Muons under extreme conductions: Pressure

Rustem Khasanov, PSI, Switzerland
International Advanced School on Muon Spectroscopy, 15-22 August 2019
• Principles of μSR under pressure experiments:
  – beam-lines
  – Spectrometers
  – Pressure cells
• “Decay” beam-lines
  • μE1 decay beam-line at PSI
  • General Purpose Decay (GPD) spectrometer
• Pressure cell construction(s)
• Pressure measurements

• Scientific examples
  – Interplay between superconductivity and magnetism in CrAs
μSR under pressure: Basic principles
Principle of μSR under pressure experiments

Muon momentum tuning

Beam-width tuning
The schematic view of the pressure cell (black contour) with the sample (red rectangular). Muons are implanted along the vector \( \vec{P}_\mu \). (b) The cross sectional view (X-Z plane) of the double-wall pressure cell. The colored areas represent the muon stopping distributions in parallel (red) and perpendicular (green) direction to the muon beam. The energy of implanted muons is 44 MeV. The simulations were made by using TRIM.SP package. The simulations reveal that approximately 37% of all the muons stop within the sample, 43% within the inner and 10% within the outer cylinder.
μSR under pressure:
muon momentum and beam-lines
Muon Implantation Depth

In order to perform muon experiments under pressure one needs to use the so-called ‘decay’ beam-lines.
Decay muon beam-line

π decay section

\[ p_\pi \text{ selector (e.g., 150 MeV/c)} \]

\[ p_\mu \text{ analyzer} \]

"Forward" μ ~160 MeV/c contam. with e^+ and π

"Backward" μ ~70 MeV/c clean, range ~4 g/cm²
Momentum acceptance (FWHM) 3%
Pion momentum range [MeV/c] 200--125
Muon momentum range [MeV/c] 125–60
Rate of positive muon [mA⁻¹s⁻¹] 6e7--3e7
Spot size (FWHM) 39 X 28 mm
μEl Beam-spot

Direct beam. Beam setting p107apr09. Muon momentum 109 MeV/c

Collimated beam. Beam setting p107apr09. Muon momentum 109 MeV/c
μSR under pressure:
GPD spectrometer
Construction of the detector block

Vertical setup

Horizontal setup
GPD instrument with the detectors
Sample cryostats available at the GPD instrument:

<table>
<thead>
<tr>
<th>Type</th>
<th>Manufacturer</th>
<th>Temperature Range</th>
<th>Available</th>
</tr>
</thead>
<tbody>
<tr>
<td>3He Sorption pumped</td>
<td>Oxford</td>
<td>0.24–325 K</td>
<td>Yes</td>
</tr>
<tr>
<td>4He gas flow</td>
<td>Janis</td>
<td>2.5–300 K</td>
<td>Yes</td>
</tr>
<tr>
<td>Closed Cycle Refr.</td>
<td>Home made</td>
<td>10–300 K</td>
<td>No</td>
</tr>
<tr>
<td>N₂ gas flow</td>
<td>Home made</td>
<td>80–500 K</td>
<td>No</td>
</tr>
</tbody>
</table>
Type of the pressure cell selection
μSR experiments under pressure – cell type selection

S. Klotz, Techniques in High Pressure Neutron Scattering
Contradicting criteria

- The pressure cell need to be small enough to fit inside the detector block
- The pressure cell need to carry at least few hundred mm$^3$ sample
- The pressure cell need to carry the highest possible pressure

Only the piston-cylinder type of cell could satisfy them
The highest pressure is limited by ~2.5 GPa
psi pressure cells

- 2001. Clamp cell. $p_{\text{max}} = 0.9-1.4$ Gpa / Liquid [Andreica, PhD thesis, ETHZ, 2001]
- 2009. Double-wall clamp cells. $p_{\text{max}} = 2.8$ Gpa / Liquid [Khasanov et al., High Pr. Res. 36, 140, 2016]

isis pressure cells

- 2009. Clamp cell. $p_{\text{max}} = 0.6$ Gpa / Helium [Watanabe et al., Physica B, 404, 993, 2009]

Triumf

- 2008. Clamp cell. $p_{\text{max}} = 2.3$ Gpa / Liquid [Goko, private communication]
Pressure cell: design selection
FIGURE 1.2
Stress distribution in a monobloc cylinder under two different conditions. Left: Internal pressure $p = p_i$ and external pressure $p = 0$. Right: Internal pressure $p = p_i$ and external pressure $p = p_e$.

