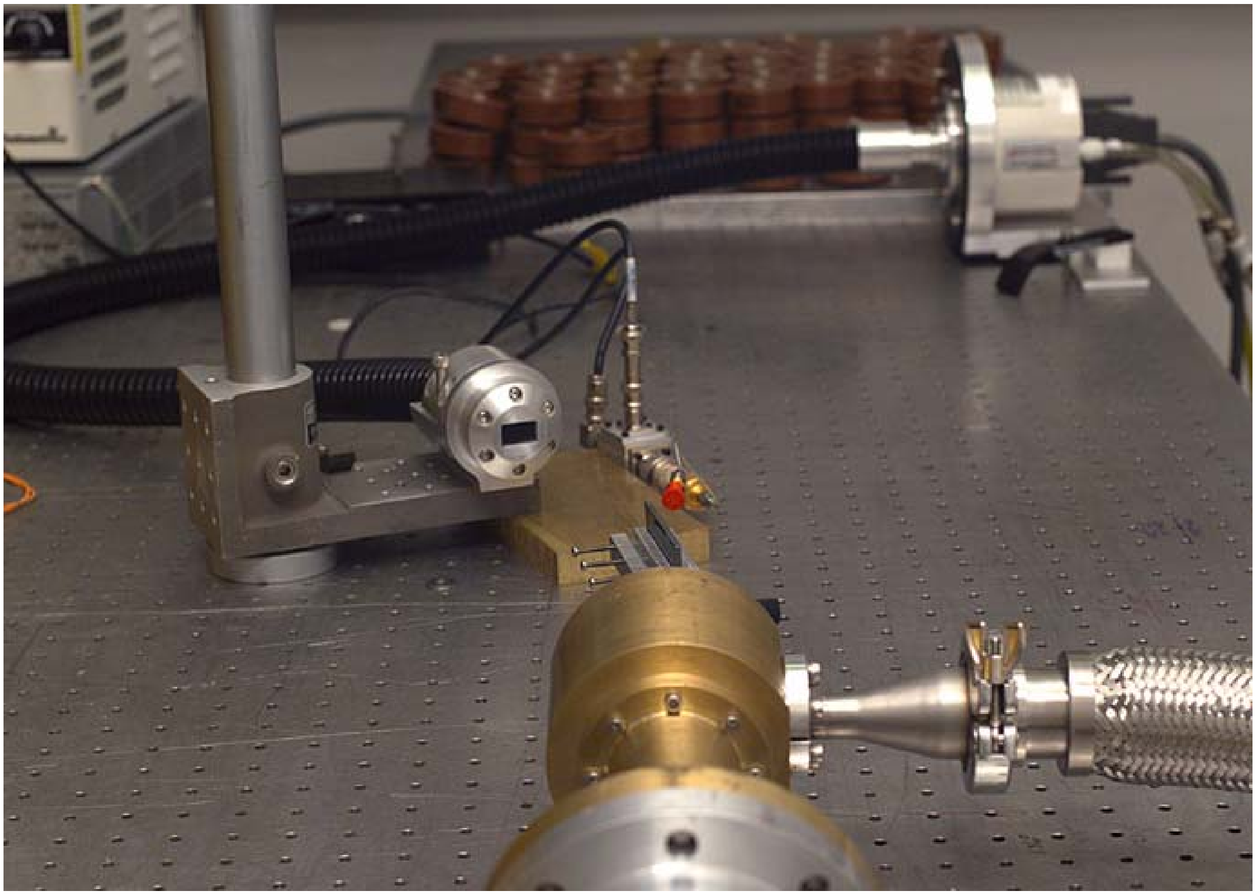
DOE/NV/25946--078

X-Ray Diffraction Project Final Report, Fiscal Year 2006



October 2006

Prepared for the U.S. Department of Energy, Nevada Operations Office
by National Security Technologies, LLC under Contract No. DE-AC52-06NA25946

Sandia National Laboratories

National Nuclear Security Administration

National Security Technologies LLC

DOE/NV/25946--078

Available for sale to the public from:

