

An ultra-compact in-situ ^3He polarizer for high Q-range SANS: on-beam test and future upgrades

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Abstract. We present an ultra-compact ^3He polarizer to be used as a polarization analyzer for separation of incoherent background for soft matter research. The ^3He will be polarized in-situ within a very compact, 18 cm long, magnetic cavity that can be placed between the sample and the detector tank of small-angle scattering diffractometer KWS2 at MLZ [1]. This allows for the angular coverage of 38° , that corresponds to the maximal momentum transfer $Q_{\text{max}}=0.85 \text{ \AA}^{-1}$ for neutrons with wavelength $\lambda=4.5 \text{ \AA}$. The Q_{max} can be extended to 1.28 \AA^{-1} following the recent upgrade of KWS2 for neutron wavelength $\lambda=3 \text{ \AA}$. The optical pumping will be done by the diode laser array bar frequency narrowed by the ultra-compact volume Bragg grating. The full system will be readily hand transportable and thus useful as an instrument add-on.

1. Introduction.

Incoherent background can create an intrinsic problem for standard small angle neutron scattering measurements. Biological samples contain hydrogen which is a strong incoherent scatterer thus creating an intrinsic source of background that makes determination of the coherent scattering parameters difficult in special situations. This can especially be true for the Q-range from around $0.1\text{--}0.5 \text{ \AA}^{-1}$ where improper knowledge of the background level can lead to ambiguity in determination of the samples structure parameters. Polarization analysis is a way of removing this ambiguity by allowing one to distinguish the coherent from incoherent scattering, even when the coherent scattering is only a small fraction of the total scattered intensity [2]. ^3He spin filters are ideal for accomplishing this task because they permit the analysis of large area and large divergence scattered neutron beams.



This work describes a design for a new ultra compact SEOP [3-4] polarized ^3He neutron spin filter that can be used for this application. The magnetic system has been completed and tested. The mechanical components for online polarization, such as a compact laser system, the oven and the results of in-situ test will be described in this document.

Our system is based on a 17 cm long mu-metal solenoid. Fully capped mu-metal solenoids can be used to create very uniform magnetic fields. However, for neutron applications there must be holes in the caps to allow access for incident and scattered neutron beams. These holes, especially the larger one required for the scattered beam, undoubtedly create field gradients that significantly reduce the lifetime of the polarized ^3He in the cavity. To decrease the effect of these gradients we have used an approach that combines partial shielding and the compensation of the holes in the end caps with additional coils (figure1).

A $1 \times 1 \text{ cm}^2$ beam is assumed to be incident on the sample, which would be a typical soft matter sample located in the brown holder directly before the right end cap. The cell is placed within 2cm of the $1.5 \times 1.5 \text{ cm}^2$ hole in this end cap. After passing through the continuously polarized ^3He cell of 6cm diameter and 5cm length a square cross section beam can pass the partially shielded opening in the end cap on the left of the drawing.

Expansion of the laser beam to $D=6 \text{ cm}$ will be done with optic inside the magnetic cavity and the expanded beam will be reflected longitudinally along the neutron beam via a gold coated Si-wafer as shown in Figure 1. The collimation optics will include a telescope utilizing a large diameter 45 degree parabolic metal mirror as the last element which will be located inside the magnetic cavity.

The heating system use static air heated by electric cartridge heaters mounted in aluminum heat sinks. These stainless steel electric cartridge heaters (220 V CSS-series from Omega Engineering) now used for heating have not been found to decrease the expected ^3He polarization, or room temperature T_1 lifetime. There is indeed some magnetic components to these heaters, namely the nickel plated lead wires, however in our installation, the heater is placed inside the before mentioned aluminum heat-sinks, about 10 cm away from the cell, with the wires exiting away from the cell position. The aluminum heat sink is grounded to limit pickup of electrical noise by the NMR FID system [5-7].

