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Variable Temperature Insert for Paris-Edinburgh Press

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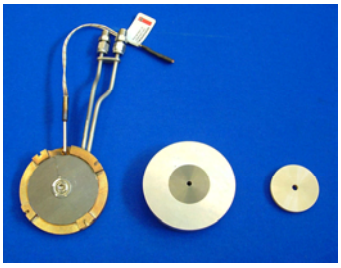
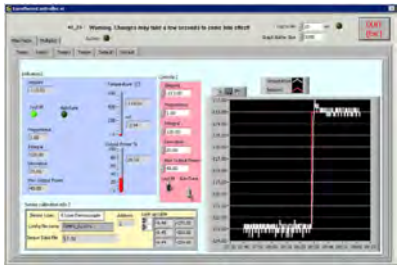
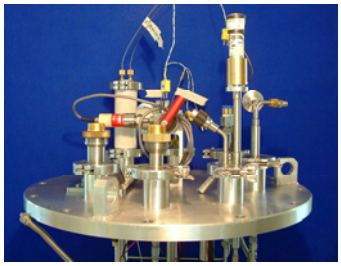
OXFORD INSTRUMENTS

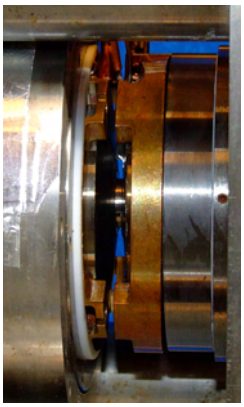
A prototype compact variable temperature (v-T) insert for the Paris-Edinburgh (P-E) press has been designed and developed at ISIS which is capable of varying the sample temperature over the 110 – 500 K interval with excellent control characteristics. The insert utilises a liquid nitrogen circuit to cool just the sample and the standard profile tungsten carbide anvils, with the latter incorporating the 240 W resistive heaters used for temperature control and heating.

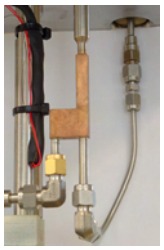

Tomkinson flange mounted PE Press


Anvil mounted in cooling ring with heater fitted shown with zirconia cored seat and backing disc


Eurotherm VI screenshot showing the stability & control characteristics of the v-T insert when operating at low-temperature.


Tomkinson flange complete with all necessary vacuum feedthroughs


Close up of insert in PE Press

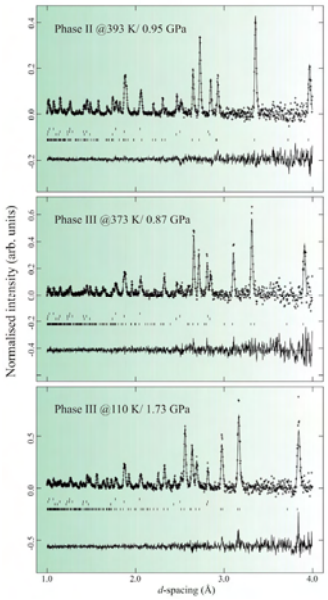


Liquid Nitrogen flow is controlled by an Oxford Instruments motor driven needle valve.

The v-T insert assembly is thermally insulated from the P-E press body by zirconia-cored seats and backing disks and PTFE insulation. The temperature of the P-E press cylinder housing is maintained close to ambient by means of a separate constant-temperature circuit. The whole installation in mounted on a Tomkinson flange and is designed to operate in all standard ISIS instrument vacuum tanks. The sample temperature and pressure can be monitored and controlled remotely from the ISIS instrument computer used for neutron data acquisition.

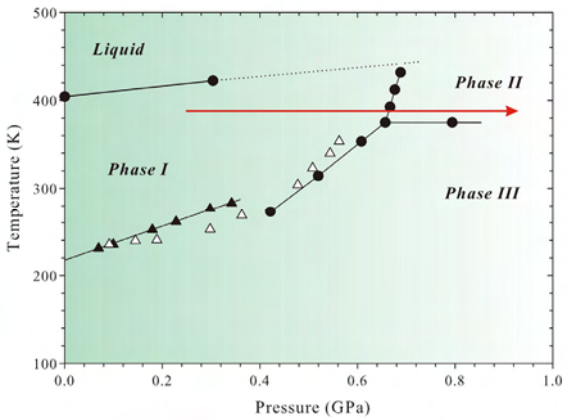
Cool-down times of the previous low-temperature P-E press setup – which required cooling the entire press using liquid nitrogen inside a large vacuum-walled tank – were typically 4-5 hours from ambient to 120 K. Recent commissioning tests of the v-T insert demonstrated the ability to cool the sample to 100 K or warm to 473 K from ambient temperature within ~45 minutes. In addition to a more efficient and productive use of neutron beamtime, this compact insert now makes possible high-pressure neutron diffraction experiments over the 100-500 K and 0-10 GPa regimes with a single P-E press sample loading, an entirely new capability.

The three figures below and to the left illustrate some recent results obtained using a sample of d_4-urea during the online commissioning of the v-T insert on the PEARL beamline at ISIS.

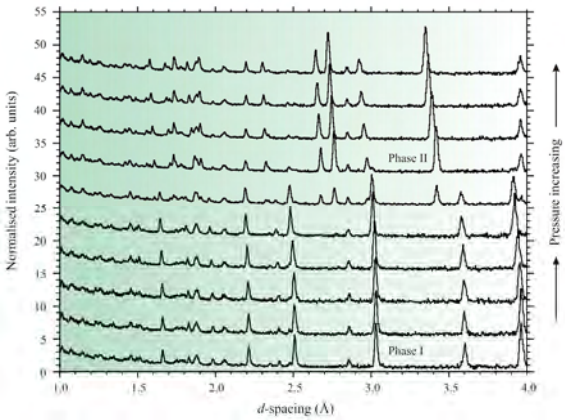

Anvil machined to take heater



Rietveld profile refinements at 393, 373 and 110K.



The urea p-T phase diagram. The arrowed red line indicates the p-T trajectory followed in collecting the multiplot patterns (right).



Multiplot showing the urea I-II transition along the 388 K isotherm.

Acknowledgements: The authors would like to thank the ISIS Sample Environment Electronics Section and ISIS workshop technicians for their help with this project.