U.S. Department of Commerce
National Technical Information Service
5285 Port Royal Road
Springfield, VA 22161-0002
Phone: 800.553.6847
Fax: 703.605.6900
e-mail: orders@ntis.gov
online ordering: http://www.ntis.gov/ordering.htm

Available electronically at http://www.osti.gov/bridge

Available for a processing fee to U.S. Department of Energy and its contractors, in paper, from:
U.S. Department of Energy
Office of Scientific and Technical Information
P.O. Box 62
Oak Ridge, TN 37831-0062
Phone: 865.576.8401
Fax: 865.576.5728
e-mail: reports@adonis.osti.gov

Table of Contents

List of Abbreviations and Acronyms

CCD	charge-coupled device
DXD	dynamic x-ray diffraction
FWHM	full width at half maximum
IDL	Interactive Data Language
LAO	Los Alamos Operations (NSTec)
LiF	lithium fluoride
LSF	least squares fit
SNL	Sandia National Laboratories
STL	Special Technologies Laboratory (NSTec)
TTL	transistor-transistor logic

X-Ray Diffraction Project Final Report, Fiscal Year 2006

Dane V. Morgan
National Security Technologies, LLC

1.0 ABSTRACT

An x-ray diffraction diagnostic system was developed for determining real-time shock-driven lattice parameter shifts in single crystals at the gas gun at TA-IV at Sandia National Laboratories (SNL). The signal-to-noise ratio and resolution of the system were measured using imaging plates as the detector and by varying the slit width. This report includes tests of the x-ray diffraction system using a phosphor coupled to a charge-coupled device (CCD) camera by a coherent fiber-optic bundle. The system timing delay was measured with a newly installed transistor-transistor logic (TTL) bypass designed to reduce the x-ray delay time. The axial misalignment of the Bragg planes was determined with respect to the optical axis for a set of eight LiF [lithium fluoride] crystals provided by SNL to determine their suitability for gas gun experiments.

2.0 SINGLE CRYSTAL DIFFRACTION MEASUREMENTS WITH IMAGE PLATES

The purpose of these image plate experiments was to obtain quantitative flash x-ray diffraction images using the fluorescent K-shell emission spectral lines of copper and molybdenum. Precision silicon crystals with (111) and (220) orientations purchased from Inrad were used to produce the diffracted spectrum. Data files were obtained from the scanned image plates that allow quantitative measurement and comparison to optimize the diffraction system. Both the resolution of the image plates and the signal-to-noise ratio were measured.

The dynamic x-ray diffraction (DXD) experimental setup is shown in Figure 1. Experiments were performed both at the Boom Box at Special Technologies Laboratory (STL) and in the Pulsed X-ray Laboratory at Los Alamos Operations (LAO). The DS-2158 cable diode assembly was used in conjunction with both 25-stage A3 Marx banks and a 38-stage Supersaver Marx which generated single-pulse exposures. One-eighth-inch-diameter anodes of copper and molybdenum were used to generate the K-α1 and K-α2 characteristic emission lines. The cathode diameter was 0.25″.

