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COMPASS dilution cryostat: testing of a new mixing chamber and analysis of thermometer data

Summer Student Report

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1 Introduction

COMPASS (COmmon Muon and Proton Apparatus for Structure and Spectroscopy, NA58) is a fixed target experiment in the Super Proton Synchrotron (SPS) of CERN. It had its first technical run 2001 and has taken physics data since 2002. The purpose of the muon programme is to study nucleon¹ spin structure.

COMPASS investigates the nucleon by scattering a beam of leptons off a target of nucleons. This leads to deep inelastic scattering where the high energy muons break apart the nucleon. The internal structure of the nucleon and its spin can be studied by using polarized beam with a polarized target.

Previous experiments at CERN (EMC and SMC²) and DESY (SLAC and HERMES) have shown that valence quarks carry 20 – 30% of their parent nucleon spin. The remaining nucleon spin can be explained by gluon spin or by angular momentum of partons. COMPASS aims to measure the gluon contribution to nuclear spin using a longitudinally polarized 160 GeV muon beam and longitudinally polarized nucleons in a solid state target. Photon-gluon fusion process is needed to obtain the gluon contribution.

2 ⁶LiD target material

The target consists of small crystals of ⁶LiD each a few mm in diameter. The material is held in a mesh container and surrounded by the liquid helium below 1 K in

¹'nucleon' is the collective term for a proton or neutron

²Spin Muon Collaboration

a mixing chamber. The overall effective polarization in the target material is a key factor since there is no difference in obtaining gluon spin from a proton or a neutron. ${}^6\text{LiD}$ has a large fraction of polarizable nucleons and high effective nuclear polarization. These factors reduce beam time needed to obtain certain statistical accuracy in asymmetry measurement. The target is separated into 60 cm long and 3 cm in diameter upstream and downstream parts. The two cells are oppositely polarized to cancel the false asymmetry due to the time-dependent variations in the beam intensity.

3 Dilution Refrigerator

A dilution refrigerator uses dilution of ${}^3\text{He}$ in ${}^4\text{He}$ to reach temperatures as low as 0.002 K. The target lattice temperature is directly related to the maximum polarization that can be achieved. Thus the target should be cooled below 300 mK for efficient dynamic nuclear polarization. Temperatures below 100 mK are required to slow down sufficiently the spin-lattice relaxation of the polarization in the fixed target. A superconducting solenoid magnet with 2.5 T field surrounds the target and the nuclear spins are polarized parallel or anti-parallel to this magnetic field. The magnet must be kept at a low temperature around 4 K with liquid helium to retain its superconductivity. A dilution refrigerator is the only practical method for cooling the target material since it can have high cooling power to compensate the heat generated by the microwaves during dynamic nuclear polarization.

3.1 Cooling power of a dilution cryostat

The cooling relies on the enthalpy of mixing of two quantum liquids. The temperature of the superfluid phase transitions of liquid ${}^4\text{He}$ is lowered if the Bose liquid is diluted with the Fermi liquid ${}^3\text{He}$. Eventually the superfluidity of ${}^4\text{He}$ ceases to exist. If the helium mixture is cooled to below its triple point at 0.87 K the liquid separates into two phases: one rich in ${}^4\text{He}$ and the other of pure ${}^3\text{He}$. ${}^3\text{He}$ maintains a concentration of 6.6 % in the ${}^4\text{He}$ rich liquid even at 0 K.

The ${}^3\text{He}$ atoms drift toward the top of the surface due to a difference in densities of the liquids or the ${}^3\text{He}$ atoms are lighter than ${}^4\text{He}$ atoms. Cooling is achieved by pumping on the mixture from a separate evaporator volume (still) and forcing the ${}^3\text{He}$ atoms to move from the pure ${}^3\text{He}$ phase to the ${}^4\text{He}$ rich phase in mixing chamber. The still and mixing chamber are connected together with heat exchangers. They operate at different temperatures: for the still typically 0.6 - 0.8 K and for the mixing chamber 20 - 400 mK. The enthalpy of ${}^3\text{He}$ in the dilute phase is larger than the enthalpy of ${}^3\text{He}$

in the concentrated phase. The flow of ^3He atoms across the phase boundary causes cooling. Enthalpy balance for the mixing chamber is

$$n_3(H_d \cdot T_{mc} - H_c \cdot T_{mc}) = n_3(H_c \cdot T_{ex} - H_c \cdot T_{mc}) + Q + Q_{leak} + Q_\mu. \quad (1)$$

