Analysis and Tests of Hybrid Start-up Process of a Space Dilution Refrigeration Unit

Zijie Pan[1], Lingjiao Wei[1*], Maowen Zheng[1*], Jingtao Liang[1,2], Shubao Zhao[1,2], Ziyao Liu[1], Jiarun Zou[1,2], Haowen Guo[1,2], Houlei Chen[1]

[1] Key Laboratory of Cryogenic Science and Technology, Technical Institute of Physics and Chemistry, Chinese Academy of Science, Beijing 100190, China
[2] University of Chinese Academy of Sciences, Beijing, China

[*]E-mail: weilingjiao@mail.ipc.ac.cn; zhengmaowen@mail.ipc.ac.cn

Abstract. Space dilution refrigerators for space applications have evolved over the past thirty years. The microgravity condition makes the helium isotope fluid flow and mix difficult in space. The surface tension of capillaries replaces gravity in the space dilution refrigerators, resulting in a different operating process compared with that of the ground systems. The start-up process of the space dilution refrigeration unit with a high pre-cooling temperature is carefully analyzed and tested. The dilution unit starts successfully and obtains 162 mK at a flow rate of 4 mL/min ^{3}He and 50 mL/min ^{4}He. The operating conditions and the phase interface location are carefully discussed for the performance and optimization based on the experimental results.

1. Introduction

The dilution refrigerator which is a mature extremely low temperature refrigeration equipment has been widely used in quantum computing, physical property measurement and other advanced technological fields. The space dilution refrigeration technology which is relatively challenging has been small-scale studied over the past three decades. The space dilution refrigerators (SDR) must rely on other forces instead of gravity to fix the interface between the concentrated and diluted phase, as well as the gas-liquid phase [1]. In 1988, Alain Benoit et al. developed an open-cycle dilution refrigerator (OCDR) that utilized surface tension in capillaries to fix the interface in the mixing chamber [2]. The system used the cosmic vacuum instead of mechanical pumps to avoid the problem of fixing the gas-liquid interface in the still. After a series of improvements, the OCDR capable of 0.1 μW at 100 mK was successfully applied in the Planck mission in 2009 [3]. In the same year, Florian Martin proposed a closed cycle dilution refrigerator (CCDR) with a helium isotope separation device based on the OCDR [4]. The device can fix the liquid ^{3}He in a porous still and extract the ^{4}He with a fountain pump. In 2014, Angela Volpe conducted anti-gravity experiments on the inverted dilution unit and still, promoting the space application of dilution refrigerators [5]. The CCDR has been the back-up for the Sub-Kelvin stage of Athena, which is expected to be launched in 2035 [6]. Multiple space X-ray missions in China are under preparation including HUBS and DIXE [7]. The demand for the Sub-Kelvin space refrigeration technology becomes urgent. Compared to the magnetic refrigerator, the SDR with the advantages of continuous cooling power, zero magnetic interference and light weight has application prospects for space detection missions in the future.

Compared to the conventional dilution refrigeration (CDR), researches and experimental results on the SDR are much fewer, especially regarding the start-up mechanism and the concentration effects. The CDR starts by reducing vapor pressure to directly reduce the temperature of the mixture to below 870 mK and achieve the phase separation. However, due to the presence of capillary flow resistance, the evaporation and heat transfer capabilities of the SDR are limited. Thus, the start-up method of the CDR cannot apply to the SDR. Moreover, only high-purity ^{3}He circulates in the stable fluid cycle in CDR, while both ^{3}He and ^{4}He flow in the cycle of SDR. The adjustment of the circulation concentration in SDR has a significant impact on the refrigeration performance and stability. Therefore, there are some uncertainties in the start-up process and the steady operation of the space dilution unit. A space dilution unit is designed and tested by an extremely low temperature platform. The hybrid start-up mechanism of the space dilution unit is explored and the influence of different circulation concentrations is analyzed to guide the design and optimization of future space dilution refrigeration prototypes.

