M2PoB-01: A cryogenic tensile testing apparatus for micro-samples cooled by miniature pulse tube cryocooler

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1. Introduction

➢Tensile tests are widely applied to measure the mechanical properties of materials. At present, most of the cryogenic tensile testing apparatus are designed for samples with standard sizes, while for non-standard size samples, especially for micro-samples, the tensile testing cannot be conducted generally.

➢The general approach to cool down the specimens for tensile tests is by using of liquid nitrogen or liquid helium, which is not convenient to a certain extent. Compared with the cryogens, cryocooler has some disadvantages in vibration and long-term stability. However, cryocooler is much easier to operate: just press the On/Off switch then the cooling begins.

➢At present, there are many kinds of cryocooler can be available at the temperature range of 20 K to the room temperature. While most of the commercially available cryocooler working in temperature below 20 K are G-M cryocooler or G-M type pulse tube cryocooler, which employ the oil-lubricated compressor to drive the cooler with high electric powers, they have an appreciable vibration and periodic maintenance is generally needed.

➢To overcome these limitations, a cryogenic tensile testing apparatus for micro-samples cooled by a miniature pulse tube cryocooler will be introduced in this paper. With the developed apparatus, samples with a diameter as small as 3 mm can be tested. Instead of employing oil-lubricated compressor, a linear dual-opposed compressor, which characterized by high efficiency, low vibration and long-time stability, has been used to generate the pressure wave to drive the pulse tube cryocooler. Figure 1 is the schematic of the pulse tube cryocooler driven by a linear dual-opposed compressor, which is the same with the cryocooler we published before.

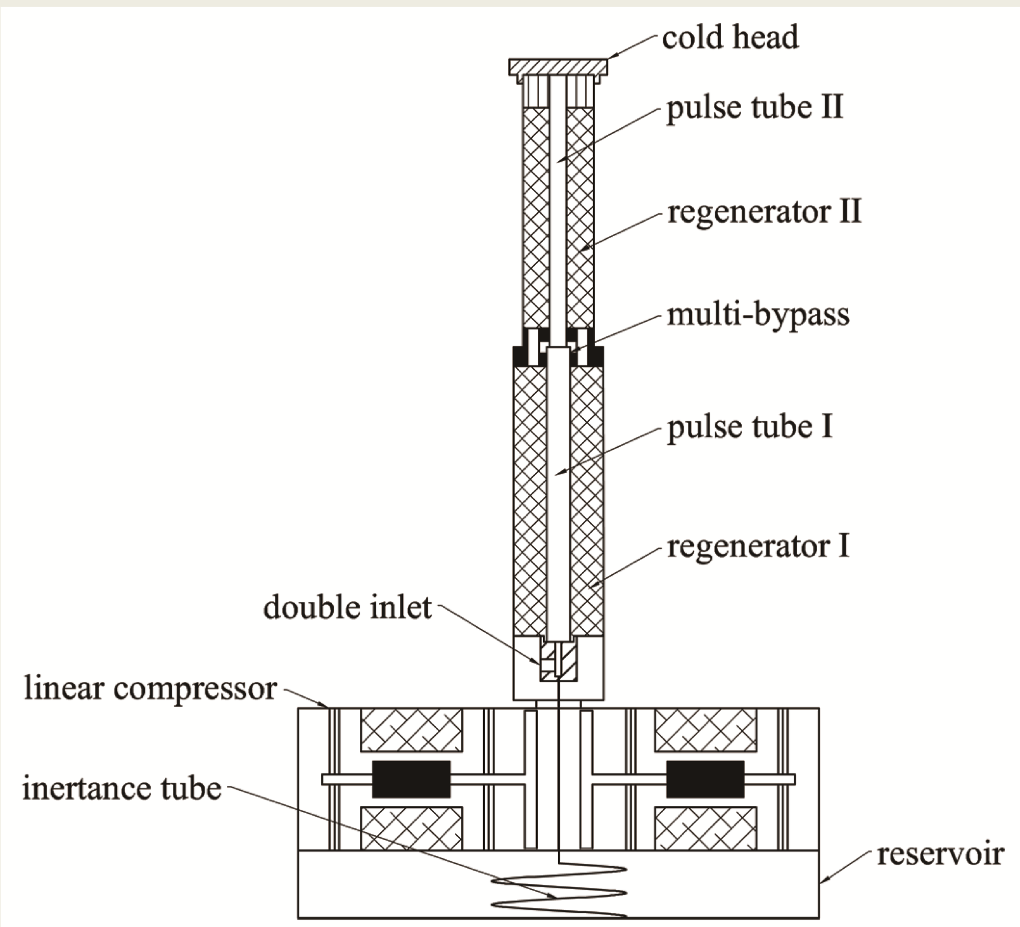

Figure 1. Schematic of the pulse tube cryocooler.

