E_G vs T in Fig. 2. Above 300°K, $dE_G/dT = -3.3 \times 10^{-4}$ ev/degree. At lower temperatures dE_G/dT is numerically smaller, as expected.

The room temperature value of E_G (2.86 ev) agrees with the thermal gap value found from measurements of the forward characteristics of p-n junctions⁶ within the experimental error of those measurements.

We believe that the error in $k\theta$ is not more than 0.01 ev. Adding an uncertainty in the energy intercept of about 0.01 ev, we find a total uncertainty in E_G of about 0.02 ev. Above 300°K our values of E_G are all within 0.005 ev of the straight line shown in Fig. 2.

To calculate A by the formula of Bardeen, Blatt, and Hall requires chiefly a knowledge of the electron-phonon matrix element and of the energy denominator which enters into their second-order perturbation treatment of the problem. The latter is known approximately for materials in which direct transitions are observed. In our case we did not see any sudden increase in absorption rate which might be regarded as the onset of direct transitions. We therefore have only a lower limit for the energy denominator. Because of the temperature dependence of the energy gaps, we expect some temperature dependence of the energy denominator and hence of A. Experimentally we found that A decreased slowly from about 3500 at 77°K to about 2500 at 717°K.

The electron-phonon matrix element may be estimated from the mobility if it is assumed that the same phonons are involved in both scattering and absorption. This is unlikely, but we have nevertheless used this estimate for want of a better one together with the lower limit for the energy denominator. The value of Acalculated in this way is approximately three times as large as our experimental value.

The phonon energy used in fitting the data (0.09 ev) is somewhat less than that of the optical phonons responsible for the strong infrared absorption found by Picus.⁷ We estimate from his curve that these energies are about 0.12 ev for the longitudinal and 0.095 ev for the transverse phonon.

⁶ Lyle Patrick (to be published).

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Detection of Directional Neutron Damage in Silicon by Means of Ultrasonic Double Refraction Measurements*

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The presence of oriented regions of displaced atoms (directional radiation damage) produced by a beam of approximately collimated fission neutrons in silicon single crystals has been observed by an ultrasonic double refraction method. The effects observed are anisotropic velocity or modulus changes which do not appear to be accompanied by attenuation changes. The irradiated crystal shows a very pronounced ultrasonic double refraction effect with waves propagated at right angles to the bombardment direction together with the absence of such a double refraction effect when the waves are propagated in the direction of the bombarding neutrons. Separate, independent, ultrasonic velocity measurements indicate that the irradiation has lowered the velocities perpendicular to the bombardment direction in a manner entirely consistent with the double refraction measurements. The observed velocity effects are not accompanied by detectable attenuation changes and are therefore not connected with dislocation damping phenomena.

W HEN fast particles such as neutrons collide with atoms in a crystalline substance, the recoiling atom produces what is commonly called radiation damage in the material. The region of damage may be thought of as an elongated ellipsoid, roughly the length of the recoil range, containing many atoms displaced from normal lattice sites. On the average, these elongated regions of damage should be oriented with their long dimension directed along the path of the bombarding particles. Berman, Foster, and Rosenberg¹ have pointed out that the existence of such long thin regions is indicated by their thermal conductivity measurements. Consequently, in a crystal that has been bombarded with a collimated beam to produce a large number of oriented regions, anisotropic physical properties are to be expected. In what follows, anisotropic elastic properties are of particular interest. Such oriented domains of damage will be referred to as directional damage.

An investigation of fast-neutron radiation effects in silicon has shown that it is possible, by means of highfrequency ultrasonic measurements, to observe the effect of directional damage in well-oriented single crystals.

⁷ E. Burstein and P. H. Egli, in *Advances in Electronics and Electron Physics* (Academic Press, Inc., New York, 1955), Vol. VII, p. 24.

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¹Berman, Foster, and Rosenberg, *Defects in Crystalline Solids* (The Physical Society, London, 1955), p. 321.



FIG. 1. Experimental arrangement for obtaining a partially collimated fast neutron flux.

