An introduction of air-protection cryo-holder

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1. Introduction

When milled samples using focused ion-beam instruments, increases in sample temperature occur in the case of samples with low thermal conductivity rates, such as organic materials and resins. In addition, when samples are examined by transmission or scanning electron microscopy, the sample temperature sometimes increases due to the effects of the electron beam, rendering evaluation of the original morphology and condition of the sample difficult. The risk of increases in the sample temperature when materials such as thermolabile resins, low-melting-point metals, and low-temperature phase-change substances are treated and examined using charged-particle instruments is currently a major issue in the field of materials science. In addition, some materials react sensitively to water in the atmosphere, and inter-instrument transfer during stages from milled to observation is therefore another issue to which consideration must be given. In response to the above issues, a refrigerated sample-holder equipped with an air-protection mechanism has been developed. An overview of such air-protection cryo-holders, together with their advantages, and examples of applications, is introduced in the present report.

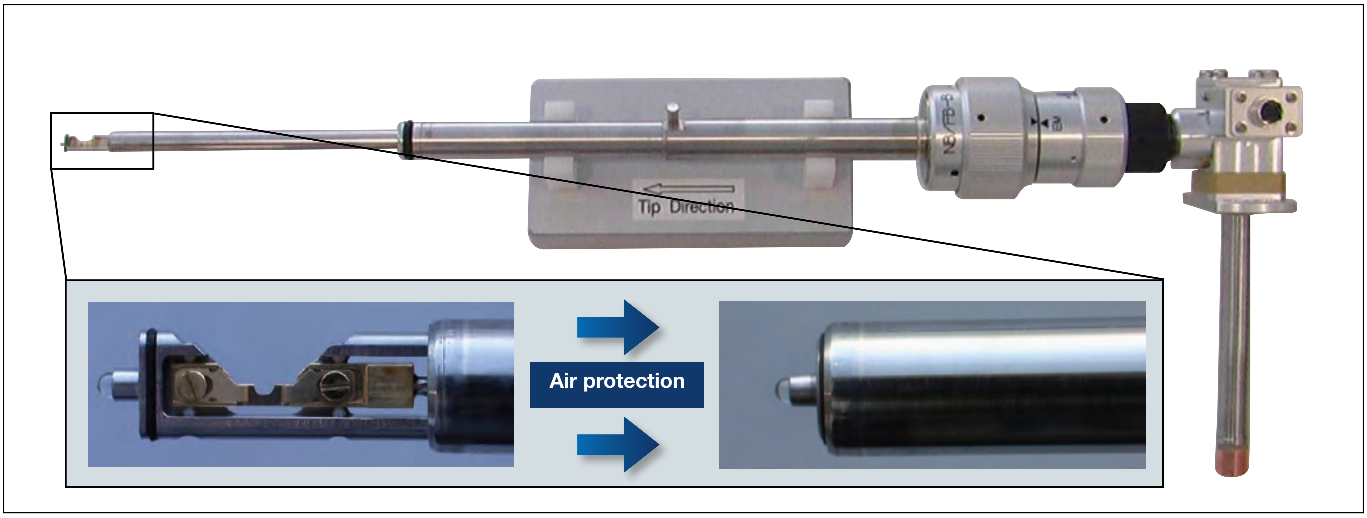


Fig. 1 Air-protection Cryo mesh holder

Fig. 2 External view of an instrument with air-protection cryo-holder equipped (@NB5000)

2. Overview of air-protection cryo-holder

There are two types of air-protection cryo-holders, of which one, the mesh type, is for micro-samples, and depends on the sample morphology; and the other is for bulk samples. The mesh type can be converted for use with all Hitachi focused ion-beam, and scanning/transmission electron microscopy instruments by replacing the adaptor. Figure 1 shows an external view of an air-protection refrigerated mesh holder with an NB5000 adaptor fitted. In addition, Fig. 3-1 shows a structural overview of the holder, and Figs. 3-2 and 3-3 show the opening-and-closing structure at the tip of the holder.