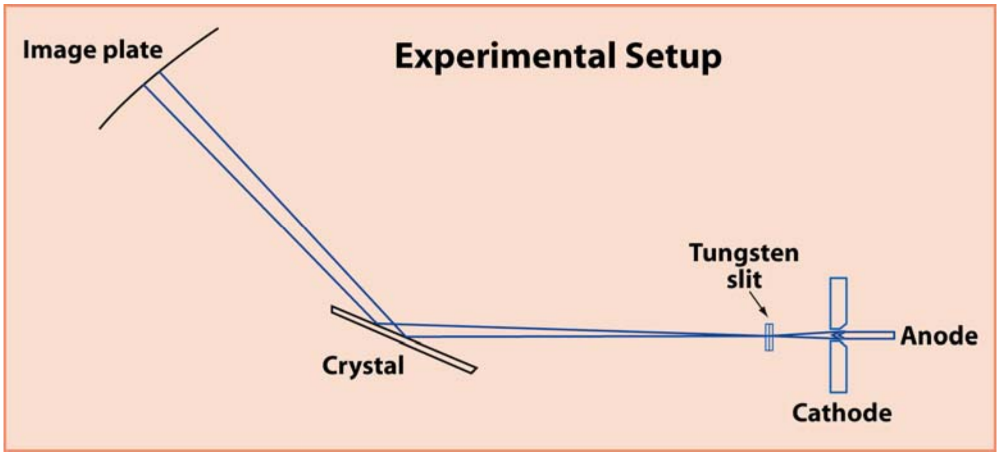


Figure 1. Schematic diagram of the DXD experimental setup

Resolution measurements were only possible when the doublet was resolved. The slits are a pair of 0.25″ diameter rolled tungsten edges, and measurements were performed with the slit openings set between 0.002″ and 0.030″ using a feeler gauge. The image plate was loaded in a specialized film holder apparatus that is curved in a 7.0″ radius arc to ensure the source slits and the image plate are equidistant from the crystal. These distances replicate the configuration at the SNL TA-IV gas gun. The image plate scanners used at STL and LAO were Fuji Medical Systems Model FLA 5100. Diffraction images were recorded on Fuji BAS-MS imaging plates. These systems were initially set to scan in 10-µm increments; however, it was later determined that 25-µm scan increments were sufficient considering the limiting resolution of the image plates, thereby reducing the size of the image file. The files were recorded in both a linear TIFF format and a logarithmic Fuji image format.

A total of eight diffraction images were taken with the x-ray source at different slit settings, at which time the diode was cleaned and refurbished and the anode was replaced. A typical diffraction image observed with a copper anode using a Si (220) diffraction crystal is shown in Figure 2.

Line-outs were generated by averaging over a 0.5-mm vertical section of the image. Line-outs of the copper K-α emission spectrum with the Si (220) crystal with slit separations of 0.102 mm, 0.203 mm, and 0.381 mm are shown in Figure 3. For slit separations less than approximately 0.25 mm, the doublet is easily resolved. A Gaussian least squares fit (LSF) of the K-α1 and K-α2 lines was also performed to obtain the full width at half maximum (FWHM) for the two lines. The width parameter was locked for the K-α1 and K-α2 lines so they would have the same width for the purposes of the fit. The blur in all cases was large compared with the associated rocking curves of the Si (111) and Si (220) crystals and the intrinsic line width of the fluorescent decay.

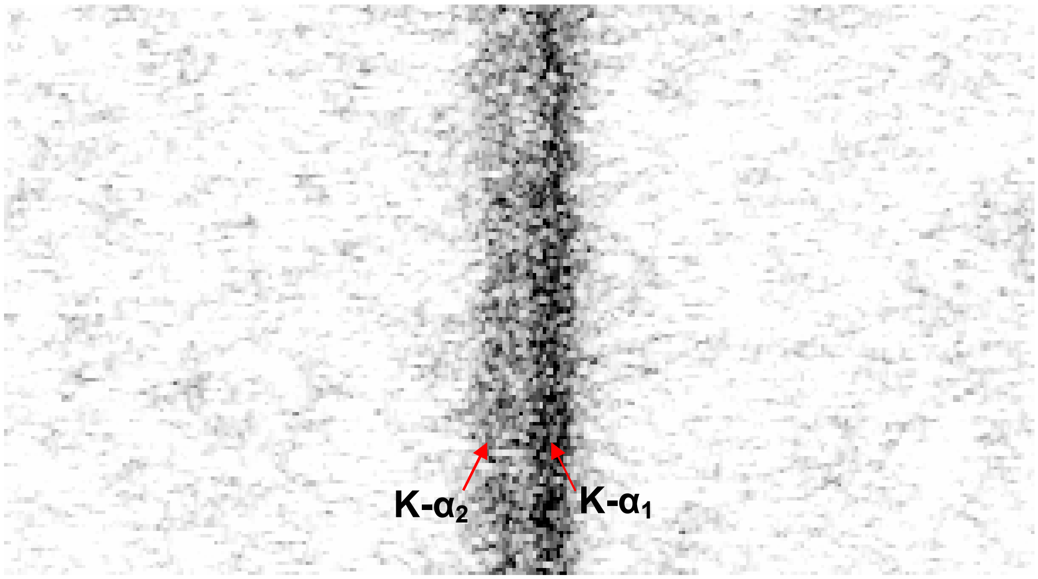


Figure 2. Copper K-α emission spectrum observed on image plates with a Si (220) crystal. The slit setting for this image was 0.254 mm.