2. The principles of the space dilution refrigeration

2.1 The space dilution unit

The space dilution unit mainly consists of a series of capillary counter-flow heat exchangers (HEX) and a three-way mixing chamber (MC), as shown in Figure 1. The pre-cooled ^{3}He and ^{4}He are respectively injected into the dilution unit at constant flow rates and finally mixed in the MC. The concentration of ^{3}He decreases with the increasing temperature under the influence of the osmotic pressure, which could cause counter diffusion of ^{3}He in the return pipe. To avoid the counter diffusion of ^{3}He, the ^{3}He is injected in excess and not completely dissolved in the ^{4}He in the MC. Under the influence of the surface tension, the excess ^{3}He produces 'droplets' in the return pipe [8]. The 'droplets' flow alternately with the diluted phase to lock the ^{3}He. The 'droplets' dissolve as the temperature increases and the frictional force between ^{3}He and ^{4}He is the mean of suppressing counter diffusion. The pipeline is set as an extractor by accelerating the ^{4}He speed higher than the supercritical speed in a very small inner diameter capillary. The superfluid ^{4}He transforms into a viscous normal state to lock and extract the ^{3}He. The return pipelines with 'droplets' are called the two-phase section, while those without 'droplets' are the single-phase section or the extractor.

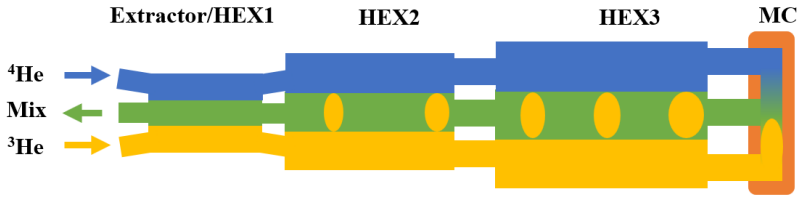

Figure 1. Schematic diagram of a space dilution unit

The space dilution unit is designed based on the laboratory model [9]. Figure 2 displays the image of the space dilution unit. The first stage is a 1 m long stainless-steel capillary extractor with an inner diameter of 40 μm, and the second and third stage are a 3 m CuNi capillary pipe with inner diameters of 200 μm and 400 μm respectively. To overcome the Kapitza thermal resistance and extract cooling capacity, the copper mounts are installed after the MC to provide sufficient heat exchange area. The return capillary is wrapped around the mounts with a 6 m length to provide a heat exchange area of 75 cm^2.

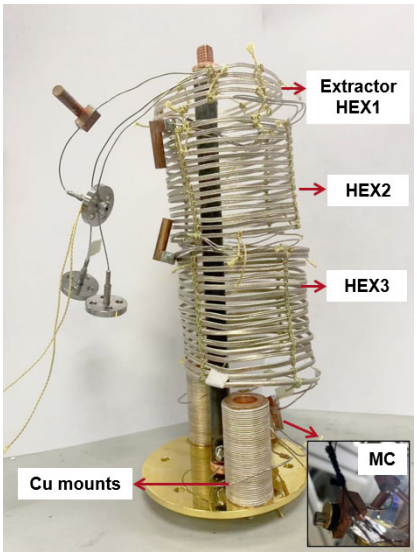

Figure 2. Image of the space dilution unit

2.2 Hybrid start-up process

The key of the start-up process of the dilution refrigerator is to achieve the phase separation temperature in the MC. The start-up method of CDR is reducing the pressure and evaporating the mixture. It is enough to create a phase interface in the MC and complete the start-up. The start-up process of SDR is more difficult and complex. The flow resistance of capillaries limits the performance of evaporation and heat transfer and the adjustment of the circulation concentration affects the start-up process. In addition to the evaporation start-up scheme, the system adopts a high-temperature dilution refrigeration scheme, as described in Martin's doctoral thesis [10]. In fact, the dilution process can be divided into two types based on the ^{4}He chemical potential μ_4: direct dilution process ($\Delta\mu \neq 0$) and osmotic dilution process ($\Delta\mu = 0$). The refrigeration capacity of the direct dilution depends on the excess enthalpy H^E with a temperature limit of only 180 mK. The cooling temperature of the osmotic dilution can theoretically reach 0 K, depending on the osmotic enthalpy H^{os}. In the space system, the pure ^{3}He diffuses back up the ^{4}He pipe effectively acting as a superleak and keeping osmotic dilution [11], which occurs under the phase separation temperature. The hybrid start-up process combines the two types and takes the direct dilution as the pre-cooling for the osmotic dilution.

