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A compact membrane-driven diamond anvil cell and cryostat system for nuclear resonant scattering at high pressure and low temperature

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A new miniature panoramic diamond anvil cell (mini-pDAC) as well as a unique gas membrane-driven mechanism is developed and implemented to measure electronic, magnetic, vibrational, and thermodynamic properties of materials using the nuclear resonant inelastic X-ray scattering (NRIXS) and the synchrotron Mössbauer spectroscopy (SMS) simultaneously at high pressure (over Mbar) and low temperature (T < 10 K). The gas membrane system allows *in situ* pressure tuning of the mini-pDAC at low temperature. The mini-pDAC fits into a specially designed compact liquid helium flow cryostat system to achieve low temperatures, where liquid helium flows through the holder of the mini-pDAC to cool the sample more efficiently. The system has achieved sample temperatures as low as 9 K. Using the membrane, sample pressures of up to 1.4 Mbar have been generated from this mini-pDAC. The instrument has been routinely used at 3-ID, Advanced Photon Source, for NRIXS and SMS studies. The same instrument can easily be used for other X-ray techniques, such as X-ray radial diffraction, X-ray Raman scattering, X-ray emission spectroscopy, and X-ray inelastic scattering under high pressure and low temperature. In this paper, technical details of the mini-pDAC, membrane engaging mechanism, and the cryostat system are described, and some experimental results are discussed. *Published by AIP Publishing.* https://doi.org/10.1063/1.4999787

I. INTRODUCTION

Over the last forty years since Ruby[1] first proposed the idea of studying the Mössbauer effect using synchrotron radiation and Gerdau *et al.*[2] successfully demonstrated its experimental possibility ten years later, nuclear resonant scattering (NRS) using synchrotron radiation has greatly evolved and has attracted many new research applications over a broad range of sciences.[3–8] In addition to the synchrotron radiation Mössbauer spectroscopy (SMS) which can be performed either in the time domain[9] or the energy domain,[10] nuclear resonant inelastic X-ray scattering (NRIXS) was developed about two decades ago[11,12] and was immediately recognized as a powerful tool to study the thermal and lattice dynamic properties of materials. The resulting partial phonon density of states is readily comparable with theoretical simulations via density functional theory. Determinations of thermodynamic, vibrational, and elastic properties such as sound velocity, vibrational entropy, kinetic energy, and specific heat made the technique attractive to diverse fields such as physics, chemistry, biology, materials science, and geology. Due to its isotopic selectivity, well-collimated beam properties of SR, and enhanced scattering cross-section of nuclear isotopes, NRS from small samples and thin films have been particularly attractive. Especially, the application of the NRS on small samples in high pressure research using the diamond anvil cell (DAC) technique has been extremely fruitful and productive in the last two decades. NRIXS experiments under extreme conditions of pressure[13] and temperature[14] have been performed extensively, and studies under high pressure and high temperature have been carried out using the laser heated diamond anvil cell techniques.[15–18] Recently we added new capabilities at 3-ID, Advanced Photon Source (APS), Argonne National Laboratory (ANL), that made it possible to perform simultaneous SMS and NRIXS experiments at high pressure (HP) and low temperature (LT).[19] However, in our earlier development, the lowest temperature reached on the sample was around 20 K due to the fact that the DAC holder is not directly cooled by the liquid He. This limited our abilities to study the magnetic properties and superconductivity of some materials since it was important to lower the temperature below 20 K. Furthermore, in our previous design, the pressure of the DAC was not *in situ* adjustable, meaning that the pressure has to be adjusted after warming up the whole system to an ambient temperature, which is inconvenient, unstable, and time consuming. Also, this lack of *in situ* pressure adjustability makes some experiments such as phase boundary mapping difficult and less accurate.