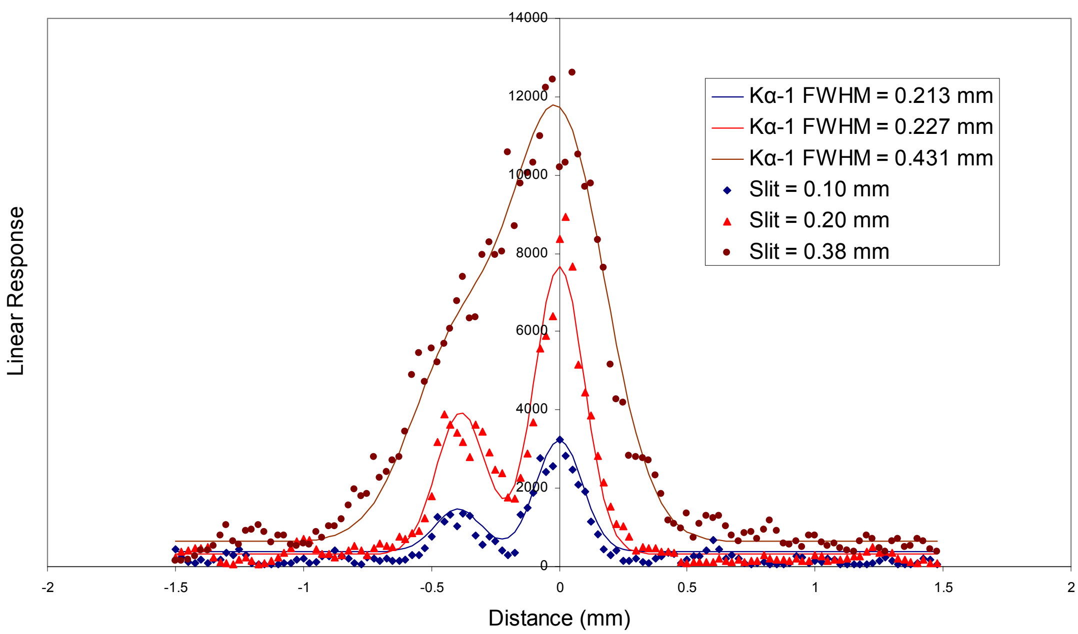


Figure 3. Line-outs of the diffracted copper K-α line, along with the Gaussian LSF calculation. The blur increases with increasing slit separation.

If the crystal effects and the intrinsic line width of the spectral lines are neglected, the total image blur at 8.0 keV can be approximated by

$$W_{tot} = (W_{IP}^2 + x^2)^{1/2}, \qquad [1]$$

where x is the slit separation and W_{IP} is the image plate blur. Blur measurements for the Si (220) copper K-α diffraction images are plotted as a function of slit separation in Figure 4. From Figure 4, we can infer a reasonable agreement between the experimental data and the model described by equation [1].

An LSF calculation of the experimental data was performed using W_{IP} as a free parameter. From this analysis, the zero crossing of the fit curve is the image plate resolution at 8.0 keV. The image plate blur determined by this method was 0.181 ±0.005 mm. This information is also useful for radiographic applications and analysis.