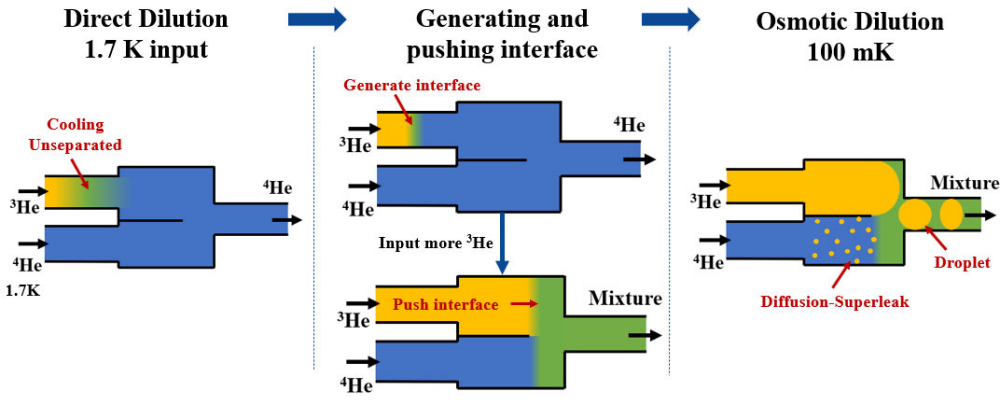

Figure 3. The theoretical predictions of the hybrid start-up process

The theoretical predictions for the hybrid start-up process are conducted, which can be

divided into the following three steps. Firstly, a direct dilution process occurs without the phase separation when ^{3}He and ^{4}He are input at 1.7 K. Secondly, a stable phase interface begins to form when the temperature drops to the phase separation temperature. After continuing to input ^{3}He, the concentrated phase pushes the phase interface all the way to the MC. Finally, part of ^{3}He 'droplets' diffuses into the ^{4}He pipe, forming a superleak and beginning the osmotic dilution process. The temperature decreases to 100 mK or lower. The diagram of the theoretical prediction is shown in Figure 3. Next, the start-up experiments will be conducted to verify the theoretical predictions.

3. The start-up experiments

The evaporation cooling and the hybrid dilution refrigeration around 1 K are tested as the start-up methods of the space dilution unit. The former mainly relies on the latent heat of ^{3}He, while the latter relies on the ^{3}He enthalpy difference before and after mixing.

3.1 Evaporation experiment

Figure 4 presents the experimental results of the ^{3}He evaporation cooling method. When the still reaches 0.7 K, the MC remains at 1.5 K because the single-phase capillary structure restricts the heat transfer between the two devices. Therefore, the evaporation method is not suitable for the space dilution units indeed.

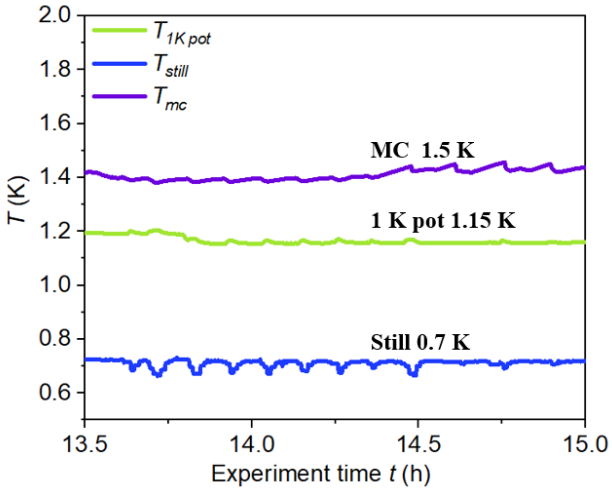

Figure 4. The ^{3}He evaporation cooling process

3.2 Hybrid start-up experiments

The experimental results of the hybrid dilution refrigeration around 1 K are shown as follows. With 10 L ^{3}He and 50 L ^{4}He prepared, an open cycle dilution refrigeration experiment is conducted to verify the start-up mechanism of the SDR.