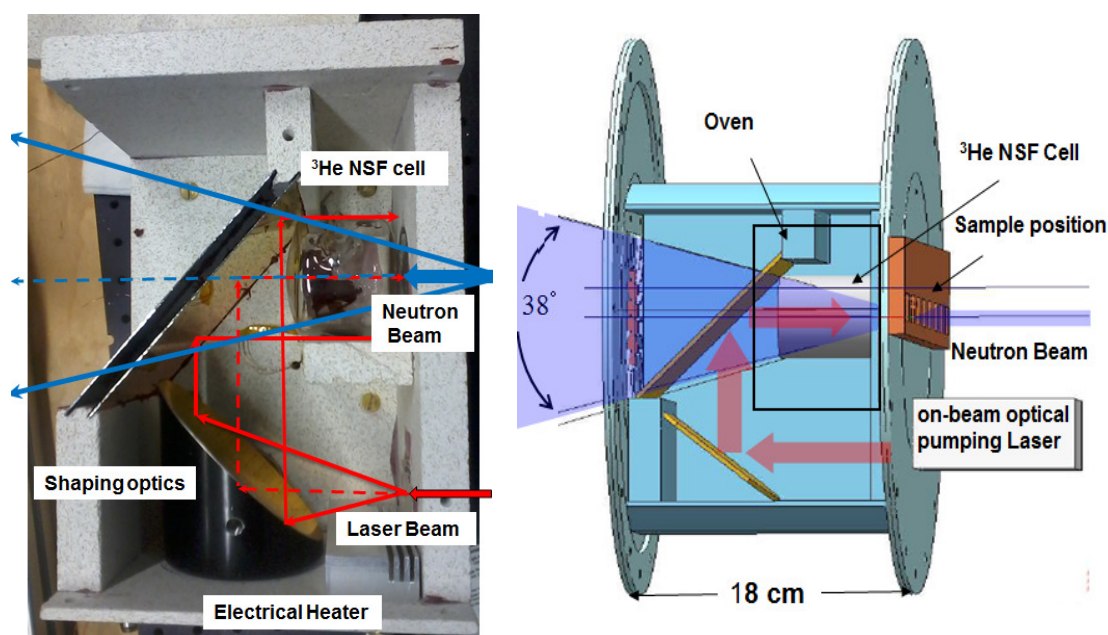


Figure 1. A diagram of the KWS2 magnetic system (right), oven and optics (left). Cut-away drawing of the magnetic cavity showing the optical access (red arrows) and the neutron access, violet.

2. Test of the magnetic cavity

The magnetic cavity has been constructed and tested for the polarized ^3He magnetic lifetime. All of the field coils, the main solenoid with 1 turn/mm and the end-hole compensating coils have been optimized with respect to the number of turns to operate in series from a single power supply. Initial experimental optimization was performed with the 3-axis hall probe mounted to a rail to make 1-D longitudinal field maps of the magnetic cavity. The current of the compensating coils was varied to minimize the magnetic field gradients at the desired cell position of 2-8 cm from the inside of the front of the cavity. Then the number of turns in the compensating coils was adjusted for the series operation. As the result, the final number of turns did not vary by more than one or two turns from calculations.

After this optimization a 5 cm diameter 5 cm long SEOP cell with 0.5 bar ^3He pressure and 930 hour lifetime was polarized externally in our lab and placed in the magnetic cavity aiming the measurements of the ^3He lifetime. The cell was placed at the distance of 2 cm from the front $1.5 \times 1.5 \text{ cm}^2$ hole in the magnetic cavity (right side in Figure 1). Figure 2 shows an NMR free induction decay measurement of the ^3He lifetime [5-6].

After subtracting of the contribution for the intrinsic lifetime of the cell which includes ^3He dipole-dipole relaxation [8] and the wall relaxation of this cell [9], we obtain the measured ^3He magnetic lifetime of 1450 hours for 1 bar pressure or the averaged over the cell equivalent magnetic field gradient of about $2.5 \times 10^{-4} \text{ cm}^{-1}$. Such magnetic performance proves that this non-conventional, ultra short solenoid cavity with rather large openings for scattered beams can provide the required, if not excellent, performance.