Another important consideration for the analysis of dynamic x-ray diffraction on image plates is the signal-to-noise ratio. The intrinsic noise, or the noise that exists on the image after scanning the plates after erasure, depends on many factors including the scan parameters, the image plate properties, and the elapsed time since image plate erasure. The image plate background noise measurements were conducted with both the Supersaver and the A3 Marx. The dose output of the Supersaver is approximately double the output of A3, with an endpoint energy that is higher by a factor of about 1.5. Shielding was sufficient to eliminate direct exposure from the anode, which implies that the x-ray background appearing on the image plates is a combination of inelastic (Compton) scattering from the crystal and the air in the beam path. This

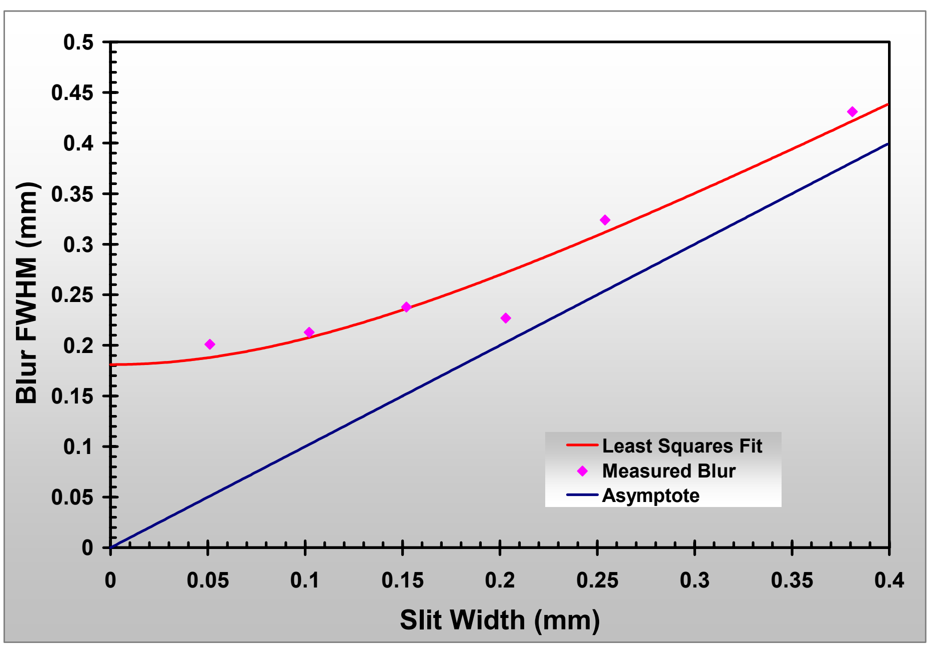


Figure 4. Image plate blur vs. slit width

hypothesis is supported by the results shown in Figure 5. The increase in background noise with increasing slit width shows that the background noise and level are dominated by the x-ray intensity at the crystal.

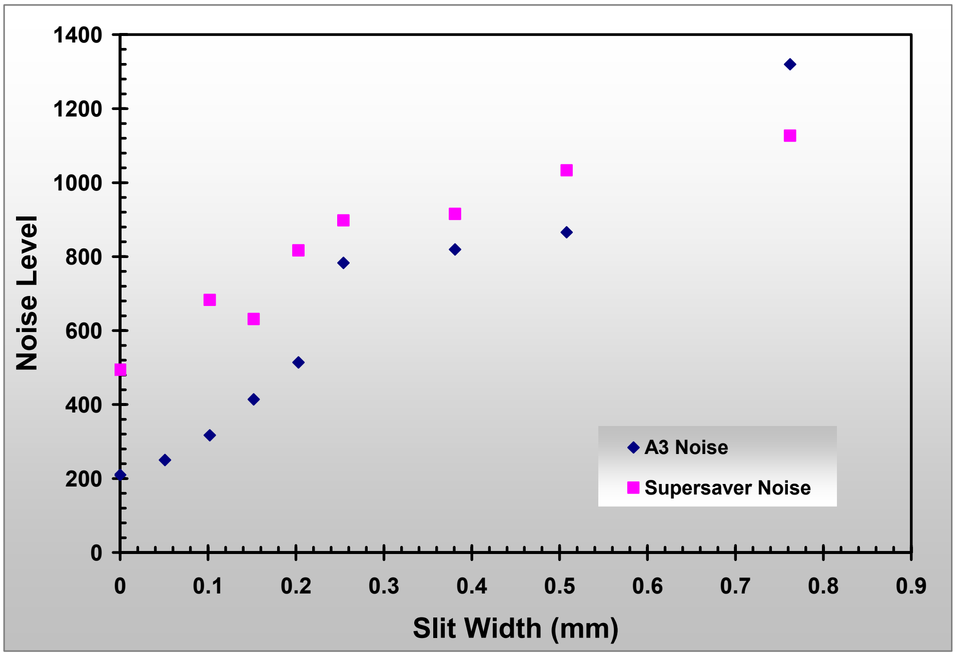


Figure 5. Root-mean-square background noise vs. slit width for the A3 Marx and the Supersaver

