

## X-ray Diffraction Studies of Argon in Confined Geometries

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**Introduction:** Confinement in disordered porous material has been known to affect the thermodynamic properties of imbibed liquid and solid phases. Confinement can not only change the microscopic structure of the adsorbed solid, but also eliminate or introduce new transitions or stabilize new phases. For example, studies of Ar and Kr in vycor glass report crystallization structures different from those in the bulk.

**Methods and Materials:** The Ar was confined in both powdered xerogel (Britesorb<sup>®</sup>) and powdered vycor glasses. The vycor and xerogel have nominal pore diameters of 70Å and 130Å and porosities of 30% and 72%, respectively. The powders were tightly packed into a cylindrical cell, with Be windows to allow the passage of X-rays. The cell was mounted on a closed cycle refrigerator and the temperature monitored with a silicon diode thermometer. The samples were filled with Ar such that they were 95% full at zero vapor pressure.

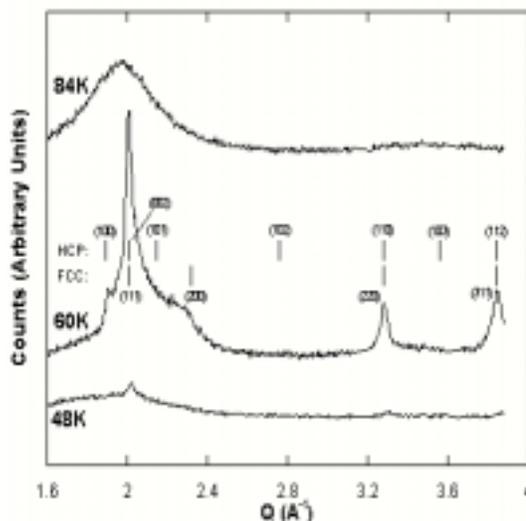
**Results:** The observed scattering from Ar in powdered xerogel, with the scattering from the xerogel removed, is shown in the figure below. At 85K the confined Ar has a typical liquid-like  $S(Q)$ , which is similar to that of bulk liquid Ar. There is a large peak at  $Q \sim 2\text{\AA}^{-1}$ , representing short-range correlations in the liquid, with several small oscillations (not seen here) at higher  $Q$ .

When the temperature is lowered below the freezing point of the confined solid ( $T \sim 77\text{K}$ ), crystallization occurs as evidenced by the appearance of moderately sharp diffraction peaks. These peaks can be indexed as the (111), (220), and (311) of an fcc structure. However, the (200) peak, which is very intense for the fcc structure, is missing. This is characteristic of the dhcp structure, which can be viewed as fcc with a maximum number of stacking faults as reported by Brown, et. al.. The width of these peaks,  $0.06\text{\AA}^{-1}$ , gives an approximate crystallite dimension of  $100\text{\AA}$ , comparable to the pore size of the Vycor. No changes in this structure were observed as long as the temperature remained above 54K.

The confined Ar exhibits a phase transition at  $\sim 54\text{K}$ , similar in temperature to that reported by Brown, et. al.. The nature of the transition, however, is markedly different from the previous reports. We find that on cooling below 54K, the dhcp peaks diminish considerably in intensity in a matter of 1-2 hours. This can be clearly seen in the 48K measurement. The diffraction peaks continue to diminish with time and after several hours, are no longer observable.

**Conclusions:** This suggests a migration of the Ar out of the pore walls. See Abstract No. *silv9791*, **Small Angle X-ray Scattering Studies of Ar in Xerogel**, for further investigations on this topic.

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**Figure 1.** Diffraction Scans of Argon in Xerogel at temperatures of 84K (liquid), 60K, and 48K. Note the disappearance of the DHCP peaks at 48K.