From this the cooling power of the dilution refrigerator

$$Q = n_3(H_d \cdot T_{mc} - H_c \cdot T_{ex}) - Q_{leak} - Q_\mu. \quad (2)$$

Here n_3 is the ^3He flow rate, H_d the enthalpy of ^3He in diluted phase, H_c the enthalpy of ^3He in concentrated phase, T_{mc} the outlet ^3He temperature, T_{ex} the inlet ^3He temperature, Q the cooling power, Q_{leak} the external heat leak, and Q_μ the energy deposit of the muon beam.

3.2 Operation of a dilution refrigerator

^3He gas returning from the pumping system is precooled by cold ^4He gas at 10 - 300 K before condensing in a pumped ^4He evaporator bath at 1.5 K. The flow impedance before the still is controlled by a needle valve. It produces sufficient pressure so that the ^3He will condense at 1.5 K. Heat exchangers cool the ^3He to a low temperature 100 - 300 mK before it enters into the mixing chamber. The dilute phase on the bottom of the mixing chamber is connected to the still with the heat exchangers. The ^3He liquid going from the mixing chamber to the still cools down the incoming ^3He in the heat exchangers.

By pumping on the still and resupplying the condensation line with ^3He gas, there is a closed ^3He circuit producing continuous cooling. In the mixing chamber the ^3He crosses the phase boundary which gives rise to the cooling. It leaves the mixing chamber to enter into the still where it will evaporates. The circulation of the ^3He is maintained by a 8 Pfeiffer roots blowers with pumping speed 13500 m³/h.

4 The mixing chamber

The mixing chamber is a continuous fiber-glass/epoxy tube with a hollow hemispherical top. It was made by the Yamagata University in Japan. The fiber-glass has a high mechanical strength, which is needed due to the 1 atmospheric operation pressure inside. The epoxy makes the material leak tight.

The chamber holds the helium mixture, the target cells and the NMR coils around the ^6LiD . It must be able to withstand cool down from room temperature to below 1

K. Before use the mixing chamber must be leak tested. The leak test should be carried out at as low temperatures as possible in order to extrapolate its behavior during the experiment.

4.1 Preparing the mixing chamber

The new mixing chamber came as two separate parts (cylindrical tube and hemispherical top) which had to be connected before testing and using the chamber. Before attaching these pieces together, measurements of the tube and top were taken.

Width of tube [mm]	inner diameter [mm]	outer diameter [mm]	width of top [mm]
0.51	50.4	50.75	0.54
0.47	50.44	50.57	0.5
0.50	50.5	50.54	0.52
Average			
0.505	50.44	50.62	0.52

Table 1: Measured dimensions of the mixing chamber at room temperature.

The cylindrical mixing chamber tube was glued to a steel (AISI 316L) flange. Stycast 1266 used takes approximately 8 hours to cure. The epoxy was painted onto the inner upstream part of the tube and on the flange. Weights held the tube parallel and perpendicular to the flange while drying.

This bond was reinforced by wrapping layers of fiber-glass thread around the point of contact together with Stycast 1266. Only two layers of fiber-glass thread were used not to apply too much of a contracting force on the chamber while cooling down.

It was necessary to glue the top of the mixing chamber to the tube. To ensure no glue dripped inside the vessel, tissue was packed into the downstream end of the tube. A wire attached to the tissue allowed it to be easily removed once the glue set.

5 Strength of the tube: positive pressure

The mechanical strength of an object is the maximum stress that the material can withstand before failure. It is necessary to calculate the mechanical strength of the mixing chamber to avoid exerting too much pressure on the structure.