In our current design, we have developed a new cryostat system where the liquid helium flows through the DAC holder so that the temperature on the sample can be further lowered. The DAC holder has been revised so that the contact area between the holder and the DAC is greatly improved. In addition, we have incorporated a compact and efficient

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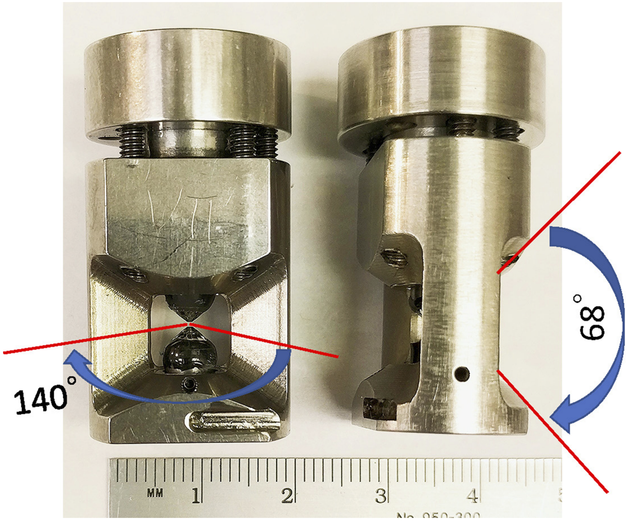

FIG. 1. The mini-pDAC made of hardened Vascomax 350, with two wide side-openings for NRIXS signals.

gas-driven membrane system to precisely control the pressure of the sample at cryogenic temperatures. With these improvements, we can control and adjust the pressure accurately at an even lower temperature on the sample for both NRIXS and SMS measurements. As a matter of fact, due to the wide accessible openings of our system, the same instrument can be easily used, and some may need slight modification, for X-ray radial diffraction, X-ray momentum-resolved inelastic scattering, X-ray emission spectroscopy,[20] and X-ray Raman scattering.[21] In this paper, the detailed design of the system is discussed, and some experimental results are presented.

II. CONSTRUCTION OF THE GAS MEMBRANE-DRIVEN MINIATURE PANORAMIC DIAMOND ANVIL CELL SYSTEM

The major challenge for NRIXS studies at high pressure and high temperature (HPLT) is that it requires big solid-angle openings around the sample and a short distance between the detector and the sample in order to maximize nuclear resonant signals. In the regular panoramic DAC[13] [with outer diameter (OD) of 50 mm] for high pressure NRIXS at room temperature, the shortest distance between the sample and the avalanche photodiode detector (APD) (with a detector size of 10 mm × 10 mm) while the detector is placed through the wide opening of the DAC is 10–12 mm. However, it is fairly difficult to use such a bulky panoramic DAC in a vacuum chamber to achieve low temperature while still maintaining a small distance between the detector and the sample. To overcome this intrinsic problem, a new miniature panoramic diamond anvil cell (mini-pDAC) has been developed at 3-ID, APS, ANL, as shown in Fig. 1. The current mini-pDAC is an improved version of the earlier design[19] with its body length extended by about 6 mm to improve its stability. The standard mini-pDAC is made of hardened Vascomax 350 with a hardness of Rockwell C55. To study the magnetic properties of materials at LT and HP conditions, a non-magnetic mini-pDAC made of BeCu is also manufactured. The mini-pDAC is a piston-cylinder type of diamond cell and pressure can be held and adjusted through four screws on the piston side. The outer diameter of the mini-pDAC is 20.0 ± 0.1 mm. Two wide openings are cut on the cylindrical part, each with 140° equatorial angle and 68° along the DAC axis for the acceptance of nuclear resonant fluorescence signals from the sample. When a low-Z material, e.g., Be, is used as the gasket, nuclear fluorescence photons are collected through these two side openings while the incident X-ray travels along the DAC-axis through the diamond.

In addition to the four screws to adjust pressure on the mini-pDAC manually, a gas membrane mechanism was developed so that the pressure on the sample can be adjusted and maintained at low temperature remotely and accurately without the need to warm up the cryostat. This is particularly important in phase boundary determinations since pressure adjusted at ambient conditions cannot be maintained at low temperatures.

