alkali halide crystals by X-irradiation is far greater than that which can be accounted for in terms of the free vacancies originally present in the unirradiated crystals.[1] Thus, additional vacancies must be generated during the irradiation. Because a change in number and distribution of vacancies is produced by X-irradiation, it follows that a study of the change in mechanical properties resulting from irradiation with X-rays provides a unique method for obtaining information concerning the interaction between point defects and dislocations.

It has been known for many years that irradiation of alkali halides with X-rays produces marked changes in various mechanical properties of the irradiated crystals. Early experiments by Kusnetzow and Sementzow[2] and by Podaschewsky[3] have shown that the yield-strength of irradiated rock-salt is greater than the breaking strength of unirradiated specimens. Substantial increases in hardness of the alkali halides have also been observed to result from X-irradiation by Li[4] and by Westervelt.[5] These radiation-induced hardening effects are permanent; they are not reversed by optical bleaching. Recent experiments[6] have shown that for unfiltered radiation the hardness increase occurs only in the thin layer of the crystal close to the irradiated surface.

Frankl[7] discovered that irradiation has a marked effect on the internal friction properties of rock-salt, viz. that the amplitude-dependent part of the damping is almost eliminated by a brief irradiation. He found that this effects occurs throughout the entire volume of the crystals studied (in contrast to the hardness increase), and further observed that the amplitude dependence of the damping is not restored by optical bleaching of the crystal. In addition to the changes in decrement, Frankl also noted an increase in the elastic modulus (measured at low-strain amplitudes) of about 0.1% in some irradiated crystals.

According to Frankl, the observed suppression of the damping occurs because positive-ion vacancies which are freed during the irradiation, can pin down the dislocation segments responsible for the damping. (A positive-ion vacancy may be freed from a vacancy pair which dissociates when the negative-ion vacancy of the pair becomes an F center.) In considering this mechanism, Seitz[1] has suggested that it is more likely that the pinning is due to clusters of vacancies of both signs which condense near dislocations during irradiation at room temperature.

Unfortunately, the three mechanical properties which have been investigated in greatest detail—yield-strength, hardness, and internal friction—are relatively difficult to relate quantitatively to the dislocation distribution. The effect of dislocations on the elastic moduli, on the other hand, is predicted by a quantitative theory. If N is the density of dislocation lines (in lines/cm^2 or total line-length per unit volume) which are pinned down at regular intervals to form segments of length l, then, according to Mott[8] and Friedel,[9] the movement of these segments in phase with an applied stress results in a *lowering* of the shear modulus of an isotropic material by an amount $\Delta \mu$ given by

$$\frac{\Delta \mu}{\mu} = \frac{N l^2}{\xi'} \tag{1}$$

where ξ' is a slowly varying function of l having a magnitude near unity. In view of the simplicity of this result, elastic-modulus measurements would appear to provide an excellent means for the investigation of the pinning of dislocations by point defects. The present investigation is directed primarily toward a detailed study of the increase in dynamic modulus which results from X-irradiation of alkali halide crystals. In some of the experiments, internal friction is also measured simultaneously with the dynamic modulus.

2. EXPERIMENTAL METHODS

Modulus Measurements

The modulus measurements are made by the dynamic method, using the three-part composite piezoelectric resonator described by Marx.[10] Two identical quartz crystals are used in this resonator, one which serves to drive the resonator in forced vibration, and the other (the gage crystal) which indicates the resulting amplitude of vibration by means of the voltage developed across its electrodes. The driving crystal is excited by an a.c. signal taken from a variable-frequency oscillator. The specimen, whose length lies in a $\langle 100 \rangle$ direction, is thus driven in the fundamental longitudinal mode. From the measured resonant frequency of the composite resonator, the resonant frequency on the specimen alone, f_s, may be calculated. The elastic constant s_{11} of the specimen crystal is related to the frequency f_s by means of the relation

$$f_s = \frac{1}{2 L_s} \sqrt{\frac{1}{\rho s_{11}}} \tag{2}$$

where L_s is the length and ρ the density of the specimen. (The quantity s_{11}^{-1} may be regarded as the appropriate elastic modulus.) Thus, the relationship between a change in f_s and the corresponding change in elastic constant is