We consider the first the cross section of the glass fiber tube. Here z-axis is along the tube and x- and y-axis in transverse direction. Given a fixed pressure inside the tube

$p = 1$ bar the force on a small surface element on the tube can be separated into two components: $F_x = F \cdot \cos\theta$ and $F_y = F \cdot \sin\theta$. The area on which the force is acting is

$$dA = L \cdot R \cdot d\theta, \quad (3)$$

where L is the length of the tube, R is the inner radius of the tube and $d\theta$ is the small angle subtended by the sector. Thus the force in the x-direction can be calculated to be $2 \cdot L \cdot p \cdot R$ where p is the pressure inside the tube from:

$$F_x = \int_{-\pi/2}^{+\pi/2} L \cdot p \cdot R \cdot \cos\theta d\theta. \quad (4)$$

The tension inside the tube wall is calculated by dividing the force by the area of the wall

$$\sigma_{xy} = \frac{F_x}{d \cdot L \cdot 2} = \frac{p \cdot R}{d}, \quad (5)$$

where d is the thickness of the tube. For the tube radius of 25 mm and wall thickness of 0.5 mm the tension is $\sigma_{xy} = 5$ MPa or 50 bars. The longitudinal stress σ_z can be estimated to be

$$\sigma_z = \frac{\pi R^2 p}{2\pi R d} = \frac{pR}{2d} \quad (6)$$

giving $\sigma_z = 2.5$ MPa.

The Young's modulus of the fiber glass is $E = 72,000$ MPa [1]. According to the Hook's law the tension is directly proportional the the deformation. Thus the dimensions of the tube change

$$\varepsilon = \frac{\sigma}{72000MPa}. \quad (7)$$

This is about $7 \cdot 10^{-5}$ in transverse direction and only half of that in the longitudinal direction. Thus the radius of the tube changes only about $2 \mu\text{m}$ and the length of the tube $52 \mu\text{m}$. These are negligible small numbers compared to the thermal shrinking -0.1 - -2.2 % for different materials when cooled to below 1 K. Thus thermal shrinking can produce much higher tensions than the pressure inside the mixing chamber. This is especially true for the gluing of the glass fiber to the steel flange.

The spherical shape of end top part helps to reduce the material thickness without introducing too much stress to the material. For a flat top part also the torque inside the wall should be considered, but for the spherical part this contribution vanishes. The stress inside the hemisphere is the same as shown in Eq. 6 or about 2.5 MPa.

5.1 Negative pressures

Parameters of the tube are:

- Modulus of elasticity $E = 72.0$ GPa
- Poisson's ratio $\rho = 0.22$
- Theoretical collapsing pressure P_{crit}
- Fiber stress at yield point $\delta = 60$ MPa
- Length of the chamber $L = 1.5$ m
- Approximate eccentricity of tube $R0 = 6 \cdot 10^{-8}$

Collapsing pressure for the hemispherical end

$$P_{crit1} = \frac{2 \cdot E \cdot t1^2}{R1^2 \cdot (3(1 - \rho^2))^5} \quad (8)$$

so $P_{crit1} = 3.646 \cdot 10^4$ Pascals.

Collapsing pressure for a cylindrical tube ($L > 20 R^2$) P_{crit2} is

$$\frac{\delta \cdot E \cdot t^2 \cdot 4 \cdot m \cdot (1 - \rho^2) \cdot R3^2}{4 \cdot m \cdot (1 - \rho^2) \cdot R2^3 \cdot ((4 \cdot m \cdot (1 - \rho^2) \cdot R3^2) - (\delta \cdot 4 \cdot (1 - \rho^2) \cdot R3^2) + m \cdot (1 + 6 \cdot m \cdot n) \cdot E \cdot t2^3)} \quad (9)$$

Here $m = R2/t2$ and $n = R0/R2$. This gives a value $P_{crit2} = 1.512 \cdot 10^5$ Pa or 1.5 bar.

Put here reference from where these formulas were taken!

6 Leak-testing the Mixing Chamber

With the mixing chamber assembled it is necessary to test its performance. Using a leak detection system, the chamber is tested for helium leaks at room temperature and below (the ^6LiD must be kept below 100K to retain the paramagnetic centers)

The mixing chamber must first be placed in a vacuum chamber. The old layer of indium is removed with wooden utensils from the large flange [f2] to which the mixing chamber must be attached. After cleaning the surface with alcohol, a new layer of indium is added to f2 to ensure an airtight fit between the two flanges. Indium is chosen here above copper since it is more malleable.

The two flanges are fixed together with screws. Opposite screws are tightened simultaneously so the indium is compressed equally around the circle. A rubber o-ring coated with vacuum grease is added to f2 to give better contact and avoid dust.

The vacuum tube is checked for foreign objects inside before inserting the mixing chamber. It is important to clean all surfaces with alcohol to avoid dust errors.

