X-ray scattering experiment, the dynamic precipitation in an Al-Zn-Mg-Cu alloy was studied during quenching. It was shown that the precipitation process is much faster during straining of the materials as compared to static ageing. The 3D-XRD microscope at ID11 has been used to study the individual grain growth during annealing of Al single crystals. A movie of the grain growths of several grains in three-dimensions was produced. The third example presents a novel method to study stress and strain fields in amorphous materials. Previously only crystals could be characterised by diffraction, but now, by employing high-energy X-ray correlation functions, the strain tensors in a bulk metallic glass was determined. The dynamics of amorphous materials can also be followed using the coherence of the X-ray beam. The fourth example shows how the intensity fluctuations in the speckle pattern can be analysed by autocorrelation functions. In this example, X-ray photon autocorrelation spectroscopy was used to investigate the antiphase domain dynamics in the intermetallic phase $Co_{60}Ga_{40}$.

High-pressure studies are gaining in importance for a number of areas of materials science research. The new applications of laser heating have now opened up the field for studies both at high pressures and high temperature (> 5000 K). In one study the matter of electronic topological transitions due to distortions of the electronic band structure was studied in osmium by angle-dispersive X-ray diffraction. During the study of the equation of state it was found that the Fermi surface of osmium passes through a topologic singularity at 25 GPa. A second study deals with the synthesis of nitrogen. Nitrogen usually consists of dimers with strong triple bonding. In a synthesis above 2000 K and pressures above 110 GPa, a theoretically predicted cubic phase, polymeric nitrogen, was found as evidenced by a combination of X-ray diffraction and Raman techniques. The final example deals with the boundary between the earth's mantle and the core. In this region one notices a large contrast in the properties across the region. The interaction between iron and silica (SiO_2) was studied in an electrically and laser heated diamond anvil cell at pressures up to 140 GPa and temperatures over 3500°C. It was shown that iron and SiO_2 do not react at high pressures and iron-silica alloys dissociate into almost pure iron and a CsCl-structured FeSi compound. The existence of the FeSi at the base of the Earth's lower mantle could explain the anomalously high electrical conductivity of this region and provide a key to the understanding why the amplitude of the Earth's nutation is out-of-phase with tidal forces.

Å. Kvick

Materials and their Properties

Photodissociation of $C_2H_4I_2$ Studied by Picosecond X-ray Diffraction

How does the structure of a molecule change during a chemical reaction and how does the molecule interact with its nearest neighbours during the reaction? This is a very difficult question since most molecules are formed and broken extremely quickly. In fact the primary step in the formation of a molecule is the creation or breakage of chemical bonds or the transfer of electrons or protons. These steps are typically completed in 10-1000 femtoseconds, which is about 1000 times faster than the X-ray pulse from a synchrotron. A newly-formed molecule is usually created in a high-energy state, which relaxes into secondary structures by exploring all internal degrees of freedom in the molecule (turn angles, bond angles, bond lengths, etc). In this process, the molecule may reach a local minimum on the potential energy surface and later de-excite through collisions with its neighbours. These secondary structures, which typically have picosecond lifetimes in liquids, can be determined by single pulse X-ray diffraction from photosensitive molecules that can be activated by a short laser pulse. Here we report on the photo detachment of I_2 from diiodoethane $C_2H_4I_2$, see **Figure 22**. We now know that molecular iodine I_2 is formed in a two-step process, which involves the unstable C_2H_4I radical:

$C_2H_4I_2 + h\nu \rightarrow C_2H_4I + I \rightarrow C_2H_4 + I_2$

Note that quantum chemistry predicts two intermediate structures of C_2H_4I in methanol (CH_3OH) at nearly the same energy, the bridged and the anti structure. The experiment, which was done on beamline **ID9B**, shows that the bridged structure alone is formed in methanol, a fact that we ascribe to nearest neighbour interaction in liquid methanol.