FIGURE 1.3
Stress distribution in a fretted (compound) cylinder. The left half shows the situation in the unloaded state ($p_i = p_e = 0$), the right half under internal load.

S. Klotz, Techniques in High Pressure Neutron Scattering
Three wall double wall and single wall cells

![Diagram showing single, double, and three wall cells with dimensions labeled.]  

$$p_{\text{max}} \propto \frac{1}{2} - \frac{a^2}{2b^2}$$  

$$p_{\text{max}} \propto 1 - \frac{a^2}{2c^2} - \frac{c^2}{2b^2}$$  

$$p_{\text{max}} \propto \frac{3}{2} - \frac{a^2}{2c_1^2} - \frac{c_1^2}{2c_2^2} - \frac{c_2^2}{2b^2}$$

For $a=6\text{ mm}$ and $b=24\text{ mm}$, $\frac{p_{\text{max}}^s}{p_{\text{max}}^d/p_{\text{max}}^t} = 1 / 2 / 2.4$
Pressure cell: material selection
• Strong enough to hold the pressure
• Should not have “strong” μSR response
• Should have temperature independent response
### Nonmagnetic Alloys

<table>
<thead>
<tr>
<th></th>
<th>CuBe</th>
<th>TiAl$_6$V$_4$</th>
<th>NiCrAl</th>
<th>MP35N</th>
</tr>
</thead>
<tbody>
<tr>
<td>Yield strength</td>
<td>1.1 GPa (300 K)</td>
<td>1.05 Gpa (300 K)</td>
<td>2.06 Gpa (300 K)</td>
<td>2.15 Gpa (300 K)</td>
</tr>
<tr>
<td>Young modulus</td>
<td>131 GPa (300 K)</td>
<td>97 Gpa (300 K)</td>
<td>190 Gpa (300 K)</td>
<td>215 Gpa (300 K)</td>
</tr>
</tbody>
</table>

### Sintered materials

<table>
<thead>
<tr>
<th></th>
<th>WC</th>
<th>cBN</th>
<th>SiC</th>
<th>ZrO$_2$-Y$_2$O$_3$</th>
<th>Al$_2$O$_3$-ZrO$_2$</th>
<th>Si$_3$N$_4$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Compressive strength</td>
<td>5.0-11.0 Gpa</td>
<td>2.9 GPa</td>
<td>7.6-8.3 GPa</td>
<td>2.20 GPa</td>
<td>4.7 GPa</td>
<td>5.1-5.5 GPa</td>
</tr>
<tr>
<td>Young modulus</td>
<td>600-670 GPa</td>
<td>918 GPa</td>
<td>210 Gpa</td>
<td>357 Gpa</td>
<td>241 GPa</td>
<td></td>
</tr>
</tbody>
</table>
Pressure cell: design and construction
Material: MP35N (Ni 35%, Co 35%, Cr20%, Mo 10%)
Sample area: ø 6mm, height 12mm.
Muon stopping fraction: ~50-55%
Modified Bridgman sealing

Finite-Element analysis [CuBe/MP35N cell]
Pressure determination
Pressure determination
Strain devices for $\mu$SR
Strain cell

Artem Nikitin
Matthias Elender

Clifford Hicks

Hans-Henning Klauss
Rajib Sarkar
Vadim Grinenko
Shreenanda Ghosh
Working principle: compressive strain

- The cell is preloaded with force on the spring of 1000 N.
- Post 1 is epoxied to the cell frame, but not to the pass-thru plate; pass-thru plate can slide over post 1.
- Apply -500 N on the piezo 1.
  - The piezo 1 shrinks and moves the bridge downwards.
  - The spring expands.

- Apply +500 N on the piezo 2.
  - The piezo 2 expands.
  - The piezo moves the post downwards.
  - The post 2 is epoxied to the pass-thru plate.
  - Pass-thru plate moves downwards with force of +500 N.
  - +500 N applies to the sample.
Lowest $T = 0.7$ K in $^3$He cryostat
Sr₂RuO₄

ZF relaxation rate (left)
AC susceptibility (right)
Future developments
Three wall double wall and single wall cells

For $a=6 \text{ mm}$ and $b=24 \text{ mm}$, $\frac{p_{\text{max}}^s}{p_{\text{max}}^d} / p_{\text{max}}^t = 1 / 2 / 2.4$
McWhan pressure cell

Components:
(1) bicone,
(2) compression pad,
(3) piston,
(4) outside locking pad,
(5) inside locking pad,
(6) Body