3.0 DETECTION SYSTEM WITH CCD CAMERA, FLUOR, AND COHERENT FIBER-OPTIC BUNDLE

An improved detection system consisting of a specialized fluor coupled to a CCD camera by a coherent fiber-optic bundle was developed for the gas gun experiments at TA-IV. The diffraction system, including the CCD fluor-coupled detector, is shown in Figure 6.

Experiments with two phosphors were conducted. The first phosphor was an HD-S Agfa mammography product typically recorded on film, and the second was a phosphor from Grant Scientific Company, and was 25 mg/cm^2 Gd2O2S:Tb (P-43). The phosphor from Grant was deposited on an aluminized Mylar mirror to increase its detectable light output.

The Supersaver Marx, described previously, became available on a continuous basis to this project in June 2006, so all experiments with the CCD-camera-coupled fluor were conducted with this source. The K-α1 and K-α2 copper lines observed with a LiF (200) crystal are shown in Figure 7. The signal-to-noise ratio for the fluor-coupled detector was about a factor of ten better than the diffraction lines observed with image plates.

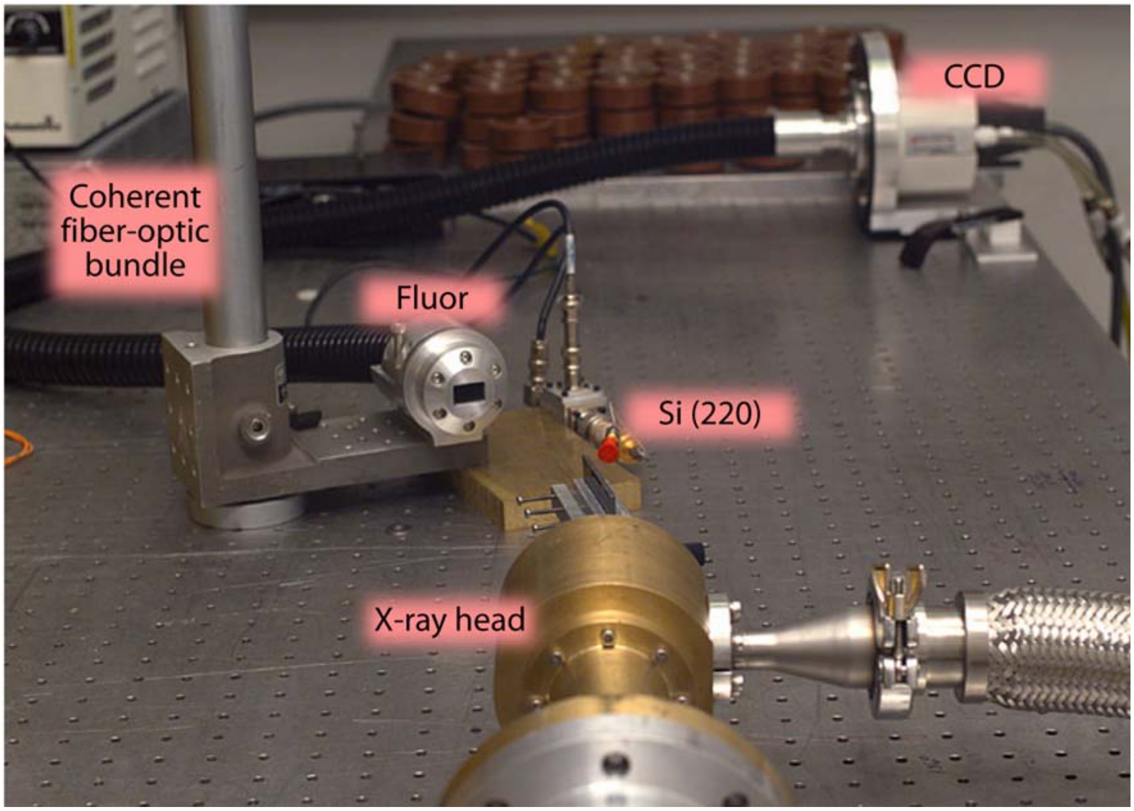


Figure 6. View of the dynamic x-ray diffraction system with a Si (220) single crystal and a fluor coupled to a cooled CCD camera with a coherent fiber-optic bundle.

