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Summary of X-ray Diffraction Experiments on KCl Performed During Spring 2000

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I. Introduction

The purpose of this report is to summarize several x-ray diffraction experiments performed on KCl single crystals. KCl undergoes a phase transition from the rocksalt structure (NaCl) to the CsCl structure when shock compressed above 2 GPa. X-ray diffraction studies have been performed[1] on KCl shocked along the [100] direction and the results demonstrated that x-ray diffraction is a useful method for determining the structure and orientation of the new phase with respect to the initial phase. Those results showed that when shocked along [100] above the phase transition, the phase II crystal structure is oriented such that the [110] axis is along the direction of shock propagation. The purpose of the experiments reported here were to repeat and verify some previously performed experiments on KCl shocked along [100] and to begin to investigate KCl shocked along [111] above the phase transition. Although the [100] shots were consistent with previous results, no diffraction data were obtained from phase II when KCl was shocked along [111] above the phase transition.

This report outlines the procedures used to prepare the samples, summarizes the results obtained from each experiment performed, and gives some recommendations for future experiments.

II. Target Fabrication

The general target configuration used to investigate the phase transition in KCl single crystals is shown in Fig 1. The front and mid buffers were z-cut a-quartz for stresses below the phase transition stress and OFHC copper for stresses above the transition stress. Samples were single crystal KCl cut along either the (100) or (111) face. The window used in all experiments was vitreous carbon. Typical dimensions are shown below.

z-cut quartz front buffer:	0.5 mm
z-cut quartz mid buffer:	3.5 mm
Copper front buffer:	0.8 mm
Copper mid buffer:	2.0 mm
KCl sample (both orientations):	0.3 mm
Vitreous carbon window:	0.4 mm

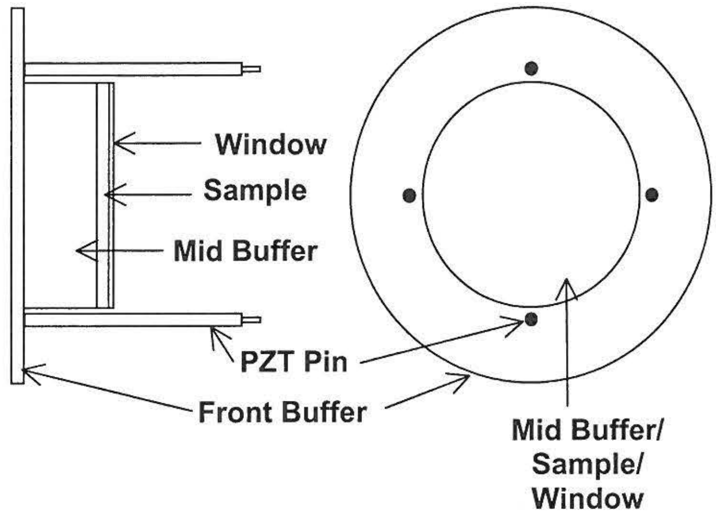

Figure 1

Sample Preparation

The following procedure was used to prepare and assemble the target components:

Buffers: If using copper, the buffers were first lapped flat using the lapping machine. Next the copper mid buffer was polished on one side using progressively finer polishing paper to remove the dull surface left by lapping and to ensure that the sample was flat. I started with 30 micron paper and ended with 9 micron paper using ethanol as the lapping medium; water can also be used. Quartz buffers had an optical finish and were used as is.

Samples: Both [111] and [100] KCl single crystal samples were prepared in the same manner. One side of the crystal was polished using 30 micron and then 9 micron polishing paper with ethanol as the lapping medium. However, I recommend that both sides be polished in this manner to ensure that all major scratches are removed from the bonding surface. It is difficult to ascertain the quality of the surface if both surfaces are not polished.

Windows: The vitreous carbon back windows were prepared as outlined elsewhere.[2]

Assembly

Step 1: Measure thickness of sample and buffer using the Super Micrometer. Bond sample to buffer using slow cure, low viscous epoxy. Allow 24 hours to cure.

Step 2: Lap KCl to within 50 microns of the desired thickness using 320 grit sandpaper and Lapmaster lapping oil. Switch to 30 micron polishing paper and ethanol and work down to 1 micron polishing paper until the desired thickness is achieved. Make sure the KCl surface remains parallel to the mid buffer surface.