Materials:
- (1), (3): tungsten carbide
- (2), (5), (6): MP35N
- (4): copper beryllium
McWhan pressure cell
Anvil-type pressure cell
Low-temperature press

- Enerpac 5 ton cylinder
- Connector between the cylinder and TAV6 rod
- MP35N spacer/center pieces
- YSZ pushing rods
- TAV6 outer rod
- Bellow
- Space for load cell
- Uniaxial device
- Space for load cell
Scientific example: CrAs
CrAs crystal structure

TABLE I. Refined structural and magnetic parameters of CrAs at $T = 1.5, 80,$ and $300$ K at ambient pressure; $a-c$: lattice parameters; $x, z$: atomic coordinates for site 4c in Pnma; $B$: temperature factor; $\mu$: ordered magnetic moment; $\phi$: magnetic phase angle; $k$: component of magnetic propagation vector.

<table>
<thead>
<tr>
<th></th>
<th>1.5 K</th>
<th>80 K</th>
<th>300 K</th>
</tr>
</thead>
<tbody>
<tr>
<td>$a$ (Å)</td>
<td>5.6040(3)</td>
<td>5.6068(3)</td>
<td>5.6472(3)</td>
</tr>
<tr>
<td>$b$ (Å)</td>
<td>3.5852(2)</td>
<td>3.5846(2)</td>
<td>3.4727(2)</td>
</tr>
<tr>
<td>$c$ (Å)</td>
<td>6.1301(5)</td>
<td>6.1304(6)</td>
<td>6.2017(6)</td>
</tr>
<tr>
<td>$x$/Cr (Å)</td>
<td>0.0068(12)</td>
<td>0.0064(12)</td>
<td>0.0060(10)</td>
</tr>
<tr>
<td>$z$/Cr (Å)</td>
<td>0.2034(10)</td>
<td>0.2026(8)</td>
<td>0.2022(10)</td>
</tr>
<tr>
<td>$B$/Cr (Å²)</td>
<td>0.20(8)</td>
<td>0.32(7)</td>
<td>0.62(8)</td>
</tr>
<tr>
<td>$x$/As (Å)</td>
<td>0.2011(10)</td>
<td>0.2033(14)</td>
<td>0.2021(13)</td>
</tr>
<tr>
<td>$z$/As (Å)</td>
<td>0.5802(12)</td>
<td>0.5792(12)</td>
<td>0.5758(10)</td>
</tr>
<tr>
<td>$B$/As (Å²)</td>
<td>0.12(5)</td>
<td>0.26(7)</td>
<td>0.51(7)</td>
</tr>
<tr>
<td>$\mu$ ($\mu_B$)</td>
<td>1.73(2)</td>
<td>1.71(2)</td>
<td>1.71(2)</td>
</tr>
<tr>
<td>$\phi$ (°)</td>
<td>$-110(4)$</td>
<td>$-108(4)$</td>
<td>$-108(4)$</td>
</tr>
<tr>
<td>$k$</td>
<td>0.3562(2)</td>
<td>0.3590(2)</td>
<td>0.3590(2)</td>
</tr>
<tr>
<td>$R_p$</td>
<td>5.92</td>
<td>5.83</td>
<td>5.89</td>
</tr>
</tbody>
</table>

Keller et al., PRB 91, 020409(R) (2015)
Incommensurate helical magnetic structure. The evolution of the moments for three unit cells along c; the four spirals are marked in individual colors. The propagation vector $k_c=0.3562(2)$ $\phi$ is defined as the angle between the moments of Cr atoms 1 and 2 (or 3 and 4). Ordering temperature $T_N=265$K.

Keller et al., PRB 91, 020409(R) (2015)
Pressure induced superconductivity in CrAs

Wu et al., Nat. Comm. 5, 5508 (2014)

Kotegawa et al., PRL 114, 117007 (2015)
Proposed phase diagrams

Wu et al., Nat. Comm. 5, 5508 (2014)

Kotegawa et al., PRL 114, 117007 (2015)
1. How magnetism is suppressed?

