

**Experiment title:**

Structural investigations of polymers at very high pressures

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Report:

We wanted to perform two series of measurements from ambient pressure up to 15 GPa in 0.5-1 GPa steps - on polytetrafluoroethylene (PTFE) and on polyethylene (PE). As we got only half of the beamtime requested we could study just one sample, polyethylene.

The aim of the high pressure powder diffraction study on PE was to see whether the orthorhombic state of PE remains stable under pressure.

In the figure the measured unit cell dimensions are shown up to about 10 GPa. Above this pressure the Bragg reflections of PE became too low in intensity to be distinguished against the diffuse scattering of the diamond windows of the high pressure cell.

As can be seen, the orthorhombic phase remains up to 10 GPa. The linear compressibilities in a and b are much higher than in c. This can be explained by the fact that a and b are perpendicular to the molecular chains whereas c is in chain direction. However, at about 6 GPa a change in the slope of the curves occur (to show this more clearly b has been drawn separately). This has been observed on another polymer system as well (SC176, [1]) and we believe that this effect is not due to the sample but to the pressure transmitting liquid (a silicon oil). Therefore, we suggest that in future studies of polymers in a diamond anvil cell liquid nitrogen should be used as pressure transmitting liquid. This has the disadvantage that due to filling procedure at liquid nitrogen temperature the thermal history of the sample is changed and, thus, the morphology. However, this should not influence the crystalline structure itself.

Up to 6 GPa the volume-pressure data were fitted with the Birch equation which leads to a bulk compressibility at zero pressure of $1.29 \cdot 10^{-4}$ 1/MPa (literature: $1.14 \cdot 10^{-4}$ 1/MPa). In the diffraction patterns we observed a second crystalline phase of PE. Monoclinic PE occurs when shear is applied to the sample [2]. In our case the preparation of PE is responsible for its occurrence: Only a very small piece of sample can be put into the diamond anvil cell. Shear is applied when cutting the PE into shape with a scalpel. However, this has the advantage that we could study this phase - which has never been studied under pressure so far - simultaneously. Only three of the four lattice parameters of the monoclinic phase could be extracted from the data due to the lack of corresponding Bragg peaks. However, the missing parameter is b, which is the molecular chain direction and, thus, the direction with negligible linear compressibility in comparison to a and c (compare with orthorhombic c direction). The results of this experiment have been presented on the AIRAPT conference in Japan in 1997 and a publication is in preparation.

References

- [1] J. Mardalen, E.J. Samuelsen, O.R. Kontestabo, M. Hanfland, M. Lorenzen, submitted to Journal of Physics, Condensed Matter
 [2] T. Seto, T. Hara, K. Tanaka, Jap. J. Appl. Phys. 7 (1968) 3 1