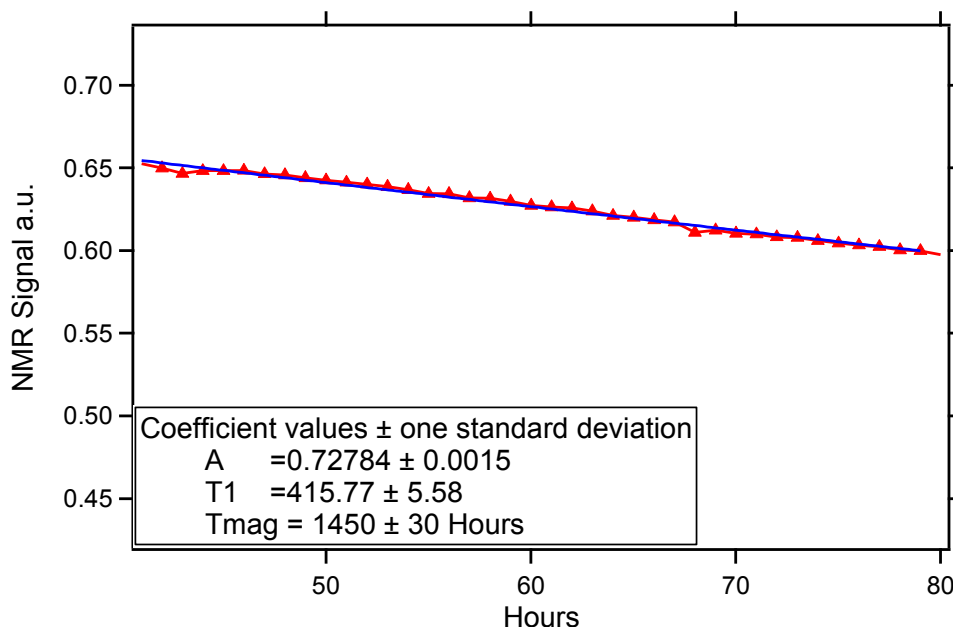


Figure 2. Measured ^3He relaxation time of 415 hours for 0.5 bar ^3He cell placed 2 cm from the entrance aperture of the magnetic cavity. Subtraction of the cells self-relaxation time yields a cell averaged equivalent gradient of $2.5 \times 10^{-4} \text{ cm}^{-1}$.

3. Laser systems upgrade

Our laser systems have also been upgraded. Formerly diode array bars (DAB) with external cavities using Littrow-type configuration were used [10] as narrow-band optical pumping light has been shown to provide the best SEOP performance. However, it was found later that a new compact method using chirped volume Bragg gratings, CVBG, provides a higher absolute ^3He polarization [11]. Additionally, CVBG narrowed lasers give higher laser intensity output for laser diodes of the same power, and contain much fewer optical elements making the cavity more compact, stable and robust for long term operations required for in-situ ^3He polarizer applications. A photo of the new laser cavity is shown in figure 3, where the laser mounted inside a newly designed compact box for mounting the laser and cavity optics is shown, as well as external connections to the power cables and cooling water. This box also provides environmental protection and increased laser security. Two such lasers are used to pump the large 0.85 liter volume of the ^3He NSF cell used on reflectometer MARIA [12], however for the application in question, where a 0.17 litre cell is used one laser is sufficient.