A picture and the schematic structure are shown in Fig. 2. The membrane is a standard double-diaphragm design[22] with a 50.8 mm outer diameter (OD) and an inner diameter (ID) of 23.8 mm. Although the OD of the mini-pDAC is much smaller than the membrane, the design of the pusher allows the large membrane area to apply sample pressure efficiently and be able to reach higher pressure on the sample. Pressurized helium gas

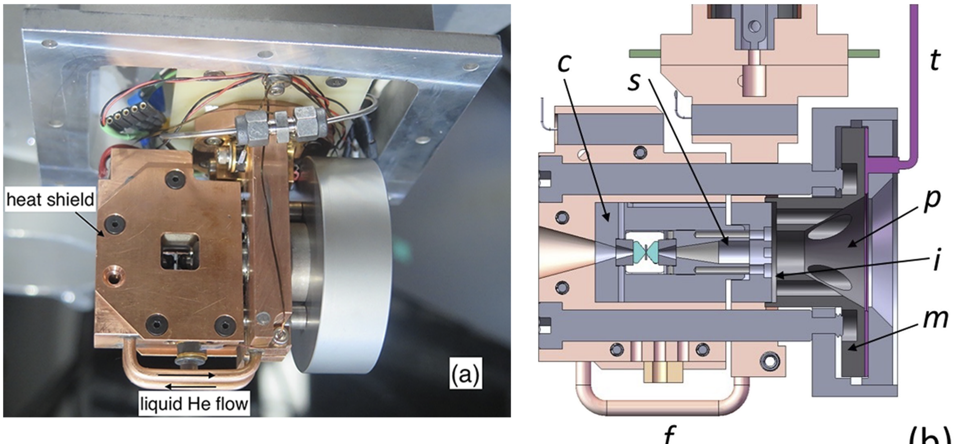

FIG. 2. (a) Picture of the cryostat cold head, gas membrane system, and diamond anvil cell holder. (b) A cross-sectional view of the instrument. "c" and "s": cylinder and piston of the mini-pDAC; "p": the pusher of the membrane; "t": a 1.5875 mm OD steel capillary tube extended to outside the cryostat for helium gas supply; "i": a ceramic thermal insulation layer between the cell and the pusher; "f": liquid helium flow line around the cell; "m": the membrane.

inflates the membrane which produces axial displacement and applies force to the diamond anvil cells through the pusher "*p*." The warm-gas chamber is thermally insulated from the cold sample by a piece of ceramic "*i*" to minimize the thermal load for the mini-pDAC in the cryostat.

III. DESIGN OF THE FLOW CRYOSTAT AND VACUUM SHROUD

For high pressure experiments dealing with small sizes (~10 μm) of samples, a continuous-liquid-helium-flow cryostat is generally chosen to avoid possible vibration of the sample. In our previous design,[19] a commercial cryostat was used. Since the DAC holder was not directly cooled by the liquid helium, the lowest sample temperature of the system was limited to 24 K. In our new design of the flow cryostat, the liquid helium flows through the copper holder of the mini-pDAC as shown in Fig. 2. With additional heat shielding between the sample and windows, the sample temperature in the mini-pDAC could be further lowered, even as close as a base temperature of 4 K of liquid helium.

In NRIXS experiments, nano-second time-resolved APDs at ambient temperatures are used to collect data. A vacuum shroud is designed and constructed to achieve high vacuum and maintain low temperature on the sample inside the chamber while accommodating two APDs outside the chamber at ambient temperature (shown in Fig. 3). APDs are placed at 90° from the SR beam to collect the NRIXS signals. The shroud is designed in such a way that the distance from the APD to the sample is minimized. In this system, the distance from APD detectors to the sample is about 12 mm, which is comparable to the shortest distance for a regular panoramic DAC at ambient temperature. During the measurement, the third APD is placed in the forward direction of the X-ray beam behind the sample, which collects synchrotron Mössbauer signals. Four windows are built on the vacuum shroud. The first window located at the upstream allowing the incident X-rays beam through is made of diamond with a thickness of 50 μm. The second one is a sapphire window with a thickness of 50 μm located at the downstream of the sample. It permits the synchrotron Mössbauer signals to reach the APD in the forward direction. An on-line ruby system or a Raman system can be moved when necessary in the forward direction to measure the sample pressure. The sapphire window allows laser and ruby fluorescence or Raman signal go through to determine the sample pressure by either the ruby fluorescence method,[22,23] or diamond Raman edge methods.[24]

Two square-shaped kapton windows (with a thickness of 25 μm and a size of 10 mm × 10 mm) located on the top and bottom of the shroud allow nuclear fluorescence signals reach the two sides of APDs (Fig. 3). The vacuum of the whole system is maintained near 10^{-5} Torr during the measurement.