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2. Configuration design and discussion

- For the first prototype, the size of the sample is designed according to a TEM specimen: a diameter of 3 mm and a thickness of less than 500 nm.
- The left part and the right part of the fixture are exactly symmetry, and both parts contain one base and one cover plate. The sample should be mounted in the notch of the base and then be compressed by the cover plate through four M2 screws. The design of the guide rod between the left and right part is not only to guide the direction in the tensile process, but also to guarantee the centre hole of TEM sample will not be compressed.

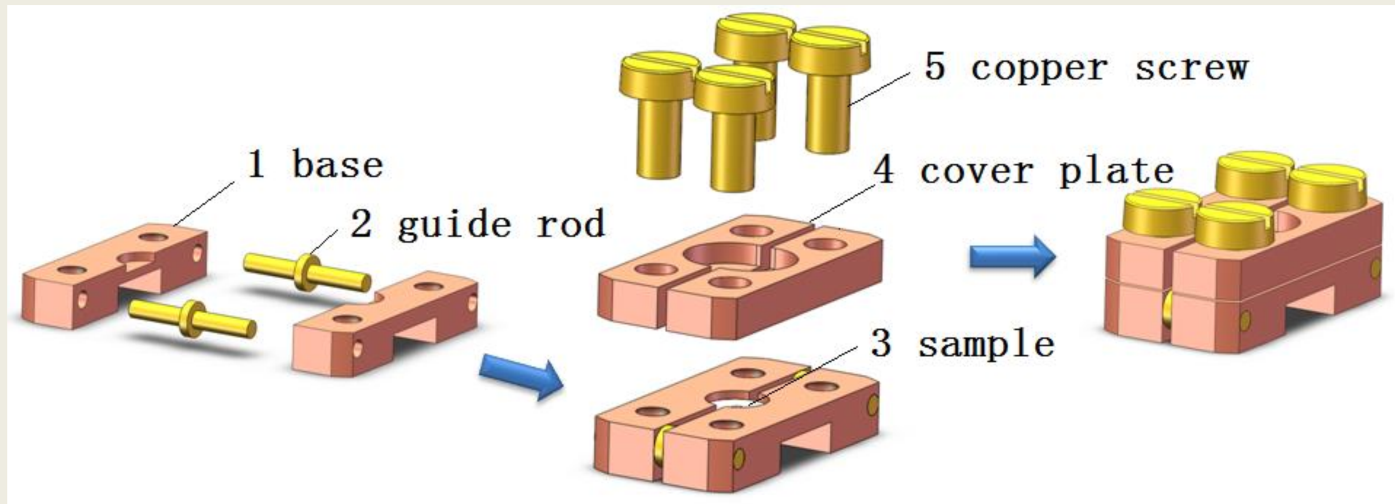

Figure 2. The install process of the sample.

- Two sample clamps has been designed to hold the sample holder firmly, and one of them (the right one in figure 3) will be fixed on the cold head tightly by a copper screw to reduce the temperature difference between the cold head and the sample holder, while the other one (the left one in figure 3) will just put on the cold head and then connected to a wire drawing.
- Although the right clamp is fixed on the cold head, it is still need to connect with the vacuum shield by a nylon rope to prevent the cooler from bending in the process of the tensile by the wire drawing