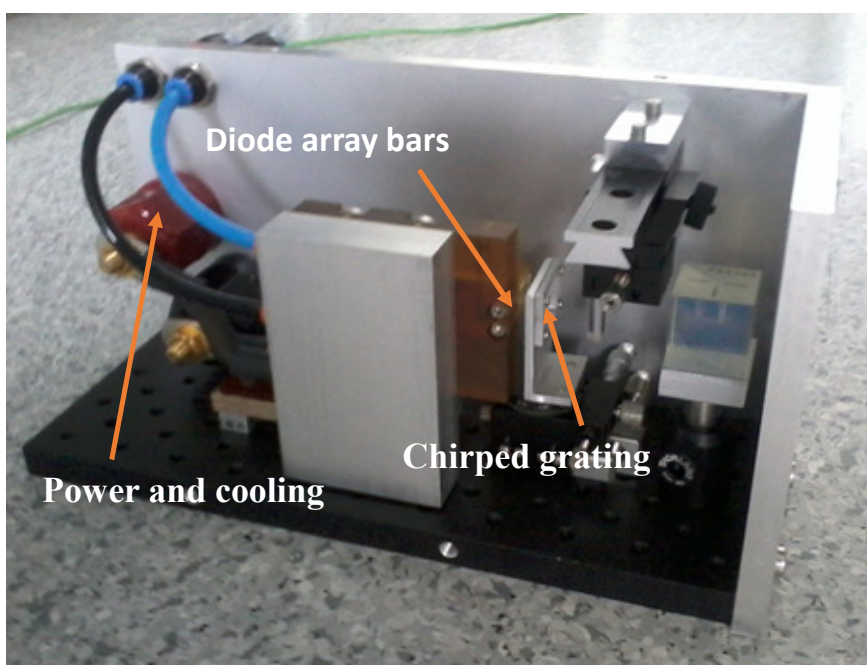


Figure 3. A picture the laser-box of the current volume Bragg grating narrowed laser configuration. This optical setup fit in a custom made enclose box to enclose and mount all the optics and provide connections to power cable and cooling water

4. On-beam test

After several tests and off line optimization, the compact cavity was installed at SANS instrument KWS2 at MLZ for absolute measurements of the ^3He polarization using the neutron transmission technique [13]. It was placed directly after the neutron sample position and next to the window of the detector tank in order to keep the maximum detector coverage as would be done in a typical polarizer SANS measurement (figure 4).

As a first step, the system was installed near the KWS2 instrument two days before the on-beam test. The cell [14] with known opacity (^3He number density-length-cross section product $[\text{He}]/\sigma$) was polarized to saturation and then placed at the sample position as described above. The ^3He polarization was monitored using an NMR system.

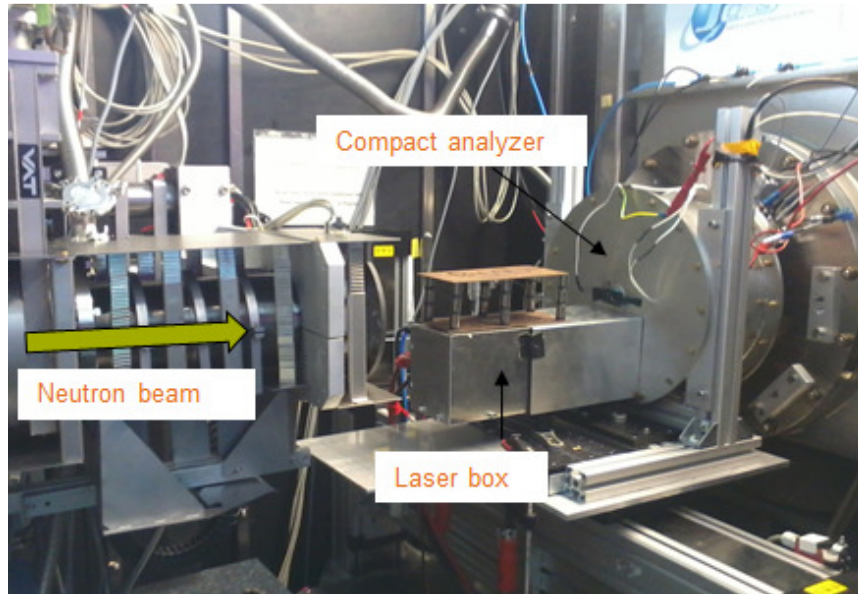


Figure 4. The ultra-compact cavity installed at the sample environment of KWS2 (SANS instrument)

The neutron transmission of the unpolarized incident beam through the ^3He cell was monitored as a function of time. The relative neutron transmission, i.e. the ratio of transmission T of an unpolarized neutron beam through a polarized ^3He cell to the transmission T_0 through the unpolarized cell is:

$$T/T_0 = \cosh([He]/\sigma P_{He} \lambda). \quad (1)$$

Here $[He]$ is the ^3He number density, σ - the spin dependent ^3He neutron absorption cross section, λ - the neutron wavelength, l - the cell length and P_{He} - the ^3He polarization. Thus, by knowing the ^3He number “density-length-cross section” product, $[He]l\sigma$, and the neutron wavelength, the measurements of the unpolarized neutron transmission is a straightforward method to obtain the absolute ^3He polarization.