IV. PERFORMANCE OF THE SYSTEM

A. Low temperature control and stability

The mini-pDAC is held by a round copper holder. In our new cryostat system as shown in Fig. 2, the liquid helium coolant flows through the copper holder. Two heat shield plates made of copper are used around the holder. Two openings on these plates around the sample area with a size of 10 mm × 10 mm are used to get nuclear fluorescence signals emitted from the sample out to reach the detector. The lowest temperature achieved on the sample is 9.0 K, while the temperature on the copper holder reaches 4.2 K. Three temperature sensors are used in the system to monitor the temperatures on the cold finger, the DAC holder, and the DAC, respectively. Two heaters are used: one attached to the DAC and the other one attached to the cold finger. The two heaters can be adjusted individually to achieve uniform temperature on the sample. Once the temperature is stabilized, the temperature is stable within ±0.05 K. At a helium flow rate of 1.5 ~ 2.0 l/h, it takes about 1.5 h to cool the system from room temperature to the base temperature of 9.0 K. During experiments, temperature can be adjusted easily and conveniently by a Lakeshore 336 temperature controller. The temperature can be stabilized within 20 min with proper PID control, which makes it practical and convenient to use for fast-paced synchrotron experiments.

B. Remote pressure control

Due to its low freezing temperature, helium gas (ultrahigh purity) has been used to inflate the membrane. The membrane gas pressure is applied via a stainless-steel capillary line connected to a standard gas cylinder with the highest pressure of 2200 psi. The pressure in the membrane is adjusted and maintained precisely through a modular pressure controller (Druck PACE5000 by GE) located outside the experimental hutch.

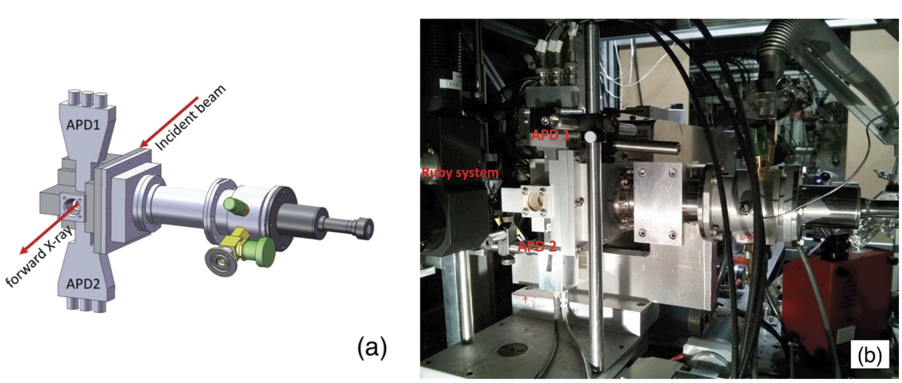

FIG. 3. (a) A schematic of the cryostat vacuum shroud with two APDs located outside the windows for NRIXS data collection. (b) Picture of the HPLT setup for NRIXS experiment at 3-ID-B, APS.

The pressure in a gas-membrane-controlled DAC can be estimated using the following formula:

$$P = \eta \cdot \frac{S1}{S2} \cdot p. \quad (1)$$

Here, P is the pressure of the sample in a DAC, $S1$ is the effective area of the double-diaphragm membrane chamber, $S2$ is the area of the diamond culet, p is the gas pressure in the membrane, and η is the coefficient of the system related to factors such as properties of the diaphragm, engagement, and initial deformation of the membrane, friction of the DAC, diamond shape, and others.

The membrane in our system is a double-diaphragm with 50.8 mm OD and 23.9 mm ID.[20] A total area is around 1580 mm^2 and the effective area is about 50%-80% depending on the membrane conditions. For commercially available gas cylinders, the maximum pressure p is about 2200 psi (150 bars). In our testing, the value of η is found to be around 10%-20%. One can estimate that about 2 Mbar is achievable in our system when using a diamond with a culet size of 0.1 mm.