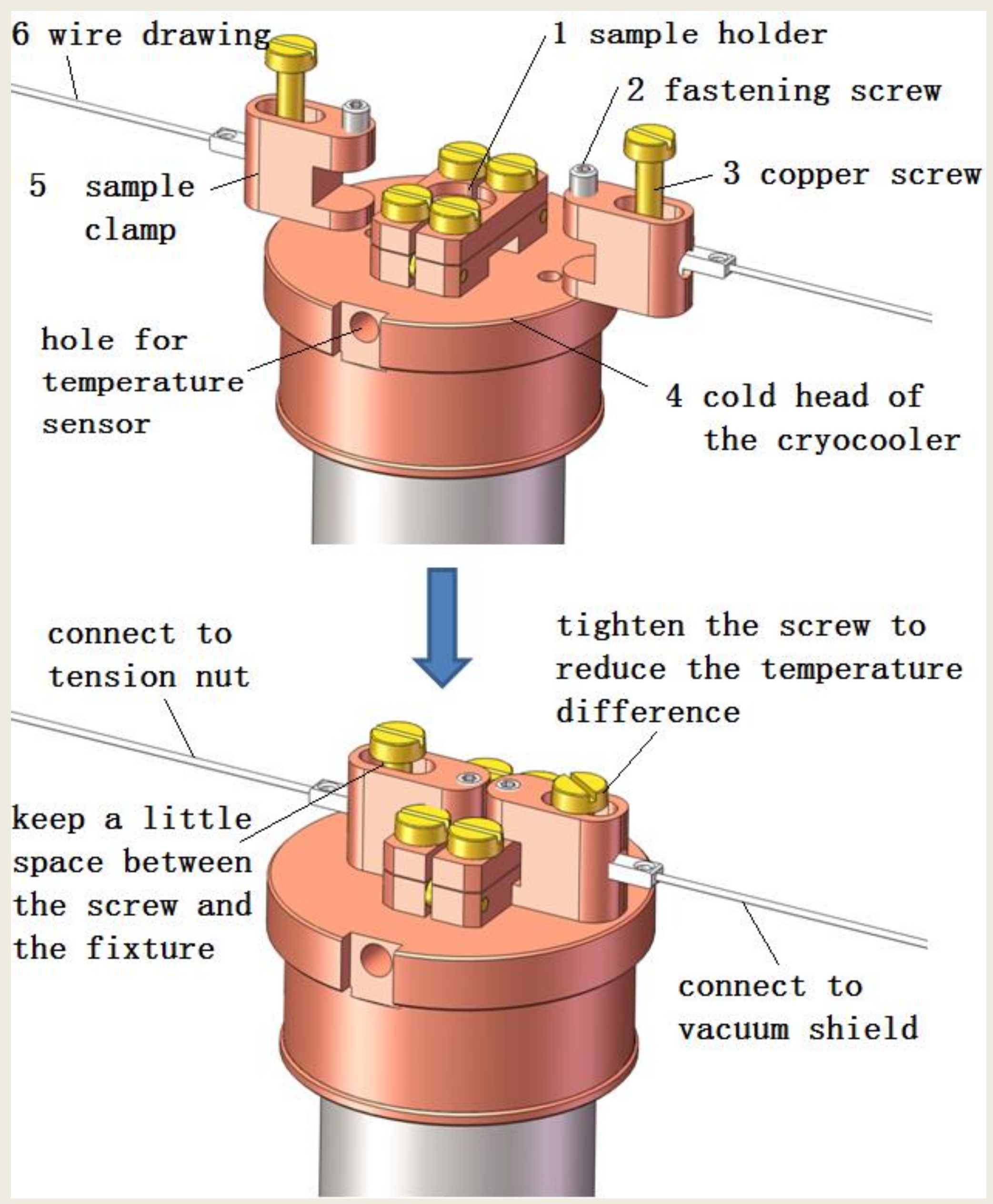

Figure 3. The install process of the sample holder.

- As shown in figure 4, there is a thermal shield connected with the cold head outside the sample holder to keep the sample temperature the same with the cold head.
- A radiation shield has also been installed between the cold head and the vacuum shield to improve the cooling performance of the cooler.
- The wire drawing connects with a tension spring before fasten with the tension nut; and there is a transparent pipe which is made of Plexiglas outside of the tension spring. The tension spring are employed as a simple indicator for estimating the tension force by its deformation degree.