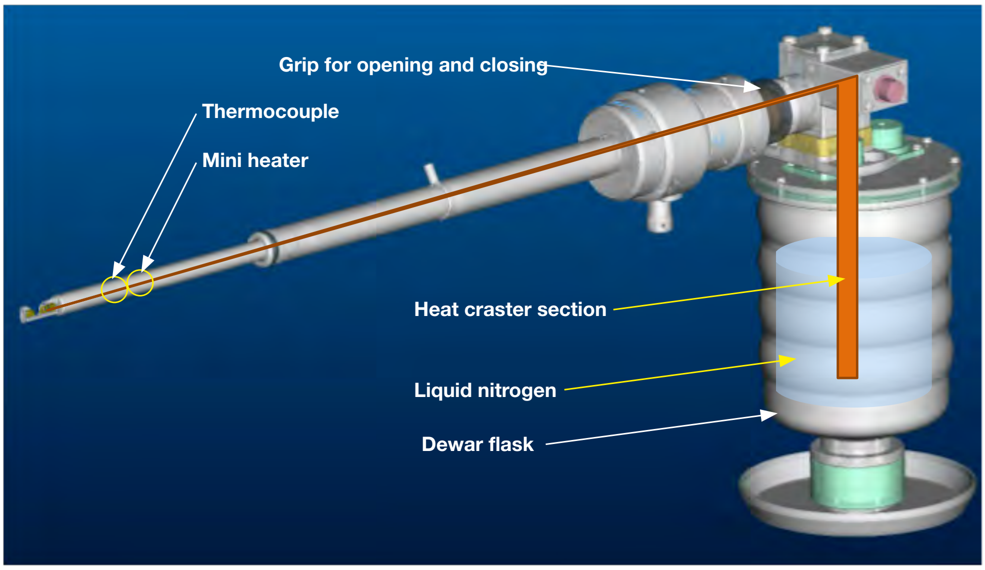


Fig. 3-1 Structural overview of the air-protection Cryo mesh holder

The coolant used is liquid nitrogen, with the samples being cooled by means of the liquid nitrogen in the Dewar flask fitted to the holder. As the Dewar flask has a vacuum heat-insulation structure, the cooling temperature is maintained, and there are no concerns about condensation. In addition, even if the stage is in a non-horizontal position, the dead weight of the Dewar flask maintains this in a vertical position, and there is therefore no risk of spillage of the liquid nitrogen inside the Dewar flask. The liquid nitrogen, which is at a temperature of -196ºC, transfers heat from the vicinity of the sample via the heat-transfer section of the holder grip, which is made of oxygen-free copper. In the vicinity of the sample, in order to prevent vibrations, heat is transferred to the mesh-attached section via a soft, braided copper wire. In addition, the instrument is equipped with a thermocouple for monitoring the temperature in the vicinity of the braided copper wire, and also a mini-heater, for temperature adjustment.

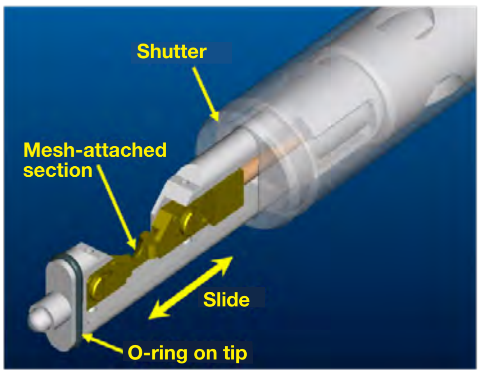


Fig. 3-2 When the tip is open, during milled and observation

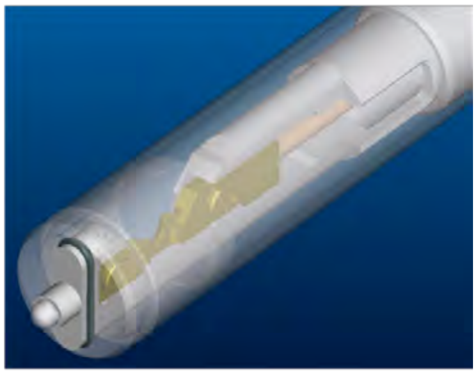


Fig. 3-3 When the tip is closed for air protection

By operating the opening-and-closing grip of the holder grip section, the holder tip is slid, and the sample and its vicinity are thus separated from the ambient air.