Figure 4 shows test results of pressure on the sample versus the gas membrane pressure. The results were from two mini-pDACs with an anvil size of 100 μm and 500 μm. The pressures were applied when the DAC temperature was around 100-120 K. In the DAC with anvils of 100 μm culet size, no pressure medium was used, while in DACs with anvils of 500 μm culet, Ne was loaded as pressure medium. Pressures were determined from the ruby fluorescence[22,23] or diamond Raman edge method.[24] In the DAC with anvils of 100 μm culet, a membrane pressure of 470 psi drives the sample pressure to 87 GPa, while in the DAC with anvils of 500 μm culet, 940 psi membrane pressure is needed to achieve sample pressures of about 40 GPa. These sample pressures along with the moderate membrane pressures show that the pressure application from the membrane system is satisfactorily efficient and above-Mbar pressure in our system is achievable. In fact, during a recent SMS experiment on ^{161}Dy measurement, the pressures on the sample reached 1.4 Mbar through the membrane

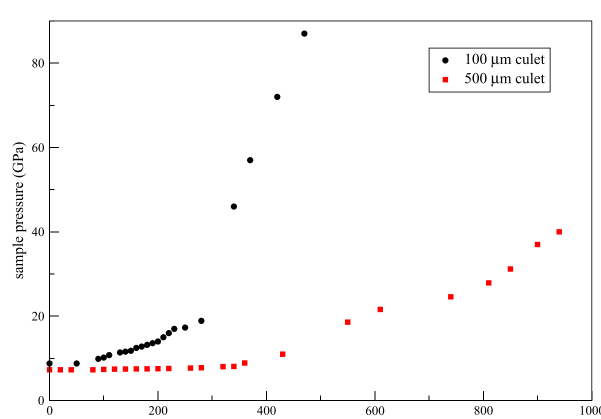

FIG. 4. Testing results of the pressure reached on the sample versus the membrane pressure in mini-pDACs. The anvil size is 100 μm and 500 μm, respectively. Sample pressures were altered when the sample temperature was around 120 K.

at 650 psi gas pressure using diamond anvils of 100 μm culet size.[30]

During the testing and regular use of the system, it was found that sample pressure could either be slightly increased or decreased when the system was cooled down to the base temperature. It happened for both Re and Be gaskets and Ne pressure medium. The cooling rate had an impact on the pressure changes. The amount of pressure change also depends on the culet size. After pressure was applied at ∼100-120 K, when the system is cooled down to ∼9 K at a cooling rate of 3 K/min, the pressure change is typically about ∼1 GPa at 10 GPa and ∼2 GPa at 20 GPa in our test.

V. NRIXS EXPERIMENT AT HPLT

The HPLT cryostat and mini-pDAC have been used by many experiments since its successful commissioning. Figure 5 shows an example of NRIXS measurement in a ^{57}Fe-enriched single crystalline $EuFe_2As_2$ sample at the resonant energy of ^{57}Fe (14.4125 keV). $EuFe_2As_2$ shows rich phase diagram with applying pressure.[25,26] In a narrow pressure range of 2.4-3.0 GPa, $EuFe_2As_2$ was found to be superconducting with T_C ∼30 K. ^{57}Fe NRIXS data on a polycrystalline $Eu^{57}Fe_2As_2$ sample at high pressure and room temperature have been reported by Kobayashi *et al.*[27] To study the lattice dynamics in the superconducting phase, it is critical to have *in situ* fine pressure tuning capability to reach the superconducting pressure regime and a cryogenic system to reach well below the superconducting critical temperature.

In this experiment, diamond anvils with 500 μm culet size were used to achieve high pressure in the mini-pDAC. To allow the nuclear fluorescence radiation to exit the sample and reach the APDs, ultra-high pure beryllium was used as a gasket material due to its low x-ray absorption. Neon gas was used as a pressure medium to achieve a hydrostatic pressure environment around the sample. Several ruby balls were loaded next to the sample to measure pressure *in situ*. An x-ray beam at 14.4125 keV was focused to ∼15 μm at the sample position. The incoming x-ray beam is along the c-axis of the single-crystalline sample. Two APDs were used to collect NRIXS spectra. Instrumental resolution function was recorded by an APD in the forward direction, and it was used to remove the elastic peak in the data reduction.