2. How occurs the superconductivity?

3. Is there any coexistence/interplay between these two phenomena?
Helical magnetic order

\[ P(B) = \frac{2}{\pi} \frac{B}{\sqrt{(B^2 - B_{\text{min}}^2)(B_{\text{max}}^2 - B^2)}} \]
Helical magnetic order

Confirmation of helical type of magnetic order in CrAs
$B_{\text{min}}$ and $B_{\text{max}}$ as a function of $p$

The internal field on the muon stopping site is proportional to the ordered moment. Increase of pressure from 1bar to 7Kbar leads to decrease of Cr moments from $1.73 \mu_B$ to $1.47 \mu_B$. 
Weak transverse field (WTF)

- Local field at the muon stopping position is the vector sum of the “internal” \( B_{\text{int}} \) and the “applied” \( B_{\text{app}} \) one
  \[ B_{\mu} = B_{\text{int}} + B_{\text{app}} \]

- In a case \( B_{\text{int}} \gg B_{\text{app}} \) (weak field regime)
  - \( B_{\mu} = B_{\text{int}} \) in the magnetically ordered parts
  - \( B_{\mu} = B_{\text{app}} \) in paramagnetic parts
WTF experiments

WTF 3mT

CrAs, $p = 1$ bar, $\mu_0 H = 3$ mT

Muon spin polarization $t (\mu s)$

$B_{\text{min}}$, $B_{\text{max}}$

ZF

CrAs, $p = 1$ Bar, $T = 5$ K

Muons spin polarization $t (\mu s)$
WTF experiments

- The magnetic transition is first order like
- Transition temperature decreases with increasing pressure
- Magnetic volume fraction decreases with pressure increase
Summary of WTF experiments

No magnetism above $p \sim 7$ Kbar!
Magnetic response of CrAs

$T_N$  

Nonmagnetic fraction  

Ordered moment

Wu et al., Nat. Comm. 5, 5508 (2014)
Kotegawa et al., PRL 114, 117007 (2015)
Shen et al., arxiv:1409.6615
Comparison with neutron data

**FIG. 4.** (Color online) Pressure dependence of the first magnetic satellite (00 ± k_z) for the pressures P = 0.35, 0.5, and 0.65 GPa. A convolution of Lorentzian and Gaussian peak shape functions was used for the profile fits.

Keller et al., PRB 91, 020409(R) (2015)
Comparison with neutron data

\[ B_{\text{int}}(\mu_0 4\pi B_{\text{int}} \mu_0 3650 \text{ (L. Keller et al.)}) \]

\[ p \text{ (KBar)} \]
Comparison with neutron data

\[ B_{\text{int}} \times (1 - \eta)^{0.5} \]

\[ \mu \times 3650 \ (\text{L. Keller et al.}) \]
Since the muon is a local probe, the $\mu$SR relaxation function is given by the weighted sum of all oscillations:

$$G(t) = \int f(B_\mu) \cos(\gamma_\mu B_\mu t) dB_\mu$$
$G(t) = \exp\left(-\frac{1}{2} \sigma^2 t^2\right) \times \cos(\gamma_{\mu} \langle B^z_{\mu} \rangle t)$

where: $\sigma^2 = \gamma_{\mu}^2 \langle \Delta B^z_{\mu} \rangle$

**Ginzburg-Landau model**

$\langle \Delta B^2_z \rangle = 0.00371 \frac{\phi_0^2}{\lambda^4}$

**London model**

$\lambda = \sqrt{\frac{m}{\mu_0 e^2 n_s}}$

$\Rightarrow \sigma \propto \frac{1}{\lambda^2} \propto \frac{\mu_0 e^2}{m} n_s$

A µSR measurement of the second moment of the field distribution allows to determine the London penetration depth $\lambda$.

The damping of a TF-µSR spectrum is proportional to the super fluid density $n_s$ (number of Cooper pairs).

*After H. Luetkens (Sunday 16 Aug 2015)*
Muon spin polarization

CrAs ($p=5.8$ kbar, 30mT TF)

Appr. 20% of the sample remains in the magnetic state
Superfluid density

Diamagnetic shift

\[ \lambda^2 \text{(\(\mu\text{m}^{-2}\))} \]

\[ B \text{ (Oe)} \]

\[ T \text{ (K)} \]

\[ 4.06 \text{ kbar} \]
\[ 4.9 \text{ kbar} \]
\[ 5.8 \text{ kbar} \]
\[ 6.7 \text{ kbar} \]
\[ 8.6 \text{ kbar} \]
\[ 10.3 \text{ kbar} \]
Phase coexistence in CrAs

- CrAs is purely magnetic up to $p \sim 3.5$ kbar.
- For $3.5 < p < 7$ kbar, magnetic and superconducting responses are detected in a set of ZF, wTF, and TF experiments. CrAs is phase separated into volumes where long range magnetic order sets at $T_N$ and into non-magnetic volumes becoming superconducting below $T_c$.
- Above 7 kbar and above $T_c$, the sample is purely in the paramagnetic state. Bulk superconductivity sets below $T_c$. 