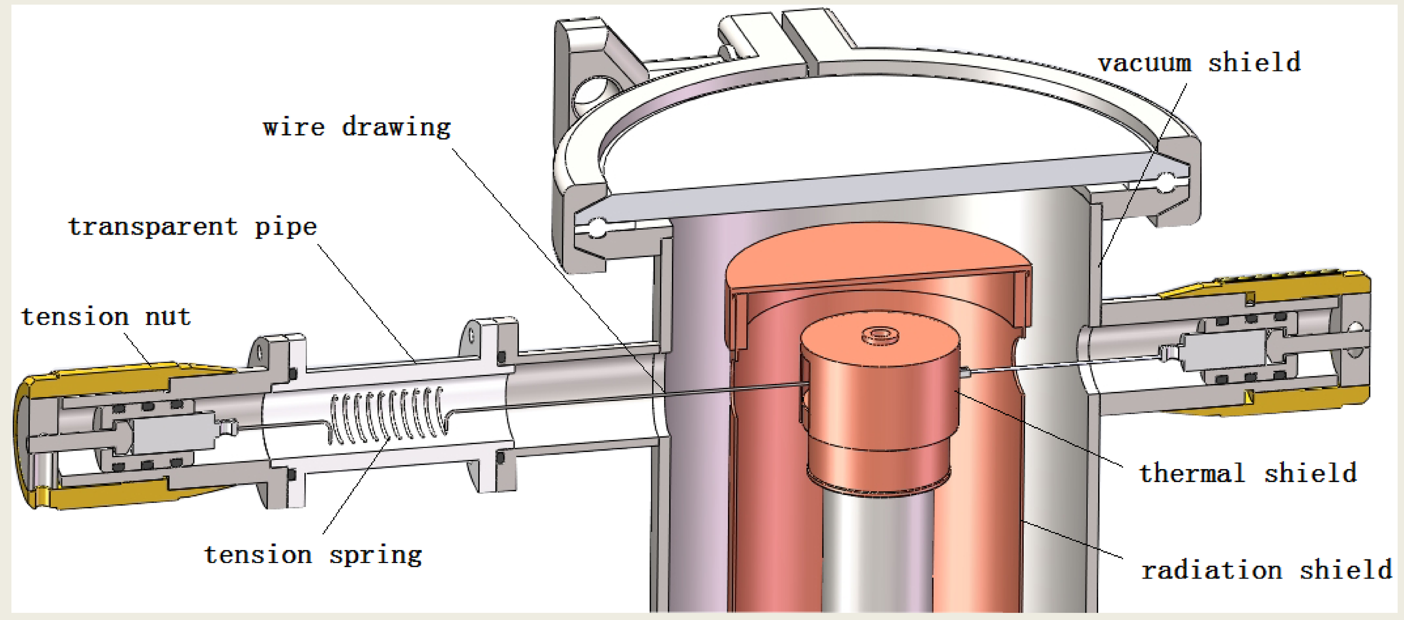

Figure 4. Cutaway views of the cryogenic tensile testing apparatus.

- The total weight of the apparatus is about 20 kg and the height is about 0.5 meter.
- The cold head of the pulse tube cryocooler is vertical up in figure 5; however, in order to achieve a lower temperature, the cold head should be flipped vertically downward. The temperature of the cold head will be 5-10 K higher when it is tipped with the pulse tube's cold end above its hot end due to natural convection for the cryocooler developed in our team.

Figure 5. Photo of the developed cryogenic tensile testing apparatus.

3. Cooling performance

- The developed cryogenic tensile testing apparatus is cooled by a single-stage high frequency pulse tube cryocooler; a lowest temperature of about 16 K can be achieved with a maximum electric power of 260 W.
- It takes about 45 minutes to reach its bottom state.