3. Advantages of the air-protection cryo-holder

The principal advantages are as follows:

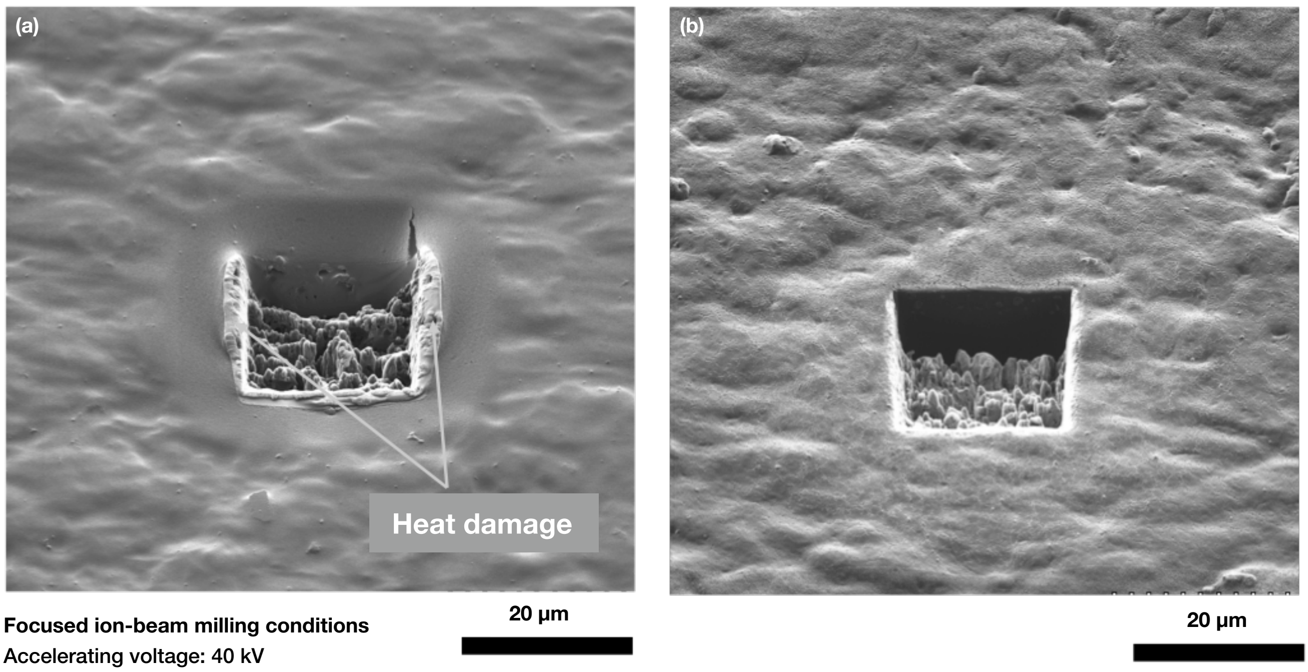
(i) Milling can be carried out while the sample is cooled, thus avoiding heat damage to the sample.
(ii) After milled, the sample can be transferred between instruments, without reloading.
(iii) Transfer between instruments can be achieved without exposure to the external atmosphere.

Samples can be cooled to -140ºC or lower by thermal conduction to the liquid nitrogen inside the Dewar flask fitted to the holder. It is also possible to adjust the sample temperature, between the cooling-limit temperature and -90ºC, using the associated temperature-adjustment unit.

4. Application examples

Figure 4 shows examples of polyester resin milled using a focused ion beam, at normal temperature (a), and at low temperature (b). The glass transition point of polyester resin is 60ºC, and severe heat damage therefore occurs in the milled section if treatment is carried out at normal temperature. When the sample is milled at -130ºC, on the other hand, the edge of the milled area is clean and sharp.

