Development of an in-situ analysis instrument for microstructure of materials with low temperature

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Background

- The development of instruments capable of dynamically observing the microstructure at different temperatures is very useful for the study of materials.
- The cold sources of cryogenic scientific instruments mainly include cryogens and cryocoolers.
- For cryogens, the most used is liquid nitrogen, but its temperature is relatively high (around 77 K). In order to obtain a lower cooling temperature, liquid helium (around 4 K) is required, but the cost will be significantly increased.
- For cryocoolers, the most widely used are GM cryocoolers or GM pulse tube cryocoolers, which have the advantage of achieving lower temperatures (less than 3K) and larger cooling capacity (larger than 1.5W/4.2K), but the large size and power consumption of such cryocoolers limits their application in some applications.
- High-frequency pulse tube cryocooler with smaller size and lower power consumption can also be used for cryogenic scientific instruments.

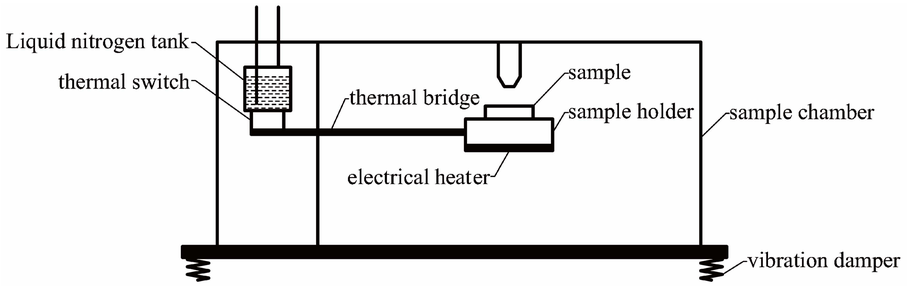
Objectives

- In this paper, based on a commercial scanning electron microscope (SEM, model SU1510), liquid nitrogen and a self-developed high-frequency pulse tube cryocooler are used as cold sources to develop in-situ observation instruments for material microstructure.
- The reason for using liquid nitrogen as a cold source before using the cryocooler is to investigate the effect of the introduction of the external cooling system on the SEM system, and explore some general methods of vibration and temperature control.

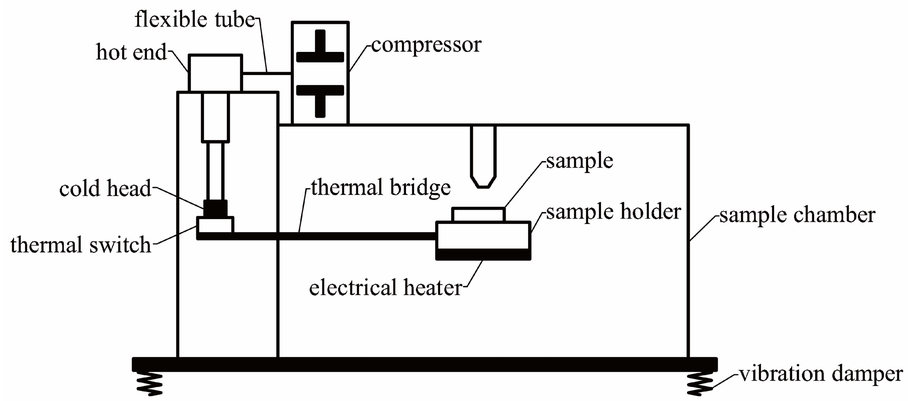
Conclusion

- Vibration reduction and temperature control are key technologies in the development of SEM cryogenic systems.
- The test results show that coupling the cryogenic system to the SEM sample cavity and sharing the existing vibration reduction device of the sample cavity does not affect the imaging quality of the SEM.
- It was found that it is necessary to fully achieve the heat balance at a certain temperature and adjust the image displacement to overcome the adverse effects caused by the thermal expansion and contraction of the low temperature components.
- When the SEM is at a high observation magnification, the cryocooler needs to use an intermittent operation mode to avoid distortion of the material microstructure image caused by vibrations of the cryocooler.
- Whether it is for liquid nitrogen or cryocooler, a thermal switch between the cold source and the thermal bridge is helpful for temperature control of the sample and reduced heat leakage of the system.
- Some new functions will be further added, for example, a deformation excitation mechanism will be added to achieve deformation excitation such as stretching and compression at different temperatures

