at ambient pressure: This type of zeolite has a mass density of about 1250 kg m$^{-3}$ and a unit cell volume of 1693 $\times$ 10$^{-30}$ m$^3$. We estimate that about 4.5 $\mu$g of zeolite were contained in our RF micro-coil, i.e., about 1.5 $\times$ 10$^{16}$ resonant nuclei for $^{27}$Al as well as $^{23}$Na NMR. With nutation experiments (the duration $\tau_{1/2}$ of the $\pi/2$ pulse was about 1.0 $\mu$s), the amplitude of the RF magnetic field in the micro-coil was estimated to be $B_1 = \pi/(2 \gamma_s \tau_{1/2}) \approx 23$ mT for both nuclei. Based on the maximal stored energy (only inside the coil) and the average power loss given by the measured quality factor (Q = 15) we estimate $B_1 = \sqrt{\mu_0 Q P/2 \omega V_{\text{coil}}} \approx 35$ mT (the average pulse power was $P = 480$ milliwatts (mW)). The good agreement with the nutation suggests that most of the RF power is indeed delivered to the RF coil. With these parameters we determine from Eqs. (1) and (2) with a noise bandwidth of 1 MHz the signal-to-noise ratios: $SNR_{\text{theo}}(\text{Na}) \approx 0.11$ and $SNR_{\text{theo}}(\text{Al}) \approx 0.20$. Experimentally, we find $SNR_{\text{exp}}(\text{Na}) \approx 0.08$ and $SNR_{\text{exp}}(\text{Al}) \approx 0.12$. Noise figure measurements with a calibrated noise source showed that the spectrometer contributed about 20% of the noise. The linewidths were 13 and 9 ppm, for the Al and Na resonances, respectively. We conclude that the cell performs reasonably well to the specs.

(2) Aluminum metal powder at 3 GPa: Here, the RF solenoid had a length of 100 $\mu$m and a diameter of 200 $\mu$m, resulting in a coil volume of about 3 nl. We estimated that about 75% of that volume was filled with aluminum powder (purity 5N, 325 mesh). We found the quality factor as well as the RF field amplitude to be almost unchanged upon loading the micro-coil with the aluminum powder, ruby chips, and pressure medium. We conclude that the RF penetration is rather high, and that most nuclei contribute to the NMR signal. We expect a $SNR_{\text{theo}} \approx 12$ at a bandwidth of 1 MHz, experimentally we found $SNR_{\text{exp}} \approx 8$. The observed linewidth of 103 ppm corresponds to previous measurements of metallic aluminum powder under pressure.

(3) 4:1 methanol/ethanol mixture at 3 GPa: This very volatile mixture has an average density of 790 kg m$^{-3}$. About 2 $\mu$g were added into the 200 $\mu$m diameter micro-coil. The measured $SNR_{\text{exp}} = 25.1$ is about half the theoretical value. The linewidth was about 1.4 kHz at 7.05 T (4.6 ppm).

(4) Liquid metallic gallium: Gallium has a mass density of 6090 kg m$^{-3}$. The filling factor of the 4-turn micro-coil was about 50%, therefore we estimate that 10 $\mu$g of the pure liquid gallium metal were present in the sample volume. The two observable isotopes ($^{69}$Ga and $^{71}$Ga) have a nuclear spin of 3/2, natural abundances of 60% and 40%, and grymagnetic ratios of 6.44 and 8.18 $\times$ 10$^{-2}$ rad T$^{-1}$ s$^{-1}$, respectively. The $B_1$ field of the micro-coil was estimated to be 57 mT for $^{69}$Ga, at an average pulse power of 500 mW and a quality factor of 24, compared to a measured $B_1$ of 32 mT. The experimentally found $SNR_{\text{exp}}(^{69}$Ga) $\approx 0.73$ and $SNR_{\text{exp}}(^{71}$Ga) $\approx 0.56$ are in agreement with the estimates: $SNR_{\text{theo}}(^{69}$Ga) $\approx 1.0$, $SNR_{\text{theo}}(^{71}$Ga) $\approx 1.2$. The reasonably good agreement of the expected and measured RF field amplitudes suggests that RF penetration effects can be neglected (Ga metal has a much smaller conductance compared to Al metal). The recorded NMR spectra are shown in Figure 4. As can be seen in the figure, the Lorenztian linewidths are increasing from 4.6 ppm (at ambient pressure) up to 13 ppm (at 1.8 GPa). Above the pressure induced liquid gallium – gallium II phase transition at 2 GPa, the observed linewidths increase up to 80 ppm ($^{71}$Ga) and 66 ppm ($^{69}$Ga) at 3.9 GPa. A detailed analysis of these data will be given elsewhere.