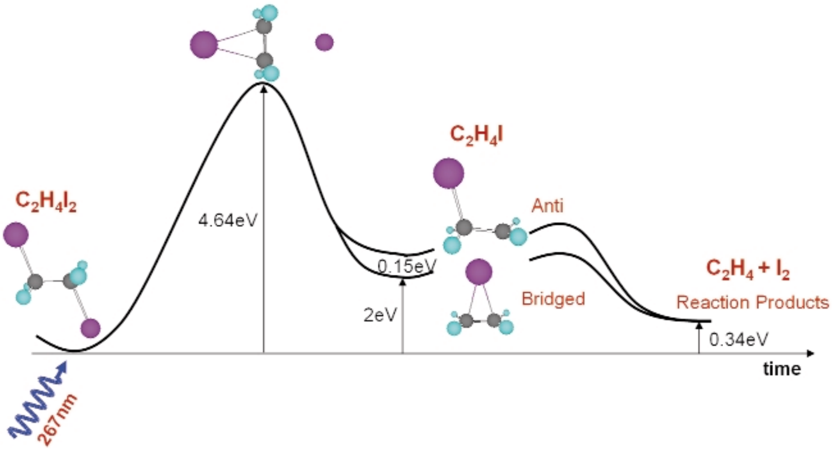

Fig. 22: Calculated energy landscape of the photoproducts of $C_2H_4I_2$. Iodine is pink, carbon gray and hydrogen blue.

The $C_2H_4I_2$ molecules were dissolved at a concentration of 1:400. The $C_2H_4I_2$ molecule is excited by the absorption of a UV pulse (2 ps, 267 nm) in the C-I stretched band, which leads to the emission of an iodine atom into the solvent. In 60% of the excited molecules, the freed iodine atom thermalises in the first solvation shell and recombines geminately. In 40% of cases, however, the C_2H_4I radical is formed and we would like to determine its structure, lifetime (decay mechanism) and the liquid structure around it. In the experiment, the pulsed laser beam (2 ps, 267 nm, 986 Hz) and the pulsed X-ray beam (100 ps, 0.07 nm, 986 Hz) were focused onto a 0.3 mm thick sheet of liquid formed by a sapphire nozzle. By suitably adjusting the speed of the liquid, a fresh sample was injected into the beam every millisecond. The time resolution is then simply the delay between the pump and the probe. The diffracted beam was recorded on a CCD detector and the images were recorded in pairs *with* and *without* excitation. The white beam from the single-harmonic undulator U17 was used to compensate for the low frequency of the experiment. The X-ray beam was focused by a toroidal mirror to 0.10 x 0.05 mm^2 and the flux was 5 x 10^{11} ph/s in a 2.5% bandwidth around 18.0 keV. The difference in the radial intensity $\Delta S(q, \tau)$, non excited – excited, is oscillatory and strongly dependent on the time delay. The associated Fourier transform is a measure of the change in the atom-atom pair distribution functions $g_{ij}(r)$ within the molecule [1,2], see **Figure 23.**

The negative peak at 5.1 Å coincides with the I..I distance in $C_2H_4I_2$. The measurements tell us that this correlation is reduced, *i.e.* at least one iodine atom has left the molecule after 100 ps. The second negative peak at 2.1 Å comes from a change in the C-I correlation. This feature is consistent with the formation of the bridged structure. The measurements at later times show, rather dramatically, that the liberated iodine atoms “swim” around the C_2H_4I radical and strips off the second iodine to form $C_2H_4 + I_2$. This diffusive process takes about 10 ns. Note finally that the radial curves $\Delta S(r, \tau)$ provide a 1-dimensional moving image of a chemical reaction in solution: it is probably the closest we get to film chemical reactions in disordered media with X-rays.

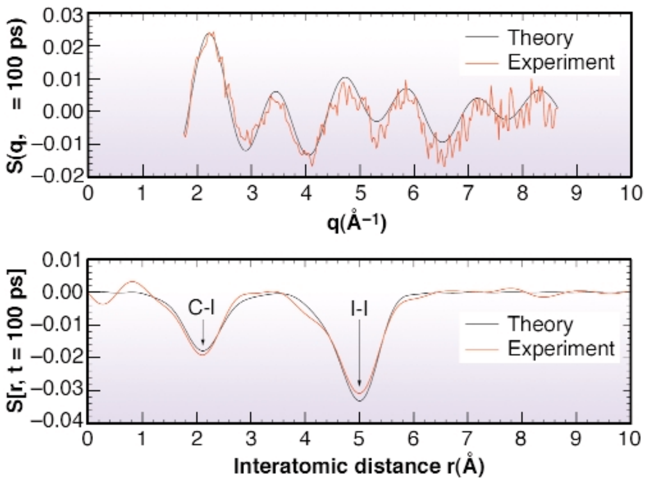

Fig. 23: The transition $C_2H_4I_2 \rightarrow C_2H_4I + I$ produces an oscillatory change $\Delta S(q)$ in the diffracted intensity measured here 100 ps after dissociation. The Fourier transform $\Delta S(r)$ is shown below.

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Principal Publications and Authors

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