The product $[He]l\sigma$ of a ^3He cell can be determined from the unpolarized neutron transmission of an unpolarized ^3He cell as a function of neutron wavelength. The unpolarized neutron transmission of an unpolarized ^3He cell is:

$$T_0(\lambda) = T_E \exp([He]l\sigma \lambda). \quad (2)$$

Indeed, by measuring the transmission as a function of neutron wavelength and fitting the function 2, one obtains the value of the exponential which is the number density-length-cross section product, where T_E is the empty cell transmission.

The results of the on-beam test are shown in figure 5. From this data one can see that the maximal obtained ^3He polarization is 56%. Though this polarization is insufficient for a high neutron performance of the SANS analyser, however improvements and further optimization should allow us to approach the level of performance achieved in other in-situ polarizers made by our group [12]. These upgrades are discussed in the next section.

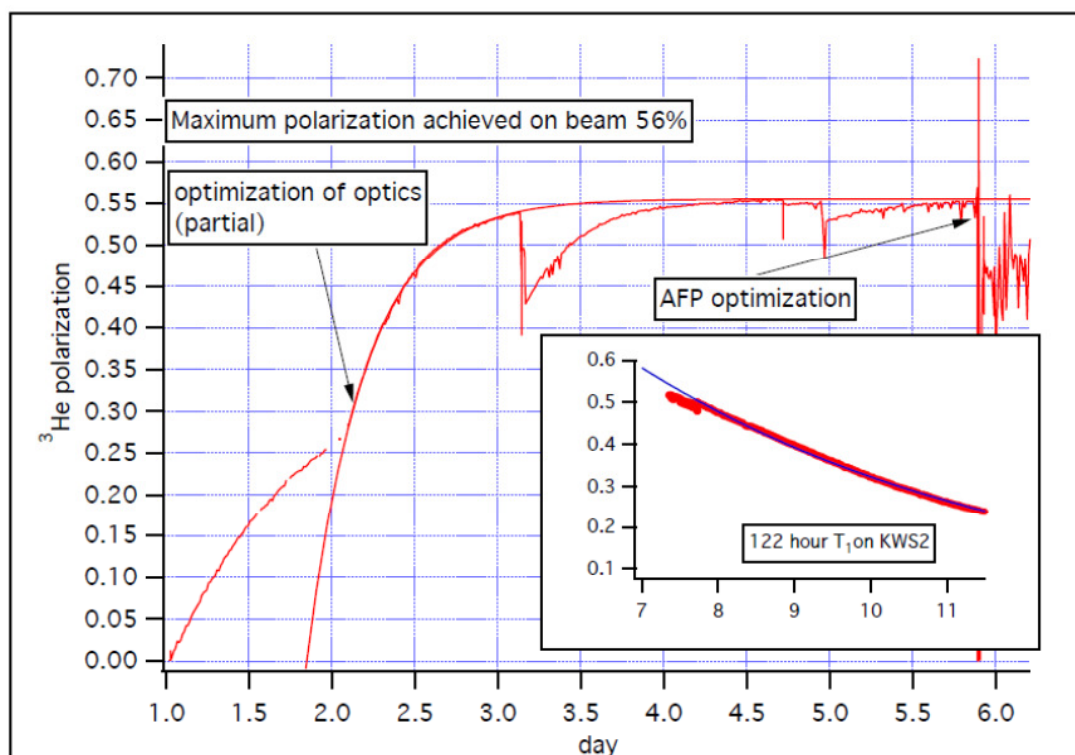


Figure 5. The ^3He polarization as function of time obtained during on-beam test at KWS2, as insert, the measured on beam relaxation time for the polarized cell. Maximum polarization achieved on beam 56% with 7% neutron transmission.