IV. CONCLUSIONS

We introduced a new anvil cell design (LAC-TM1) for high pressure NMR experiments. The small cell size makes it possible to use it with regular, but also small bore NMR magnets (e.g., cold-bore magnets). The use of especially designed large-cone Boehler anvils, as well as the non-magnetic titanium grade 23, makes it an effective as well as cost efficient alternative to the formerly used Be-Cu anvil cells. It was demonstrated that the cell is capable of reaching pressures up to 23.5 GPa for large-culet anvils of 800 $\mu$m without anvil failure. NMR test measurements revealed a magnetic inhomogeneity below 4.5 ppm, which is sufficient for many applications, and by about a factor of 2 better than our previously used Be-Cu cells. In addition, a rough analysis of the signal-to-noise ratio with various systems showed a spin sensitivity near the expected values (similar to our previously used Be-Cu cells). For comparison, Lee reports a $SNR$ of 1 for $^1$H in water at a magnetic field of 8 T, while our experiments showed a $^1$H $SNR$ that is about 25 times higher at a 7 times smaller volume for number of resonant nuclei that is an order of magnitude less at a magnetic field of only 7 T, and thus our $SNR$ is approximately two orders of magnitude higher.

ACKNOWLEDGMENTS

This research was funded by the International Research Training Group (IRTG) “Diffusion in Porous Materials.” We acknowledge the help of Gert Klotzsche and Dr. Damian Rybicki. We also thank Marcus Anton, Steven Reichardt, Dr. Thomas Meissner, and Michael Jurkutat for stimulating discussions.

1R. Hemley, High Pressure Res. 30, 581 (2010).
The coil’s resistance can be estimated using Ohm’s law \( R = \frac{\rho}{A_{cs}} \), where the total length of the coil wire is given (considering that the coil’s windings are closely wound, by \( l_{coil} = \pi d_{cs} N_{coil} + 2l_{lead} \) (\( d_{cs} \) is the wire diameter, \( l_{lead} \) is the length of the coils leads), and the effective cross-section of the wire can be approximated to be \( A_{cs} = \pi d_{cs} \delta \). The RF skin depth \( \delta \) for a certain material with a conductivity \( \sigma \) at a frequency \( f \) is given by \( \frac{1}{\sqrt{\pi \sigma \mu_0 \mu_{rel} f}} \) (\( \mu_{rel} \) is the relative permeability for copper). For a coil with 5 turns and two leads of 3 mm length, made of 18 \( \mu \)m copper wire at 120 MHz, the ac resistance could be expected to be around 1.1 \( \Omega \). This can also be measured using the quality factor \( Q = \frac{\omega L}{R} \) of the resonance circuit, taking into account the calculation of the inductance of the coil, with \( L = N_{coil}^2 r^2/(23r + 22l) \), with the coil’s radius \( r \) and length \( l \) in cm giving \( L \) in \( \mu \)H. Using an average quality factor of 15 and a total inductance of approximately 20 nH (stemming from the coil and both its leads), the resistance would be 1 \( \Omega \), in good accordance with the calculated values.

The relative number of resonant nuclei per unit volume is given as \( N_{V} = N_A \rho Z_u \), with the natural abundance \( N_A \) of the NMR-active nucleus under investigation, the mass density of the sample \( \rho \), the relative atomic mass \( Z_u \), and the atomic mass unit \( u \).