Figure 5 shows the hybrid start-up process and the cooling curve of the space dilution unit. The ^{3}He and ^{4}He are injected at a ^{3}He concentration of 17% at 1.2 K. In the stage ①, ^{3}He and ^{4}He meet in the middle of the two-phase pipe, where the direct dilution process begins with a temperature drop. In the stage ②, as direct dilution refrigeration continues, the temperature decreases and reaches 590 mK. At this point, the local concentration is higher than the saturation solubility, resulting in a stable phase interface. In the stage ③, when more ^{3}He is input, the concentrated phase begins to push the phase interface to the MC, where the temperature drops sharply to 480 mK. In the stage ④, the temperature of the MC keeps decreasing and approaches 178 mK, which is lower than the refrigeration limit of direct dilution at 180 mK. This indicates that

the droplet superleak takes effect and the direct dilution process changes to the osmotic dilution process. The space dilution refrigeration process starts successfully from a pre-cooling temperature of 1.2 K, which verifies the hybrid start-up mechanism. The four stages of the hybrid start-up experiment are highly consistent with the theoretical predictions.

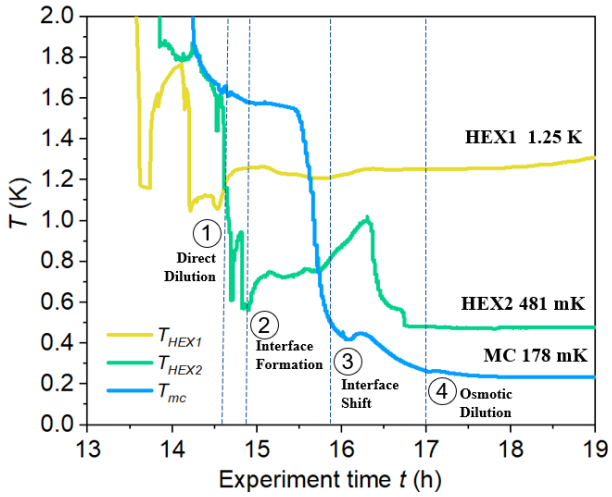

Figure 5. The hybrid start-up process of the space dilution unit

3.3 Circulation concentration experiments

The circulation concentration of ^{3}He is one of the key parameters for the operation. To avoid the counter diffusion of ^{3}He, it must be excessively injected. The excess ^{3}He generates 'droplets' that fill the pipe after mixing and maintain the two-phase interface. The excess ^{3}He does not participate in the dilution process and inevitably becomes an ineffective heat load. Figure 6 points out the experimental results of different circulation concentrations. When the circulation concentration drops from 17% to 7%, the T_{mc} continues to decrease with the start-up completed. However, when the circulation concentration drops below 5%, the T_{mc} begins to rise. Even a sudden increase occurs at 3.8%, which causes damage to the phase interface.

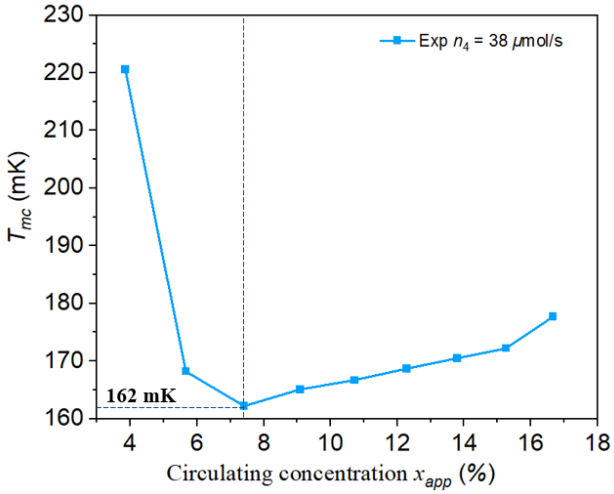

Figure 6. Experimental results of different circulation concentrations

The performance of the dilution refrigeration is closely related to the circulation concentration of ^{3}He. When ^{3}He is not excessive, the performance is greatly weakened. Reducing the ^{3}He concentration appropriately without damaging the phase interface is beneficial for obtaining a lower cooling temperature. The supercritical speed and viscous heat inside the

extractor, the heat transfer area and the capillary force of the HEX all impose different requirements for the diameter and length of the capillary pipes. Subsequent experiments will be conducted with different combinations of the single-phase and two-phase pipes to explore the optimal selection between the structure and the flow rate.