Step 3: Remove all but about a half to one millimeter of the white epoxy on the back of the VC window. Make sure that the epoxy surface is parallel to the VC surface. Bond VC to KCl using slow cure epoxy. Wait 24 hours

Step 4: Bond thin front buffer to mid buffer using 815 epoxy. Wait 8 hours. The slow cure epoxy is not required here because stresses induced by a fast curing epoxy are not an issue at this interface.

Step 5: Prepare the PZT pins in the target ring as outlined elsewhere.[2]

Step 6: If the orientation of the sample with respect to the PZT pins is not an issue, bond the sample to the target ring using 815 epoxy. If the orientation of the crystal is important, then care must be taken to ensure that after the sample is bonded to the target ring, correctly orienting the target in the gun does not place one of the trigger pins in either the incident or reflected x-ray beam.

Step 7: Mount target ring/sample to outer target ring using the procedure outlined in ref. 2. Connect cables to pins.

III. Optimizing Diffraction Signal

Polishing Sample Surface

When shocking KCl along [100] above the phase transition it is known that the (100) planes become (110) planes in the new phase.[1] In order to observe the diffraction peak from the (110) planes in Phase II, it is necessary to set the x-ray source at the correct Bragg angle. For example, the Bragg angle for the (200) planes under ambient conditions is 6.48°, while the Bragg angle for the (110) planes in the phase II structure at 70 kbar is approximately 8°. Therefore, the source must be moved over 1.5° from the optimized position under ambient conditions to observe a diffraction peak under shocked conditions. Figure 2, below, illustrates that the Bragg condition for the (200) planes should no longer be satisfied on the crystal (25 mm diameter typical) under ambient conditions after the source is placed in the shot position. However, in all cases, a weak diffraction peak was still observed and was also at the correct position on the detector predicted by the geometry shown in Fig. 2 for a larger crystal.

The origin of the signal is from the imperfect nature of the diffracting surface. The lapped surface of the crystal can though of being composed of many slightly misoriented crystallites as shown in Fig. 3. Under ambient conditions, each crystallite satisfies the Bragg condition for some ray in the diverging incident beam and because focusing geometry[2,3] is used, each diffracting ray will be incident on the detector at the

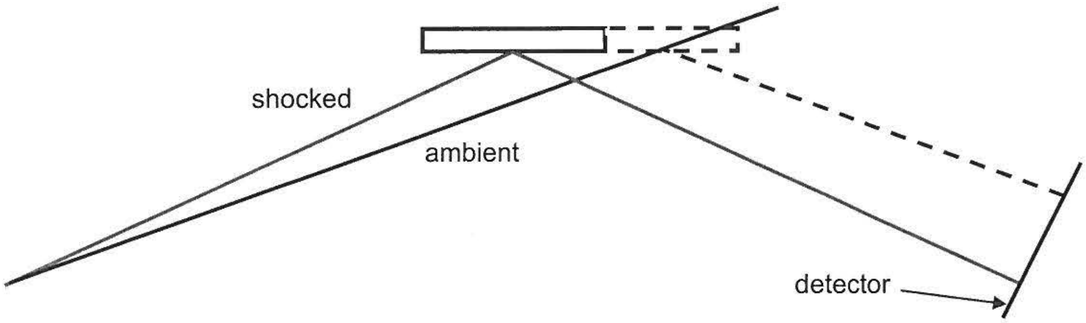

Figure 2

same point. Thus, the appearance of an ambient diffraction signal from the crystal when the x-ray source is far from the Bragg conditions is explained by the fact that a divergent beam is being used and because the surface of the crystal is highly imperfect allowing the Bragg condition to be satisfied over a large angular range. This also explains the weak intensity of the signal. As the source is moved further from the ideal diffraction angle, fewer "crystallites" satisfy the Bragg condition for the incident x-rays and, therefore, fewer x-rays are diffracted.