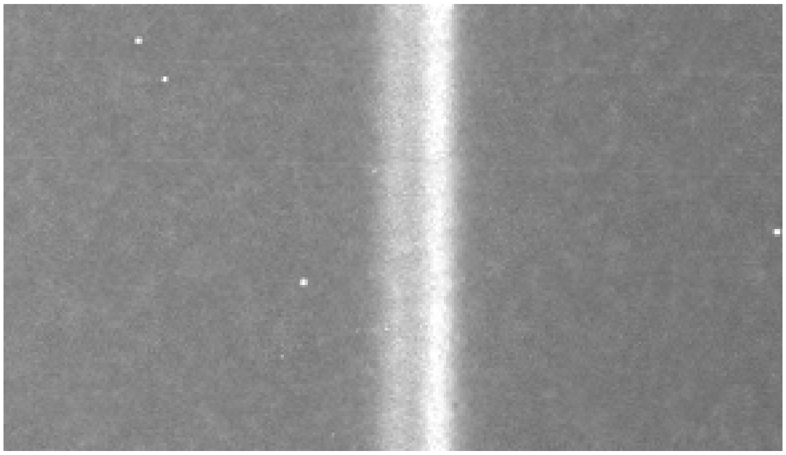


Figure 7. Copper K-α emission spectrum observed on a CCD camera coupled to a scintillator with a LiF (200) crystal. The slit setting for this image was 0.254 mm. Although a direct comparison with Figure 2 cannot be strictly applied, the improved signal-to-noise ratio is apparent.

4.0 BRAGG PLANE MEASUREMENTS OF LiF (200) CRYSTALS

A set of LiF (200) crystals were provided by SNL to determine their suitability for diffraction experiments. Specifically, in this type of application the Bragg planes of the single crystal must be aligned to the optical surface within 0.5°. The configuration shown in Figure 8 was used to measure this effect.

With the image plane well back from the para-focal point, the crystal was rotated around the optical axis. If the crystal axis was aligned with the optical axis, the diffracted image would not move. The maximum shift in the diffraction lines was measured, and the misalignment angle was determined using computer-aided design software. Only one of the eight LiF crystals tested met the misalignment < 0.5° criterion. For some crystals, the angle was large enough to cause the diffraction lines to move out of the field of view.