4. Conclusion

A dilution refrigeration unit is designed for space applications in our laboratory. The dilution unit successfully starts from the pre-cooling temperature of 1.2 K, verifying the hybrid start-up mechanism. The ^{3}He and ^{4}He are directly mixed above 1 K and a direct dilution process occurs. The temperature of the MC drops to below the phase separation temperature and ultimately reaches 162 mK by the osmotic dilution. The results of the hybrid start-up experiment are highly consistent with the theoretical predictions. The damage to the phase interface caused by insufficient ^{3}He proves evident in the experiment. The results guide the design and optimization of the space dilution refrigeration prototype. In the future, the space dilution unit will be coupled with the JT refrigerator and the sorption cooler to provide technical foundations for space projects [12-14].

Acknowledgements

Supported by the Key Research Program of Key Laboratory of Cryogenic Science and Technology; the Scientific Instrument Developing Project of the Chinese Academy of Sciences, Grant No. ZDKYYQ20220004; the Director Fund of Technical Institute of Physical and Chemical, Chinese Academy of Sciences.

References

[1] Israelsson U E, Petrac D. Dilution refrigeration for space applications. Cryogenics, 1990, **30**(3): 257-262.
[2] A Benoît, Bradshaw T, Orlowska A, et al. A Long Life Refrigerator for 0.1K Cooling in Space. SAE Technical Paper, 941276, 1994.
[3] Ade P, Aghanim N, Arnaud M, et al. Planck early results. II. The thermal performance of Planck. Astronomy & Astrophysics, 2011, **536**.
[4] Martin F, Vermeulen G, Camus P, et al. A closed cycle ^{3}He–^{4}He dilution refrigerator insensitive to gravity. Cryogenics, 2010, **50**(9): 623-627.
[5] Volpe A. Development of a closed cycle dilution refrigerator for astrophysical experiments in space. Université Joseph-Fourier - Grenoble I, 2014.
[6] Tirolien T, et al. Spacecraft Cryogenic Developments and Perspectives at the European Space Agency. International Cryocooler Conference, 2020,377-386.
[7] Cui W, Chen LB, Gao B, et al. HUBS: Hot Universe Baryon Surveyor. J Low Temp Phys 2020, **199**, 502–509.
[8] Paragina A . Etude de la refrigeration par dilution de l'helium en cycle ouvert. Application a un satellite d'observation submilimetrique. 1997.
[9] Pan Z, Zheng M, Wei L, et al. Theoretical analysis of the heat exchanger of a dilution refrigerator for space applications. Cryogenics, 2023, **130**.
[10] Florian Martin. Développement de la réfrigération à dilution ^{3}He-^{4}He en cycle fermé pour les futures missions scientifiques en satellite. Physique. Université Joseph-Fourier - Grenoble I, 2009.
[11] Chaudhry, G., Vermeulen, G. Analysis of a Mixing Chamber Developed for Use in a Gravity-Independent Dilution Refrigerator. J Low Temp Phys, 2012, 90–110.
[12] Liu Z, Pan Z, Ma Y, et al. A hybrid ^{3}He Joule-Thomson cryocooler designed for precooling the space dilution refrigerator. Cryogenics, 2023, **131**.
[13] Lei Y, Hong G, Zhao Y, et al. Analysis and test of ^{4}He gas-gap heat switch for a sub-Kelvin sorption cooler. Cryogenics, 2023, **132**.
[14] Lei Y, Hong G, Quan J, et al. Design and development of a ^{4}He sub-Kelvin sorption cooler. Cryogenics, 2024, **141**.