

estimated to give 5 to 20 kbars pressure increments, until the working limit of the unit is reached (100 to 150 kbars) or until there is evidence of a failure in the sample or diamonds. If a series is completed without a failure, a final pattern is made with all the load removed to check that the fixed diamond has not moved in relation to the film during the pressure cycle.

The x-ray films for samples under pressure require exposures of 100 to 400 hours to Zr-filtered Mo radiation from a Norelco fine focus x-ray tube, or exposures of 15 to 40 hours to radiation from a Jarrell-Ash microfocus unit (used through the courtesy of the Jarrell-Ash Co.). The better films give four to six diffraction lines for each compound and the best films, which are obtained in the Bassett⁵⁰ screw loaded squeezer, have seven or eight lines for each with 2θ as high as 44° . For each compound a separate lattice parameter value is calculated from the d-value of each line. The lattice constant adopted is usually the numerical average of all the individual values. However, on occasion one or more low angle lines may appear grossly inconsistent and are not used.

Data for a typical series of measurements are shown in Fig. II-24, where the lattice constant of $\text{TiO}_{1.0}$ is plotted vs pressure up to 108 kbars. This series was made in two different gas loaded squeezers using portions of the same $\text{TiO}_{1.0}$ -NaCl mix. The values of volume compression ($\Delta V/V_0 \Delta p$) for this and the other compositions studied are listed in Table II-9. Each value is based on the best straight line obtained by a visual fit to all the data points for that composition as shown in Fig. II-24, even though in some cases an individual run could be better described by a curve indicating a decrease in compression at higher pressures. The differences among the tabulated values, which range from 2.6×10^{-4} to $4.0 \times 10^{-4} \text{ kbar}^{-1}$, are probably not significant in view of the experimental errors.

TABLE II-9 VOLUME COMPRESSION OF TiO_x			
x	Pressure Range (kbars)	No. of Runs	$\frac{-\Delta V}{V_0 \Delta p}$ (kbar^{-1})
0.83	0-124	2	3.3×10^{-4}
0.85	0-39	3	2.3×10^{-4}
1.00	0-108	2	3.8×10^{-4}
1.10	0-139	1	2.6×10^{-4}
1.25	0-52.5	1	4.0×10^{-4}

There are two other reports of volume compression measurements on TiO_x , both carried out with tetrahedral-anvil pressure units, which generate nearly hydrostatic pressure. Taylor and Doyle⁵¹ report a value of $5.5(8) \times 10^{-4} \text{ kbar}^{-1}$ for $\text{TiO}_{1.0}$ (with 15 percent vacancies) without giving any experimental details. Iwasaki, *et al.*,⁵² measured the lattice parameter for