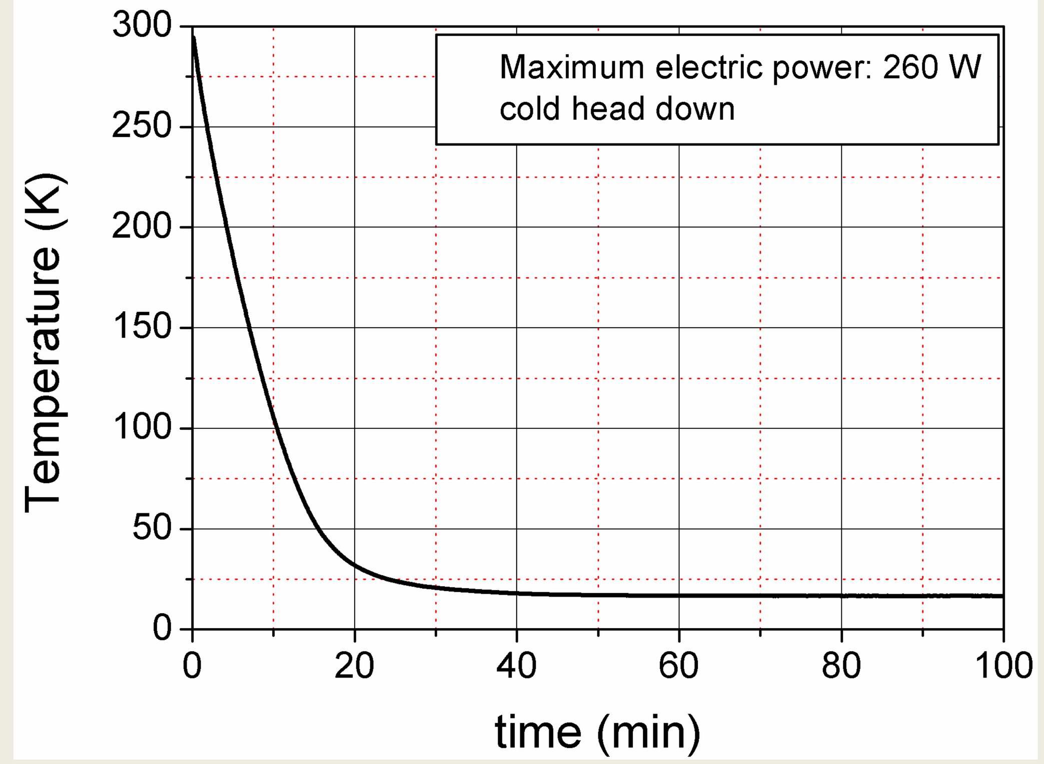

Figure 6. A typical cool-down curve

- The general temperature control method for traditional cryocooler is to control the heater power through PID adjustor control. As to this developed apparatus, the temperature is controlled by adjusting the input electric power to the compressor.
- A temperature control precision of ±10 mK can be achieved.

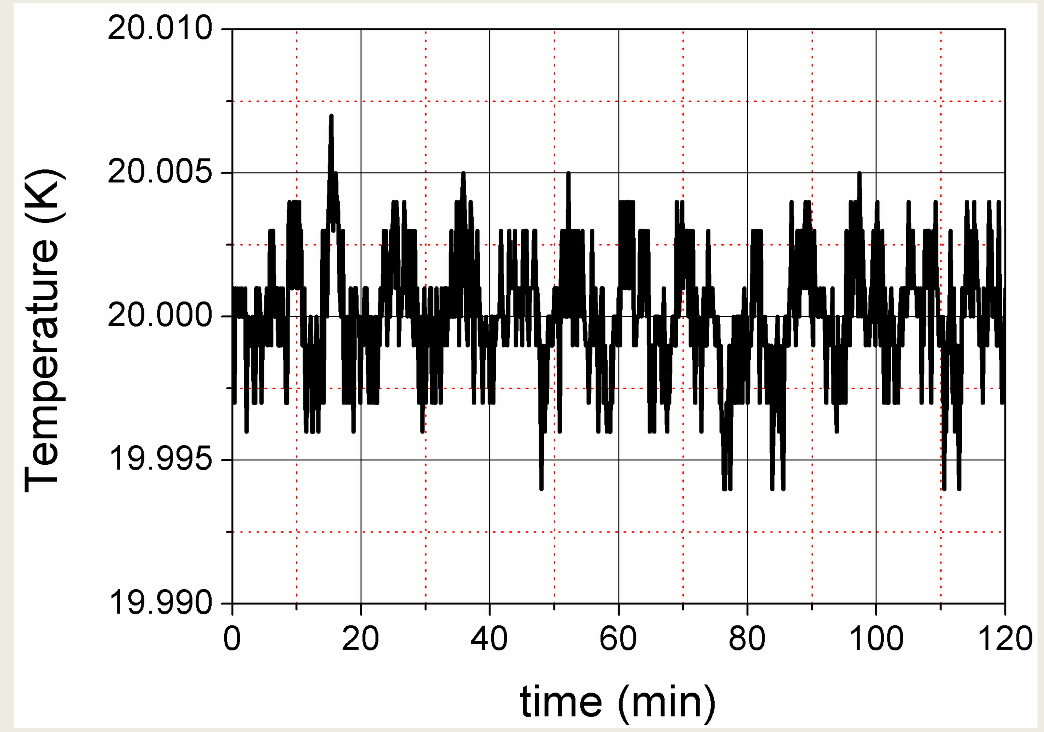

Figure 7. Temperature control curve

4. Conclusions

- A cryogenic tensile testing apparatus for micro-samples cooled by a miniature pulse tube cryocooler has been designed, built and tested. At present, a general TEM sample with a diameter of 3 mm and a thickness of less than 500 nm can be tensile tested. Kinds of samples with different sizes or shapes can also be tested by using different sample holder with the same apparatus by further optimization.
- The lowest temperature of the present apparatus is about 16 K and the temperature of the sample can be controlled at an arbitrary set point from 20 K to room temperature with a precision of ±10 mK.