A summary of the critical point drying method

Critical point drying is an established method of dehydrating biological tissue prior to examination in the Scanning Electron Microscope. The technique was first introduced commercially for SEM specimen preparation by Polaron Ltd in 1971. The original design concepts, which included a horizontal chamber, are still embodied in the design of the E3100 CPD models.

In recent years we have introduced two further models: the K850, which features built-in chamber cooling and heating, and the K850WM, which is designed for drying a 100 mm/4" silicon wafer.

The phase diagram shows the pressure to temperature ranges where solid, liquid and vapour exist. The boundaries between the phases meet at a point on the graph called the triple point. Along the boundary between the liquid and vapour phases it is possible to choose a particular temperature and corresponding pressure, where liquid and vapour can co-exist and hence have the same density. This is the critical temperature and pressure.

Critical point drying relies on this physical principle. The water in biological tissue is replaced with a suitable inert fluid whose critical temperature for a realizable pressure is just above ambient. The choice of fluids is severely limited and CO₂ is universally used today, despite early work with Freon 13 and nitrous oxide.

With CO₂ a critical point of approximately 35°C can be achieved at a pressure of around 1,200 psi. Therefore if the water is replaced with liquid CO₂ and the temperature then raised to above the critical temperature, the liquid CO₂ changes to vapour without change of density and therefore without surface tension effects which distort morphology and ultra structure.

Since liquid CO₂ is not sufficiently miscible with water, it is necessary to use an intermediate fluid which is miscible with both water and liquid CO₂. In practice intermediate fluids commonly used are methanol, ethanol, amyl acetate and acetone.