The manometer and valve are connected to the vacuum cavity. This manual valve can be used to evacuate the vacuum space slowly. These are all linked to the Leak Detector which creates the vacuum and reads the level of helium entering the vacuum cavity from the mixing chamber. A scope meter (Fluke 105B series 2) is attached to the leak detector. This device gives an instantaneous graph of the leak-rate of the chamber as a function of time.

6.1 The Leak Detector: ASM 181 t2 Alcatel

The ASM 181 t2 is the most powerful Alcatel leak detector. It can test volumes up to 1000 liters and pinpoint very small leaks in large detectors. The leak detector is a portable device consisting of a rotary vane mechanical roughing pump, a turbo-molecular pump, control console and a continuous bar graph leak rate and vacuum pressure display.

It firstly creates a vacuum in the vacuum chamber which is continuously maintained. An ideal vacuum chamber should maintain the vacuum pressure reached at the moment of its separation from the pumps. A real chamber presents a rise in pressure after being isolated from the pumping system due to gas penetrating through leaks or permeation.

The leak rate is the quantity of gas flowing per unit time into the system and is expressed in milli-bar liters/second. Once the vacuum chamber has reached a sufficiently low pressure, helium is allowed to flow directly to the mixing chamber. Any helium escaping through cracks in the tube or diffusing through the tube-walls is noticed by the leak detector in the vacuum chamber.

6.2 Helium Leak-testing

June 22: It must first be checked that the mixing chamber can survive a vacuum. Starting at 940 Grs/cm² it takes approximately 12 minutes to get to zero pressure. During this period it is important to listen for cracks possibly occurring in the mixing chamber. The pressure in the vacuum cavity steadily decreases until 500 grs/cm². The vacuum is slow to create probably due to the large volume involved. A leak test can only be started once the pressure has reached 1mbar. Since there is no problem with 1bar pressure inside the chamber, the chamber is filled with helium

A helium line is taken from the gas room. This gas is maintained at constant pressure from the pump room. After pumping the vacuum cavity for an hour, it is at a pressure

of 2mbar. The helium gas is sprayed along the piping connecting the leak detector to the vacuum chamber. This is to check for leaks along the line. A leak was found at the manometer. A rubber o-ring coated with vacuum grease was added to the manometer connections and the bolts tightened. After testing again, no leaks were found.

The helium gas is now piped directly into the mixing chamber and the leak detector console is monitored. If a leak is present in the mixing chamber, there will be a sharp rise in the leak rate on the detector console. Diffusion of helium through the walls of the chamber will result in a slow rise of the leak rate after a few minutes. The mixing chamber appears to be leak tight since there is no change. Cavity pressure is $6 \cdot 10^{-2}$ mbar and helium leak rate $4 \cdot 10^{-6}$ mbar l/s.

June 23: In order to examine the behavior of the mixing chamber at low temperatures, liquid nitrogen (at 77K) must be used to cool the system. Copper pipes conduct the nitrogen cooling and pipe the helium directly to the mixing chamber. Here the nitrogen is not directly added to the system in case the thermal tension due to the extreme temperature of the nitrogen causes cracks in the mixing chamber.

The copper pipes are held together and wrapped in polystyrene along its length and at the top to avoid damaging the inside of the mixing chamber.

A thermocouple² attached to a multimeter reads the temperature of the system. The thermocouple is first tested by comparing the room-temperature it reads to that of an Infra-Red thermometer. The thermocouple is also fit into the polystyrene case enclosing the copper pipes.

The thermocouple is not as long as the copper pipes; hence we are only measuring the upstream temperatures and have no information on the temperature downstream in the chamber. This contributes a substantial level of error to our temperature/leak rate relation.

The liquid nitrogen is connected to the mixing chamber. Rubber o-rings are not used in the nitrogen piping since it would not stand the temperatures, instead aluminum caps are used. The nitrogen leaving the chamber is directed outside so not to pollute the experimental area. The point at which the pipes enter the mixing chamber is not airtight, but this helps regulate the pressure and dispel water condensation inside the mixing chamber.

6.3 Taking measurements and analyzing the data

The heat exchange between the nitrogen and the helium regulate the temperature throughout the chamber so it will not cool too quickly. Such thermal tensions could easily cause cracks in the fiber glass. It would have been beneficial to the cooling of the

mixing chamber to pre-cool the helium entering the system, but this was too difficult a task to undertake for such an experiment. Hence the cooling of the chamber is a slow process.

