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

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Abstract. This paper introduces a cryogenic tensile testing apparatus for micro-samples cooled by a miniature pulse tube cryocooler. At present, tensile tests are widely applied to measure the mechanical properties of materials; 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. The general approach to cool down the specimens for tensile testing is by using of liquid nitrogen or liquid helium, which is not convenient: it is difficult to keep the temperature of the specimens at an arbitrary set point precisely; besides, in some occasions, liquid nitrogen, especially liquid helium, is not easily available. To overcome these limitations, a cryogenic tensile testing apparatus cooled by a high frequency pulse tube cryocooler has been designed, built and tested. The operating temperatures of the developed tensile testing apparatus cover from 20 K to room temperature with a controlling precision of ±10 mK. The apparatus configurations, the methods of operation and some cooling performance will be described in this paper.

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 with an equivalent diameter of hundreds of micrometres to several millimetres or a thickness of hundreds of nanometres to several millimetres, the tensile testing cannot be conducted generally. However, sometimes, micro-samples will be used in the study of materials cryogenics properties inevitably. Two examples: the thickness of the Polyimide films for inertial confinement fusion is only hundreds of nanometres; and the sample for Transmission Electron Microscope (TEM) usually with a diameter of 3 mm and a thickness less than 500 nm. It is practically impossible to conduct tensile test by the present commercial tensile testing apparatus. As to the cryogenic tensile testing apparatus, the general approach to cool down the specimens for tensile tests is by using of liquid nitrogen or liquid helium [1-4], which is not convenient to a certain extent: firstly, it is difficult to keep the temperature of the specimens at an arbitrary set point precisely; secondly, in some occasions, liquid nitrogen, especially liquid helium, is not easily available. Compared with the cryogens, cryocooler has some disadvantages in vibration and long-term stability.

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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 available in the temperature range of 20 K to 300 K. While most of the commercially available cryocooler working below 20 K is 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. Instead of employing oil-lubricated compressor, a linear dual-opposed compressor, which is 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 [5-8].

![Figure 1. Schematic of the pulse tube cryocooler.](image)

In this paper, a TEM specimen can be tensile tested by the developed apparatus at different set temperature. The original intension of this project is to analyse the deformation mechanisms (dislocation or twins) of samples after tensioned in low temperature, rather than to focus on the tensile test data acquisition. However, it is should be mentioned that disk shaped samples is not an acceptable tension specimen geometry; disks cannot be pulled from their edges to yield suitable test data. Therefore, to yield suitable test data in keeping with best practices established in standards, the sample should be machined out by the standard method for cryogenic tensile testing [9]. But no matter disk shaped samples or the standard shaped samples, the total apparatus configurations, the measures to enhance heat transfer or reduce the thermal loss, the basic design methods of sample holder and so on, are similar, which is the purpose of the paper. In the following sections, the apparatus configurations, the methods of operation and some cooling performance will be described.

2. Designing of a fixture for tensile testing

For the first prototype, the sample with a diameter of 3 mm and a thickness less than 500 nm is designed according to a TEM specimen. As shown in figure 2, 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. It should be mentioned that 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 assembly procedure for the sample.

The sample holder will be moved to the cold head of the cooler after the sample has been fixed. Two sample clamps have been designed to hold the sample holder firmly, and one of them (the right one as shown 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 as shown in figure 3) will just put on the cold head and then connected to a wire drawing. As shown in figure 3, 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 forces of 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 cooler performance [6]. As can be seen from figure 4, the wire drawing connects with a tension spring before fastening with the tension nut; and there is a transparent pipe which is made of Plexiglas outside of the tension spring. The linear deformation of the tension spring is used to estimate the tensile forces.

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

The photo of the developed cryogenic tensile testing apparatus is shown in figure 5. The total weight of the apparatus is about 20 kg and the height is about 0.5 meter, so it is very convenient to move. 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, and a lowest temperature of about 16 K can be achieved with a maximum electric
power of 260 W. Figure 6 is the typical cool down curve, it takes about 45 minutes to reach its bottom state.

![Temperature vs. time graph with a title](image)

**Figure 6.** A typical cool-down curve of the developed apparatus.

It is difficult to keep the temperature of the specimens at an arbitrary set point precisely by the traditional LN2/LHe cooling. But to cryocooler, the temperature control can be realized easily. The general temperature control method for traditional cryo cooler is to control the heater power through PID adjustor control. As to the developed high frequency pulse tube cryocooler in our team, we control the temperature of the cold head by adjusting the input electric power to the compressor, which is an energy-efficient way [8]. Figure 7 are the temperature control curve, a temperature control precision of ±10 mK can be achieved.

![Temperature vs. time graph with a title](image)

**Figure 7.** Temperature control curve of the developed apparatus.

4. Considerations for further improvement

As has been described above, the tension force is estimated by the deformation degree of the spring for the present apparatus. However, to some users, the magnitude of the tension force and the tensile displacement are important parameters to study mechanical properties of materials. Therefore, a force sensor will be employed to substitute the tension spring to measure the forces quantitatively, and the displacement of the tension nut will be marked in the next prototype.
The present samples holder is designed according to the size of TEM samples. In fact, kinds of samples with different sizes can be tested by using different sample holder with the same apparatus. To the Transmission Electron Microscope, it is difficult to conduct cryogenic tensile testing in-site, but to Scanning Electron Microscope, it is possible to integrate a cryocooler with a small size to realize the in-site cryogenic tensile test function, which will be our next work.

5. 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.

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