normalization of the diffraction peaks to obtain relative intensities.

The energy analysis system is calibrated with radioactive γ and X-ray sources of precisely known energies. Calibrations to better than 0.1% are possible.

RESULTS

Iron

The energy analysis data are obtained in digital form as counts versus channel number in the multichannel analyzer. The data are also displayed on an oscilloscope trace; this allows constant monitoring of the received signal. Figure 5 shows a computer plot of the digital output for an iron sample at about 25 kb. The first six lines are labeled. When the peaks are analyzed on an expanded scale, the energy position of a peak maximum can be determined to within ± 0.1%. With these positionings, it was found that the pattern can be indexed to that degree of accuracy, therefore it will be possible to calculate lattice parameters to better than one part per thousand.

The background is seen to be a fairly smooth function of energy with some structure apparent around the (200) peak. This is probably due to scattering from the amorphous boron pressure medium.

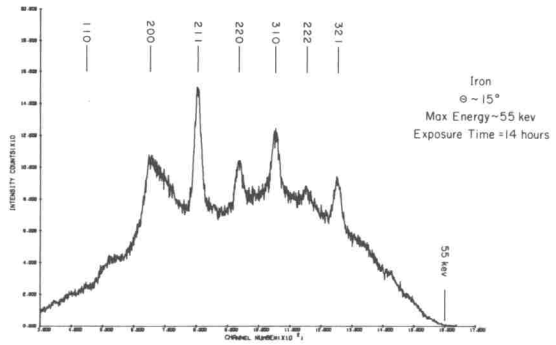

Fig. 5. Computer plot of iron pattern. Energy is linear with channel number (33.3 eV/ch).

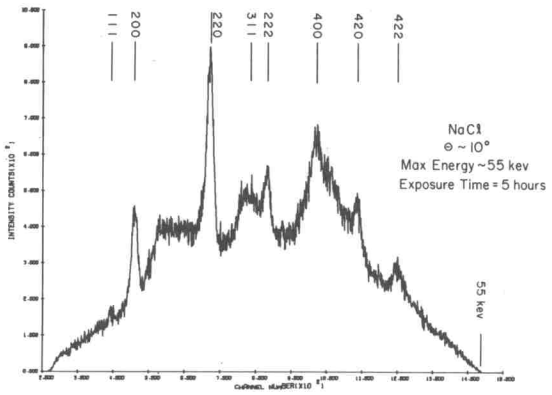

Fig. 6. Computer plot of NaCl pattern. Energy is linear with channel number (37.3 eV/ch).

NaCl

Figure 6 is a pattern for NaCl also taken under a pressure of about 25 kb but at a slightly smaller diffraction angle. Here six lines can be indexed. Indications of the weak (111) and (311) lines can be seen. The same accuracy of 0.1% per line was also found for this pattern. The broad band appearing

between (200) and (220) corresponds to an amorphous boron band observed on films.

DISCUSSION

We believe that the X-ray energy analysis system offers many advantages over the usual dispersive analysis for high pressure systems. First, we believe the method will be more accurate for determining lattice parameters than dispersive techniques. The energy calibration of the analysis system can be made easily with nuclear X-ray and γ-ray sources and can be made before and after each run. Without the use of any peak fitting formula we have been able to position and index peaks to within 0.1%. It is possible that an even higher degree of accuracy could be obtained with more sophisticated analysis techniques. By averaging lattice parameters calculated from a number of lines of one pattern, lattice parameter measurement could be made to an accuracy of a few parts per 10,000. This is at least 5 times better than we have been able to do with measurements of film powder patterns in the split die system.

The speed of making measurements is increased over dispersive techniques for a number of reasons. Because of the higher energies of the X-rays used, absorption in the pressure medium and sample is lower. Neither a monochromator nor filter are needed; there is no attenuation from these sources. The Li drifted Ge detector counter efficiency is essentially 100% over this energy range. The tungsten target can be run at a much higher power level than molybdenum increasing the relative intensity of white versus monochromatic radiation for high pressure work. Finally, the system eliminates wasted time as a continuous display of the data is available on a scope readout. (See Figure 7). If no significant change develops in the pattern (this can be determined in a few minutes) the pressure-temperature conditions can be changed.

With the proper choice of geometry, the signal to noise ratio can be enhanced. If the fan shaped slot is replaced with a collimating groove then the counter will see a greatly reduced portion of irradiated pressure medium and scattering into the counter will be greatly reduced from this source.

A blowup of the die bore, Figure 8, illustrates this point. With a 250 μ entrance groove and a 120 μ diffraction groove both extending the 2" of die radius the amount of irradiated pressure medium that is seen by the detector is very small compared with the sample volume