The pressure in the nitrogen Dewar decreases quickly due to the falling level of nitrogen. To keep the rate of nitrogen flow steady, the Dewar must be re-pressurized to maintain a pressure of 0.4bar. This can be simply done by pumping helium into the Dewar.

The plot below shows the scope meter graph for the helium leak (y-axis) as a function of time (x-axis) for the first day of the readings (23-6-2004). The leaks increased when the Dewar pressure reduced, hence reducing the cooling power of the nitrogen and thus the temperature of the mixing chamber. Once the Dewar was re-pressurized and the temperature decreased, the helium leak rate also decreased. From these results it seems that diffusion of the helium is a function of the mixing chamber temperature.

The leak rate, the pressure of the vacuum cavity and time were recorded every decreasing 5K of the temperature in the mixing chamber.

The leak rate is a more accurate temperature guide than the thermocouple since the thermocouple is highly dependent on the flow of gas around it.

The temperature ceased decreasing at 206K. Re-pressurizing the Dewar had no effect on this limit and it was concluded that the system in its current state was insufficient to reach the necessary low temperatures to test the mixing chamber. Modifications were thus made to the system.

June 24: Modifications:

1. Solder the helium tube holder closed to improve the leak rate readings.
2. Solder the helium tube to the incoming nitrogen tube. This will cool the helium gas a little before it enters the mixing chamber and not hinder the cooling mechanism as much.
3. Fix the thermocouple so that it is not touching the copper pipes, (before soldering, the copper pipes were cleaned and sanded).

With this modified system, the temperature was much slower to cool, leading to the conclusion that the thermocouple was previously touching the nitrogen tubes and hence gave false chamber temperature readings. A temperature of only 220K could be reached.

A new method of directly cooling the chamber was attempted. The helium was disconnected and instead the nitrogen was re-directed to the helium pipe to directly cool the chamber. This had little effect on the chamber temperature, probably due to the narrowness of the pipe.

As a final modification, the link joining the in/out nitrogen pipes was removed. The nitrogen and helium both flowed directly into the mixing chamber.

The temperature of the chamber decreased steadily and readings were taken for approximately every 5 degree Kelvin decrease. The following graph shows the helium leak rate as a function of temperature. It would be advantageous if a relationship between the leak rate and the temperature could be defined.

The slope of this graph is 0.000000117. The graph has a linear form so I conclude that the helium leak rate is proportional to the temperature of the mixing chamber. This is advantageous since there should thus be a very small leak in the milli-kelvin range as reached in the COMPASS experiment.

At 120K there was a sharp increase in the leak rate as visible in the scope meter graph below. Such a sudden increase had to be due to a leak in the mixing chamber, since diffusion of helium is noticed as a steady curve. It was thus necessary to warm-up the chamber to inspect the damage.

The leak decreased as the temperature was increased. Air was let in to the vacuum cavity (using the manual valve) and then pumped out. Once the pumping stopped the helium level decreased again. Restarting the pump caused the temperature to decrease, so there was possibly water vapor in the vacuum chamber conducting heat to the mixing chamber.

At room temperature the leak rate was measured every minute in order to determine the diffusion time constant.

This graph plots the leak rate (mbar l/s) against the time (seconds). The slope of this graph is found to be:

The plot is linear in form so there is a definite leak in the chamber.

June 25: After evacuating the vacuum cavity, the mixing chamber was again tested for leaks at room temperature. The helium leak rate immediately increased, implying a leak in the chamber rather than diffusion through the walls. The helium saturated at 0.00032mbar and at this point the chamber was removed.

The scope-meter graph for the room-temperature leak test after detection of a leak the previous day:

Three small cracks were visible in the centre of the mixing chamber tube.

These cracks may be sealed with Stycast glue and reinforced with fiber glass thread. The different thermal contractions between the Stycast and the fiber glass must be considered. Mixing the fiber glass thread with epoxy should curb any possible damage. Another cold leak test must then be done to test the stability of the glue.

Liquid N₂ has about 60 times the latent heat of evaporation of liquid helium and

at 77K most of the heat capacity of the substance to be refrigerated has already been removed.

An electrical circuit consisting of two dissimilar metals joined at each end, in which an e.m.f is produced when the two junctions are at different temperatures.

References

- [1] http://www.cem-filreinforcements.com/cf_ar_product.html