Polishing the surface of the sample as outlined in the previous section can dramatically reduce this effect as well as enhance the diffraction signal. This means that the diffraction signal from a polished crystal is more localized than from a lapped crystal. Since it is important that the diffracted signal come from the center of the sample during shock compression (due to the presence of edge effects), samples used in these experiments should be polished since the signal from a lapped crystal has contributions from the entire surface of the crystal, not just the central portion.

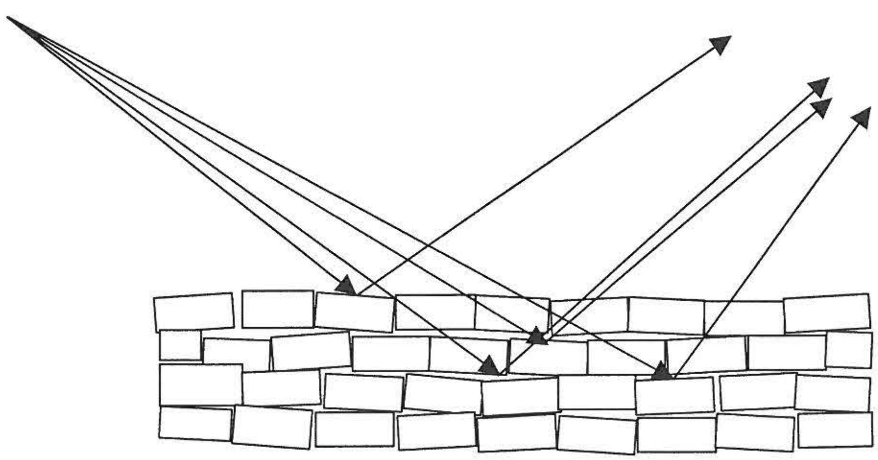

Figure 3

Centering X-rays on Sample

Due to the shallow incident angle for the diffracting x-rays in these experiments, special care must be taken to ensure that the x-rays are diffracting from the center of the sample. If the alignment laser used to indicate the axis of the main x-ray beam is misaligned by as little as 0.25°, centering the target on the laser beam can result in the diffraction signal coming from the edge of the crystal. It is not sufficient to check the alignment of the laser carefully. Other steps must be taken to make sure the diffraction signal is coming from the center of the target.

During the initial setup of the experiment, the target was positioned using the laser in the usual was. Then, once the optimal signal was obtained (which would normally indicate that the beam was centered on the target), all but the central region of the target was masked using thin (0.010") steel pieces. These pieces were affixed to the sample using a thin coating of vacuum grease so then could easily be positioned and then removed after the alignment procedure was completed. The central portion of the sample that should remain uncovered must be large enough that the "shadow" cast by the steel shims does not cover the crystal. After masking the sample, it was then translated through the beam until the most intense diffraction signal was obtained. This then indicated that the x-ray source was pointed directly at the center of the target.

IV. Experimental Results

Seven experiments were performed on KCl shocked along both the [100] and [111] directions. For each experiment, several diagnostic signals were recorded to monitor the x-ray system and to coordinate the time of x-ray output with the shock wave event.[2] The output of the SRS delay generator, pulser, and the PMT (x-ray output) were recorded on one scope, while the signal from the PZT pin 4, the tilt invariant trigger circuit and the SRS delay generator (teed from the first scope) were recorded on the second scope. The scope setup is given below.

Scope 1:

Channel 1: SRS delay generator; TTL; 500mV/div
Channel 2: Pulser; ~90V; 500mV/div (with 30X attenuation)
Channel 3: PMT; ~40 mV; 5mV/div (HV power supply set to 1000V)

Scope 2:

Channel 1: PZT pin 4; ~4V (after 10X attenuation at Tilt scope); 500mV/div
Channel 2: Tilt circuit; TTL; 500mV/div
Channel 3: SRS delay generator; TTL; 500mV/div

MoKα x-rays were used in all experiments. For crystals shocked along [100] the (200) peak was used as a reference. For crystals shocked along [111] the (222) peak was used as a reference. The procedure for calculating the position of the expected phase II peak has been given for shock compression along [100] by d'Almeida and Gupta.[1] For compression along [111] the same procedure was used, but it was assumed that the (200) planes would be perpendicular to the shock propagation direction. In other words, the (222) planes would become (200) planes in the new phase.