$$2\Delta f_s / f_s = -\Delta s_{11}/s_{11}. \tag{3}$$

The advantage of using the three-part resonator is that its resonant frequency, decrement (ratio of the energy-loss per cycle to the total stored vibrational energy), and maximum strain amplitude of vibration, can all be measured simultaneously. The decrement can be obtained with equal ease regardless of whether the damping is amplitude-dependent or not. From the measured decrement of the composite resonator, the decrement Δ_s of the specimen alone may be obtained. Furthermore, the maximum strain-amplitude in the specimen ϵ_s may be calculated from the voltage developed across the gage crystal. All of the measurements to be described were made with specimens which had a resonant frequency very nearly equal to 85 kc, and which matched the resonant frequency of the quartz crystals to better than 5%.

The frequency of the signal generated by the variable-frequency oscillator can be determined with a precision of better than 1 part in 10^5 by comparison with a quartz-controlled secondary standard. This standard oscillator is in turn monitored by comparison with a reference-frequency broadcast by radio station WWV. The precision attainable in measuring the resonant frequency of the composite resonator depends on two conditions. First, the total decrement of the resonator must be small so that the maximum of the resonance response curve can be found with precision. If the decrement of the specimen is of the order of 10^{-3} or less, its resonant frequency can be determined to within at least 1 part in 10^5 with the present equipment. Second, the composite resonator must be in thermal equilibrium. If the resonator is kept at the ambient temperature, as in the present experiments, its temperature must be measured and appropriate corrections to the observed resonant frequency made for departure of the temperature from that selected as the reference temperature. In the present case, the temperature of the resonator was measured by means of a small thermocouple mounted immediately adjacent to the specimen (the temperature coefficient of the resonant frequency of the specimen is substantially greater than that of the quartz). The practical limit of error in temperature measurement by this method is $\pm 0.01°C$, which corresponds to an uncertainty in resonant frequency of about ± 0.2 parts in 10^5.

Resonant-frequency measurements as a function of temperature were found to fall on a straight line. When the time-rate of change of temperature was less than $0.03°C/min$, the data fell on the same straight line regardless of whether the temperature was increasing or decreasing; when the temperature was changing more rapidly, it did not. Accordingly, the resonator was considered to be in thermal equilibrium with the thermocouple whenever the rate of change of temperature was less than $0.03°C/min$. In order to make the necessary temperature corrections to the observed resonant frequencies, the temperature coefficient of the resonant frequency of the composite resonator was actually measured for every specimen used.

Specimens

Almost all the experiments were carried out with crystals obtained from the Harshaw Chemical Company. The specimens used were free of mechanical defects and lineage structure, as revealed by the flatness of their cleavage planes. NaCl crystals were used in most of the experimental work. The spectroscopic analyses of Harshaw rock-salt made in a number of different laboratories are reported by Duerig and Markham.[11] These crystals generally carry a small amount of divalent impurity such as calcium along with traces of trivalent impurities such as iron.

The natural rock-salt used in some of the experiments was obtained from the Brush Mineral Collection, Yale University, and originated in the Stassfurt deposits. This material had a pronounced lineage structure, making it difficult to cleave into bars suitable for modulus measurements.

Bars of the various alkali halides used were supplied by Harshaw cleaved to the same cross-section as the quartz bars used to make up the composite resonator (0.22 in.²), and were further cleaved to the proper length (about 1.03 in.) to match the frequency of the quartz driver-gage assembly. It was found possible to cleave with an accuracy of about ± 0.003 in. in most cases. Final adjustment of the length was made by grinding the ends of the bars on metallographic polishing paper.

Specimens to be cold-worked were cut oversize by the correct amount and the ends coated with collodion. The plastic deformation was accomplished by compressing the bars lengthwise in a toolmaker's vise. The collodion coating prevents the ends of the bars from splitting during heavy deformation.

Irradiation

The X-rays used for irradiation were obtained from a copper target Coolidge-type tube containing beryllium windows and operating at 39 kV accelerating potential and a tube current of 10 mA. Unfortunately, the power supply for the X-ray tube did not include a voltage regulator, so that normal fluctuations of the voltage taken from the mains resulted in about a 10% fluctuation in the operating conditions of the tube over periods of the order of tens of minutes.