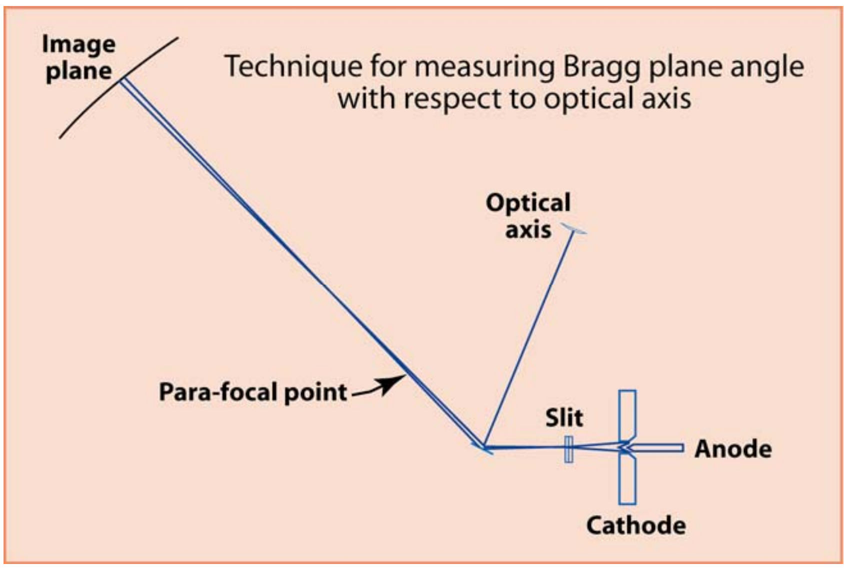


Figure 8. This configuration was used to measure the angle of the Bragg planes with respect to the optical axis. The detector was 10″ past the para-focal point, and 12″ from the crystal.

5.0 TIMING TESTS

The timing of the x-ray pulse is critical to the success of a shocked crystal diffraction measurement because the sample is only shock-loaded for a period of about 100 ns. For the gas gun experiments at SNL TA-IV, the timing will be initiated by a front-surface piezoelectric pin requiring a delay of approximately 600 ns between pin impact and x-ray pulse arrival time at the crystal for the 4-mm crystals that will be used in the experiments.

To obtain the short delay time requirements, the fiber-optic triggering system used on radiography experiments was disabled and replaced with a TTL bypass. The trigger timing configuration is shown in Figure 9. The high-voltage trigger system delay was quite sensitive to the level of the TTL trigger input.

With a 4.0-V TTL input pulse, the system delay from the BNC input on the Marx bank to x-rays on the crystal was 428 ns. Therefore, a 170-ns delay is required from the impact with the piezoelectric front-surface pins to the Marx input.

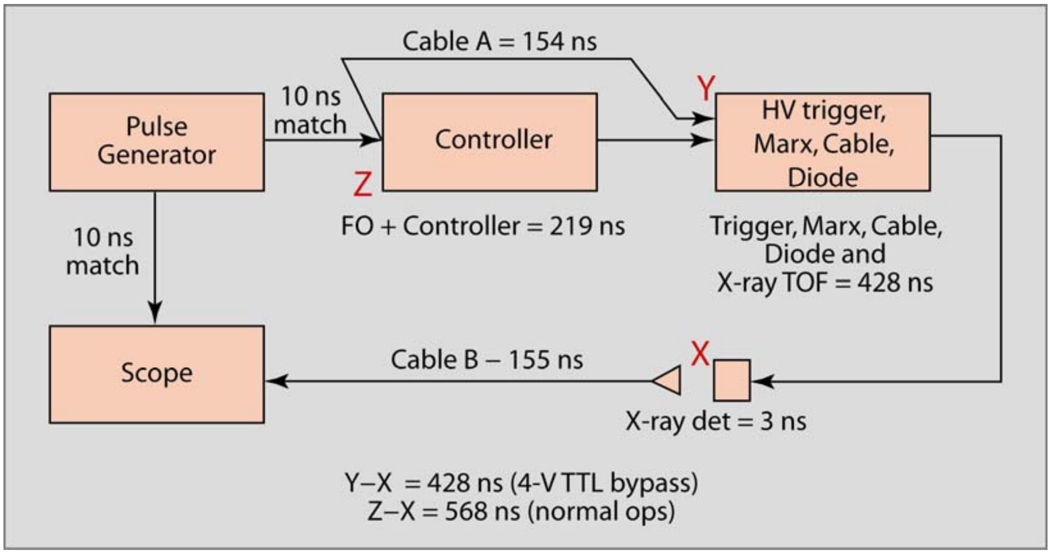


Figure 9. Timing diagram for the x-ray diffraction

6.0 FUTURE WORK

This system will be fielded on the SNL TA-IV gas gun in the second quarter of fiscal year 2007. Approximately two weeks will be required to have the system in place. LAO will assist with the design and fabrication of LiF crystal targets for these gas gun experiments. User-friendly IDL [Interactive Data Language] software for rapid analysis of the line shift data will be developed. A duplicate dynamic x-ray diffraction system, including a Marx bank, cable diode assembly, collimator, and a CCD camera coupled to a fluor, will be built for LAO.