The results of each experiment performed are summarized below. More experimental details can be found in the shot folders for each experiment.

Shot	Orientation	Impactor/ Velocity (mm/μs)	Stress (GPa)	Predicted Peak	Detector	Comments
00-306	[100]	OFHC Cu 0.881	5.0	(110)	I-CCD	Observed nothing
00-307	[111]	6061-Al 0.344	1.6	(222)	I-CCD	Isotropic compression
00-308	[100]	OFHC Cu 1.209	7.0	(110)	I-CCD	Consistent with previous
00-309	[111]	OFHC Cu 1.185	6.7	(200)	I-CCD	Observed nothing
00-310	[100]	OFHC Cu 0.905	4.8	(110)	I-CCD	Consistent with previous
00-311	[111]	OFHC Cu 1.210	6.9	(200)	Film	Film damaged
00-316	[111]	OFHC Cu 1.210	6.9	(200)	Film	Observed nothing

V. Summary

The three experiments performed on [111] above the phase transition showed no evidence of the (200) diffraction peak in phase II. In Shot 00-309, the orientation of the crystal about the [111] axis was arbitrary because we expected the (222) planes to become (200) planes in the shocked state. After nothing was observed in this experiment, a second possibility for the position of the (200) planes was considered. Since we know that the (200) planes become (110) planes in phase II when KCl is shocked along [100], the possibility was considered that this always occurs, regardless of the shock propagation direction. If true, then upon transformation the normal to the (200) planes will be approximately 10° from the original [111] axis (it is helpful to look at the ball and stick models to understand this). Thus, due to the size of the detector, we would not have been able to observe this peak with the configuration used for that experiment.

To test this hypothesis, a second experiment, Shot 00-311, was performed. In this experiment, the crystal was oriented such that the projection of the [001] axis on the (111) planes (crystal surface) was pointing along the lab frame z-axis (up). Therefore, if the crystal transformed as predicted, the normal to the (200) planes would remain in the x-z plane of the lab frame with x pointing along the direction of shock propagation. Film was also used as the detection medium so that we would have broad coverage to detect a diffraction peak in a position other than that predicted. However, to achieve this broad coverage requires that the window in the flange on the detector side of the target chamber be quite large. During the experiment, the mylar on the window blew out and heavily damaged the film cassette so no results were obtained.

After modifying the detector flange so that the window would remain intact during the shot, the experiment was repeated. Timing diagnostics showed that the x-rays were turned on while the crystal was at peak compression in phase II. However, no diffraction peak of any kind was observed on the film. The reason for this is still unclear and needs to be investigated further.

Final Note

Upon looking through Thierry d'Almeida's shot folders, I discovered an experiment that had been performed on [111] just above the phase transition (2.8 GPa). Thierry predicted that the (200) planes would be observable in phase II as I did and like for Shot 00-309, assumed that the (200) planes would replace the (222) planes and therefore the orientation of the crystal was unimportant. Film was used as the detection medium, but in contrast to my results, two diffraction peaks were observed at this stress. One peak was observed to the left of the ambient (222) peak position and Thierry proposed this to be the (200) peak from phase II and the peak position was basically in agreement with this proposal. The second peak was to the right of the ambient peak and was proposed to be a shifted (222) peak corresponding to the compressed phase I lattice at 2 GPa (the transition stress).

The reason that the experiments reported here were performed at 7 GPa is that at this stress, the phase transition is overdriven resulting in a single shock wave propagating through the crystal as opposed to the normal two wave structure associated with a phase transition.[1] In the [100] experiments, it seemed as if the transformation was more complete when the samples were shocked above 7 GPa because brighter diffraction peaks from the new phase were observed at this stress as opposed to diffraction peaks obtained at lower stresses. However, in light of the result described above, future experiments on KCl shocked along [111] should be performed at lower stresses.

References

1. T. d'Almeida and Y. M. Gupta, Phys. Rev. Lett.
2. P. A. Rigg, Ph.D. Thesis, Chap. 3, 1999 (unpublished).
3. Y. M. Gupta, K. A. Zimmerman, P. A. Rigg, E. B. Zaretsky, D. M. Savage, and P. M. Bellamy, Rev. Sci. Instrum. **70**, 4008 (1999).