The in-situ ^3He relaxation time for this experiment was measured at the end of the test by turning off the lasers and the cell heating while monitoring the decay of the polarization using NMR system., and the fit gave $T_1=122\text{hours}$ which is sufficiently long compared to the spin-exchange rate and doesn't significantly affect the maximum achievable polarization.

5. Future upgrades

During our on beam test we faced two problems. First, a difficult optics alignment because of the limited access to the laser and expansion-collimation optics inside the compact and closed cavity. In figure 5 one can see how the improvement of the polarization build-up time was achieved by just a partial optimization of the optics alignment of the laser beam. Second, μ -metal plates and the sample position was getting hot and reaching a temperature of nearly 100°C because of the direct contact with the oven inside the cavity. Here one should keep in mind that the sample position, which is placed within 3 cm from the front of the ^3He NSF cell, must not be significantly heated to avoid negative influence on the performance of the sample environment and, in turn, samples contained within it because of soft matter and biological samples are typically temperature sensitive.

To solve these problems the system is undergoing further testing, optimization and mechanical refinement before eventual user operations. The optimization and refinement include appropriate cooling of the system and the sample (to avoid denaturation of proteins placed 3cm from the oven), laser collimation and optics adapted to a such small oven (better illumination of ^3He cell with narrowed laser), mechanical design and fixation of the system to allow an easy use (automatically placed on and out of the beam when needed). The new proposed design is shown in figure 6. The system consists of aluminum box with plates fixed $\frac{1}{2}$ cm from each side of the magnetic cavity to not reduce the angular coverage of the detector, the lateral sides were used to fix two cooling units to cool efficiently the system through $\frac{1}{2}$ cm gap between the μ -metal and the aluminum plates. All cables connections are fixed via feed through on the lateral side of the box to facilitate an easy handling.

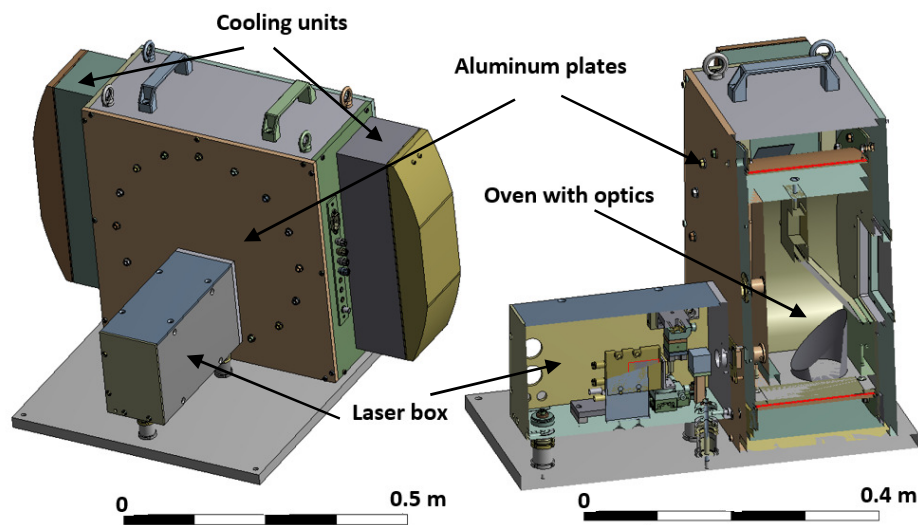


Figure 6. A new proposed design of the ultra-compact cavity including cooling unit, laser box, laser collimation and optics adapted to a such small oven

In order to optimize the cooling power needed to keep the temperature at the cell position of about 200°C needed for the optical pumping, and less than 30°C at the sample position, we have performed several FEM calculations of the heat transfer inside the cavity using ANSYS software [15]. The first calculations of the heat transfer was performed using the old design of the compact cavity without cooling units, to compare the simulation results with results obtained during the on-beam experimental test. The result of simulations (figure 7) are in good agreement with temperatures measured using PT100 temperature sensor at the surface of the μ -metal plates and inside the oven at the cell position. One can see that the temperature at sample position reach 132°C.