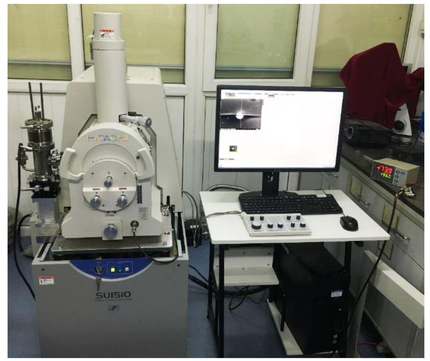
SEM cryogenic system and related technologies introduction

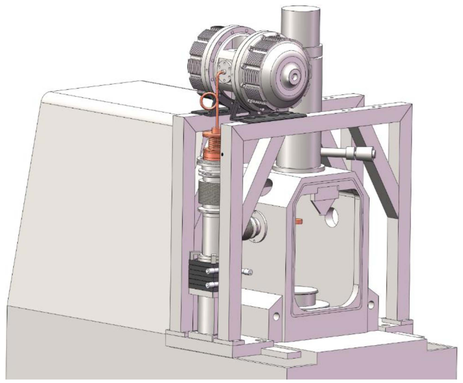


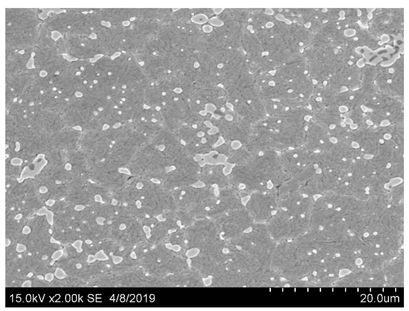
Schematic of SEM cryogenic system (cooled by liquid nitrogen)



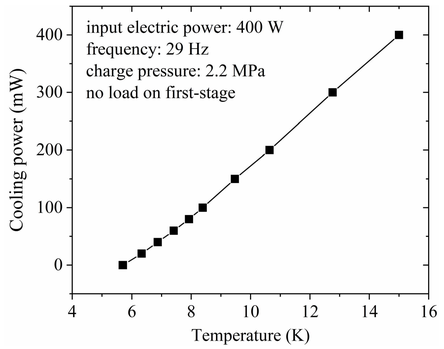
Schematic of SEM cryogenic system (cooled by high frequency pulse tube cryocooler)



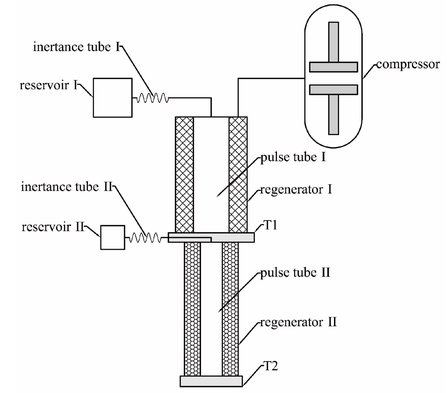




A photomicrograph of the sample holder (oxygen-free copper) without sample at 80 K.



Cooling power of the high frequency pulse tube cryocooler used.



Schematic of the developed high frequency pulse tube cryocooler for SEM.

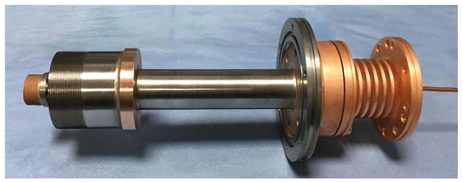


Photo of the developed high frequency pulse tube cryocooler for SEM.

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