Normal-temperature | **Low-temperature (-130ºC)**

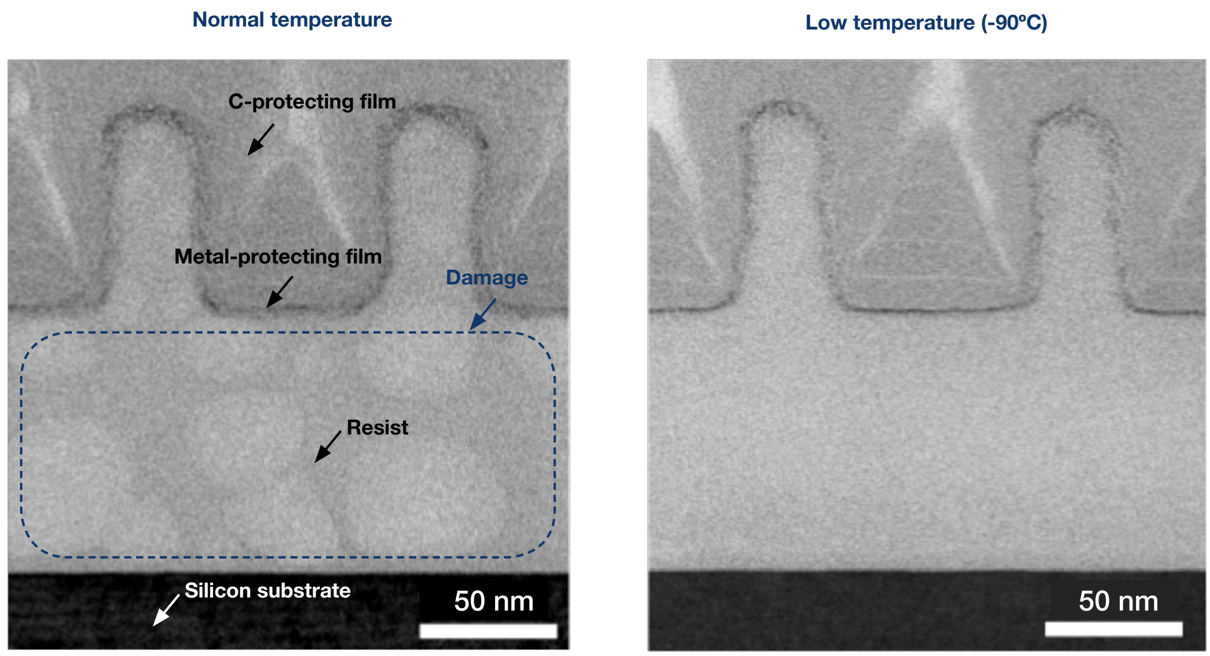


Focused ion-beam milling conditions
Accelerating voltage: 40 kV
Beam current: 26.56 nA
Magnification: × 2,000
Size of milled area: 20 µm × 20 µm × 15 µm

Milling/observation instrument: NB5000

Fig. 4 Examples of focused ion-beam milled **of polyester resin**

Resist is an organic film material, and is therefore susceptible to heat damage by ion and electron beams, and such damage, involving the film's structural density reduction, deformation, etc., occurs with milled and observation at normal temperature. Figure 5 shows the results of scanning transmission electronic microscopic observation (bright-field scanning transmission electronic micrographs) after milled of resist using a focused ion beam at normal and low temperatures. Milled and observation in the cooled state was found to markedly reduce the damage, due to the effects of cooling.



Focused ion-beam milling conditions
Milled instrument: FB-2100
Accelerating voltage: 10 kV

Scanning transmission electron microscopy observation conditions
Observation instrument: HD-2700
Accelerating voltage: 200 kV
Magnification: ×400,000

Fig. 5 Example of focused ion-beam milled and scanning transmission electronic microscopic observation of resist

5. Conclusions

The present report is primarily concerned with introducing an overview and the advantages of an air-protection cryo-holder with which samples can be milled and examined in a cooled state. Milling and morphological observation of micro-samples can be achieved without heat damage, so this cryo-holder will make a major contribution to fields such as materials analysis. In addition, the entire sequence of steps, from cooling to observation, can be carried out in a single holder, without exposing the sample to the atmosphere, markedly reducing the operating time. It is expected that this will lead to rapid progress in materials analysis and research.

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