Figure 5 shows the experimental spectra of $EuFe_2As_2$ at 2.5 GPa, 3.2 GPa and at base temperature of 9.0 K. The Instrumental resolution function (FWHM = 1.05 meV), and the derived Fe partial phonon density of states (PDOS) are also shown in Figure 5. The data collecting time is typically around 6 to 8 h for a good NRIXS spectrum. The PDOS was extracted from the data using the *PHOENIX* package.[28] The Fe PDOS is in good agreement with the previous results.[19,27] The derived Lamb-Mössbauer factor are 0.891(3) and 0.887(2). Theoretical calculations are in progress to understand the changes of PDOS with pressure and its correlation with magnetic and structural transitions. More detailed discussion about the experiment results will be published elsewhere.[29] Nevertheless, these figures show the fine-tuning capabilities of sample pressures in our system at base temperature (9.0 K).

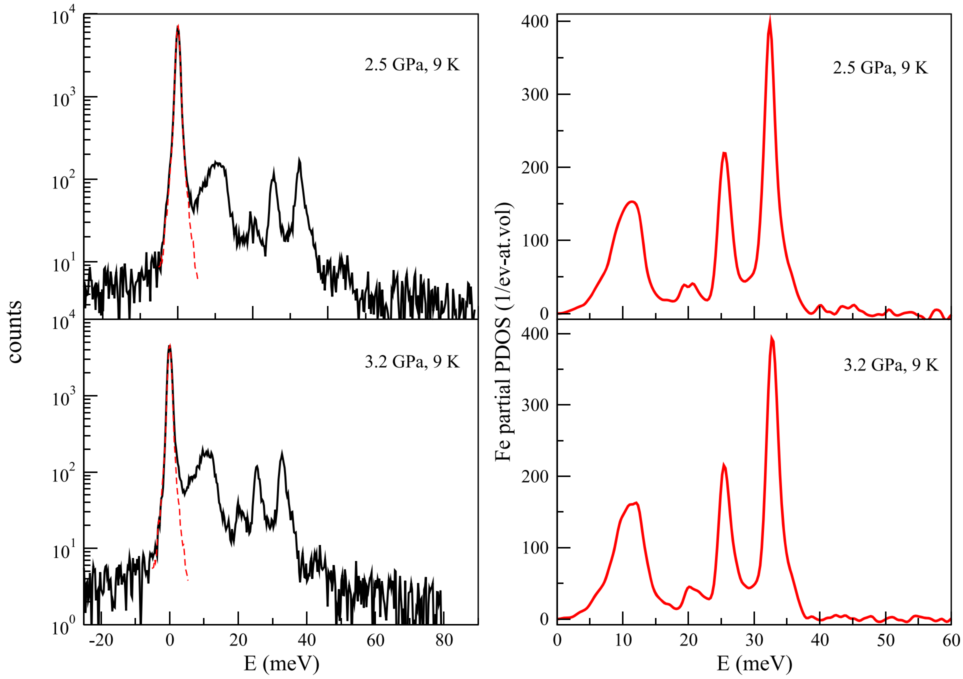

FIG. 5. (Left) NRIXS spectra (solid lines) of $Eu^{57}Fe_2As_2$ at 2.5 and 3.2 GPa and 9.0 K and the instrumental resolution function (red dashed lines) collected by the forward APD. The intensity of the resolution function is scaled to the elastic peak intensity. (Right) Derived Fe partial phonon density of states.

VI. CONCLUSION

We have designed and constructed a compact and efficient gas membrane-driven mini-pDAC and a compatible cryostat for NRIXS application in high pressure and low temperature conditions. The integrated instrument allows NRIXS and SMS measurement under the same P-T conditions with *in situ* pressure adjustability. The membrane system and the mini-pDAC have been proven to be efficient and stable in achieving multi-Mbar pressures with standard gas cylinders. Benefiting from the direct flow of liquid helium around the DAC holder, the sample was able to reach temperatures as low as 9 K. The new setup can be used for NRIXS as well as SMS experiments in various isotopes available at synchrotron sources. The performance of the setup has been tested via NRIXS experiment on $Eu^{57}Fe_2As_2$. The instrument is readily applicable to other spectroscopic experiments at HPLT conditions which requires large solid angle in the geometry, such as x-ray Raman scattering, x-ray emission spectroscopy, X-ray radial diffraction, and resonant inelastic x-ray scattering. Further improvements are possible to achieve lower temperature than 9 K if an additional vacuum jacket is added to the cryostat with APDs located inside the vacuum jacket, therefore without scarifying the sample to detector distance.

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