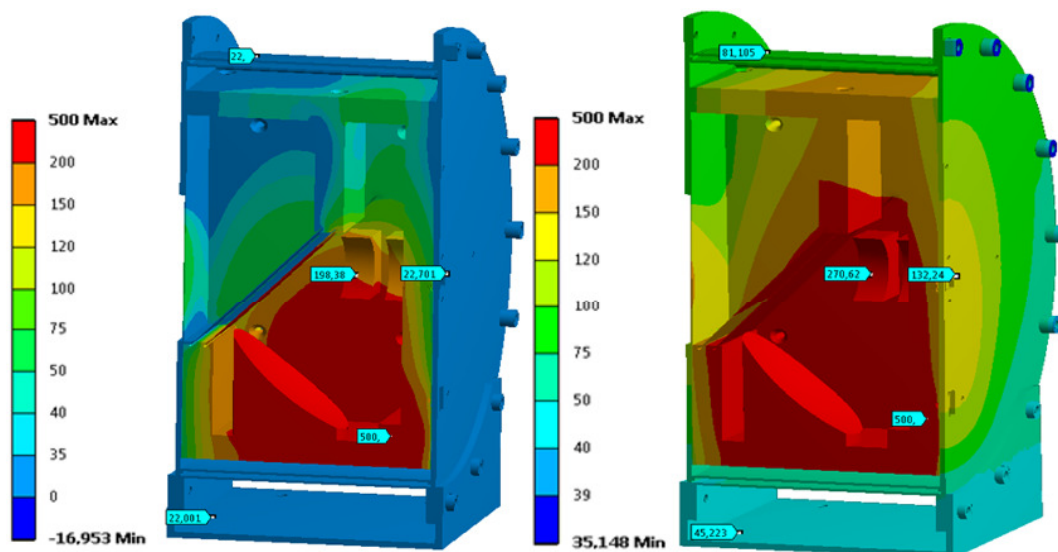


Figure 7. Results of the heat transfer calculations using ANSYS software. Right: without cooling unit, using the old cavity design; temperature at the sample position is 132°C. Left: for the new proposed design with cooling units; temperature at the sample position is 23°C.

The second series of calculations were performed using the new design taking into account the gap of 1mm between the oven and the μ -metal plates. One can see from figure 7 that with the new system we can obtain optimal parameters (200°C at the cell position and less than 30°C at sample position) with cooling power of 250 W and the same heating power as in the old design. As shown in figure 1 the sample is located in the brown holder directly before the right end cap outside the cavity and can be additionally cooled to just above the freezing temperature for biological samples in order to prevent them from degrading.

The system is now under construction and will use two cooling units, each with 150 W cooling power that will allow for a better optimization of the alkali-metal vapor density. An improved mechanical fixation of the optics and the laser box will also allow for the better alignment of the optic and the illumination of the cell. A small window at the back side of the oven will allow us to control the optics inside the oven and check the complete illumination of the ^3He cell.

6. Conclusions

We have designed, prototyped, and tested an ultra-compact SEOP ^3He polarizer to be used as the in-situ analyzer for SANS diffractometer KWS2. The magnetic cavity design assures the relative field gradients less than 10^{-4} cm^{-1} and a large detector coverage of $\pm 19^\circ$. Due to the difficulty of laser alignment (optic inside the closed oven) and the high temperature at the sample position and μ -metal plates (impossibility to increase further the temperature to optimize the alkali metal vapor density) the first on-beam test gave maximal polarization of 56%. Based on simulation results, the system will undergo upgrades and further tests to improve the maximum reachable on-beam polarization. Once the critical mass of components is available we will proceed with neutron sample measurements and data collection and analysis techniques.

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