![Graphs (a), (b), and (c)]

- Graph (a): Non-magnetic fraction $f$ vs. $p$ (KBar), data from TF300 and TF50.
- Graph (b): $T_c$ (K) vs. $p$ (KBar).
- Graph (c): $\sigma_{sc}$ ($\mu$s$^{-1}$) vs. $p$ (KBar).
• Besides the competition for the volume, there is no evidence for a competition between the magnetic and superconducting order parameter in CrAs:
  1. The ordered magnetic moment stays almost constant, by changing less than 15%.
  2. $T_N$ evolves smoothly with pressure without showing any pronounced features at $p \approx 3.5-4\text{kbar}$

-- The maximum value of $\rho_s \sim \lambda^{-2} n_s/m^*$ is observed at the low pressure side of the phase separated region - - in the region where the non-magnetic volume fraction $f$ is the smallest.

-- By neglecting the pressure effect on $m^*$, $\rho_s \sim n_s$

carriers from the 'less conductive' magnetically ordered parts of the sample can be supplied to the 'more conductive' non-magnetic parts -- self doping effect!
• The relative phase difference ($\theta$) of the superconducting order parameter between different parts of Fermi surface or Fermi surface sheets may lead either to stabilization of microscopic coexistence of the magnetic and superconducting phases or drive both to repel each others:

$$F_j \propto M^2/|\Delta_1|/|\Delta_2|\cos\theta$$

- For conventional superconductivity $\Theta=0$, $F_j$ increases making two phases unlikely to coexist.
- For $\Theta=\pi$, $F_j$ is negative. Both the superconducting and the magnetic phases tend to coexist.

Fernandes et al., PRB 82, 014521 (2010)
Correlation between $T_c$ and $\lambda^{-2}$

**Cuprates**

$\lambda^{-2} \propto T_c$

Uemura et al. PRL 66, 2665 (1991)

**Molecular s.c.**

$\lambda^{-2} \propto T_{c}^{2/3}$

Pratt and Blundell PRL 94, 097006 (2005)

**Fe-based**

$\lambda^{-2} \propto T_c$


**BCS s.c.**

$\lambda^{-2} \propto T_{c}^{3.1}$

Khasanov et al. PRB 77, 064506 (2008)
Correlation between $T_c$ and $\lambda^{-2}$

- CrAs, present study
- $T^3_{c, \text{CrAs}}$
- BCS $T_{c, \text{BCS sc}}$
- Cuprate and Fe-based $T_{c, \text{Cuprate and Fe-based sc}}$
- Molecular $T_{c, \text{Molecular sc}}$
Conclusions

• The bulk magnetism exists up to $p \sim 3.5$ kbar, while the purely non-magnetic state develops for pressures above $\sim 7$ kbar.

• In the intermediate pressure region ($3.5 < p < 7$ kbar) the magnetic phase volume decreases continuously and superconductivity develops in parts of the sample remaining non-magnetic down to the lowest temperatures.

• Both, the superconducting transition temperature $T_c$ and the zero-temperature superfluid density $\rho_s(0)$ decrease with increasing pressure in the intermediate pressure region and saturate for $p$ exceeding 7 kbar i.e. in the region where magnetism is completely suppressed.

• The pressure-induced transition of CrAs from a magnetic to a superconducting state is characterized by a separation in macroscopic size magnetic and superconducting volumes. The less conductive magnetic phase provides additional carriers (doping) to the superconducting parts of CrAs.

• The superfluid density was found to scale with $T_c$ as $T_c^{-3.2}$, which, together with the clear phase separation between magnetism and superconductivity, points towards a conventional mechanism of the Cooper-pairing in CrAs.
Thanks

• M. Elender, A. Maisuradze, Z. Guguchai, G. Simutis, Z. Shermadini, T. Goko, F. Knechet, H. Luetkens, A. Amato, E. Morenzoni
  • Laboratory for Muon Spin Spectroscopy, Paul Scherrer Institute

• D. Andreica
  Faculty of Physics, Babes-Bolyai University, Cluj-Napoca, Romania

• S. Klotz
  IMPMC, CNRS UMR 7590, Sorbonne Université, Paris, France

• K Kamenev
  Centre for Science at Extreme Conditions and School of Engineering, The University of Edinburgh, Scotland, UK