With high-frequency ultrasonic methods² one can observe changes in the state of the material such as deformation,³ diffusion, internal oxidation,⁴ and precipitation,⁵ and, in the case treated in this paper, a disruption of the crystal lattice by reactor radiation. In order to observe these changes it is necessary to measure attenuation and/or velocity changes as a function of the process to be followed. The mechanisms contributing to attenuation or energy loss include scattering by inhomogeneities, vibration of dislocations, and thermoelastic or heat flow losses. Velocity changes may accompany attenuation changes: for example, in plastic deformation, described in reference 3, where initially the attenuation increased and the velocity decreases during deformation. In such cases the velocity change is connected with dislocation density changes and dislocation loop length changes. On the other hand, velocity changes⁶ may occur where the scattering properties of a material are changed. Such velocity changes are associated with changes in the purely elastic modulus, as distinct from changes in the velocity, or modulus, from dislocation effects. One can regard velocity changes arising from scattering as resulting from distortion or perturbation of the wave front as the wave passes through the change in the elastic medium offered by the scatterer.

The preceding remarks are included here because it is necessary to explain later why the observed velocity changes, unaccompanied by a detectable attenuation change, point to a damage mechanism that appears to rule out any important participation of dislocations.

The measurement of the velocity of a pulse of ultrasonic vibrations is usually accomplished by the direct measurement of the time intervals between pulses in a pulse echo pattern resulting when a single pulse is allowed to propagate back and forth between parallel faces of a specimen. There are many factors which make high precision velocity measurements (0.01% or better) quite difficult to achieve.

A new method of measuring velocity differences induced in single crystals is that of ultrasonic double refraction which was, so far as the writers are aware,

first observed⁷ in a single crystal of germanium in looking for anisotropic effects of dislocations. The effect observed in germanium was, however, caused by imperfect orientation. Ultrasonic double refraction is discussed in detail in reference 7; it is sufficient to note that ultrasonic double refraction is in all principal features like double refraction with electromagnetic waves. Transverse or shear waves are polarized in such a way that the incident wave breaks up into two component transverse waves which, polarized at right angles to one another, propagate separately with different velocities. The fact that pulsed ultrasonic waves are used and the presence of multiple echoes make the interpretation of the results somewhat different from the usual optical or microwave situation.

In order to produce the anisotropic radiation effect described above, the arrangement shown in Fig. 1 was devised. This device was located at the edge of the uranium lattice of the Brookhaven graphite reactor, in the reflector, at a point where the cadmium ratio



FIG. 2. Silicon cube showing relation of numbered faces to bombardment direction. Each direction is an equivalent (100) direction.

² R. L. Roderick and R. Truell, J. Appl. Phys. 23, 267 (1952). ³ Hikata, Truell, Granato, Chick, and Lücke, J. Appl. Phys. 27, 396 (1956).

C. F. Ying and R. Truell (to be published).

⁵ Teutonico, Granato, and Truell, Phys. Rev. 103, 832 (1956). ⁶ C. F. Ying and R. Truell, Acta Metallurgica 2, 374 (1954).

⁷ P. C. Waterman and L. J. Teutonico, J. Appl. Phys. (to be published).



FIG. 3. Pulse echo patterns using face 3 shown in Fig. 2. (a) Pattern with double refraction present—polarization OA in Fig. 2.
(b) Pattern with polarization OB or OC in Fig. 2. In each case the picture at the right shows an expanded time scale.

was approximately 50. Thus any sample located some distance from the end of the natural uranium slug will be subjected to the randomly oriented fast flux and to the fission neutrons arising from the slug. While it is difficult to calculate, or measure, the "directional flux," a conservative calculation indicates that there are two to eight times as many neutrons coming from the slug, i.e., "collimated," as are randomly oriented. The maximum half-angle for the collimated beam is approximately 17° while the average half-angle is roughly 10° . A determination of the directional, as compared to the random, fast-neutron flux in the silicon cube shown in Fig. 1 is extremely difficult. One method of calculation gave $8.7 \times 10^{16} nvt$ as the total collimated flux and 3.4×10^{16} nvt as the randomly oriented epicadmium fast flux. It is believed that this particular calculation gives a low ratio of directional flux to random flux.

In this experiment two silicon crystals were used; they were 15 mm on edge and the faces were (100), (010), (001) faces. The two cubes were initially identical as far as attenuation and velocity measurements were concerned. The faces of these two crystals were oriented to within a few minutes of the axes and faces mentioned above. Accurate orientation is necessary because double refraction effects can arise from very small misorientation.⁸ One of the two cubes was exposed to fast-neutron irradiation directed predominantly along one of the (100) axes by using the bombardment arrangement shown in Fig. 1.

In the irradiated crystal there was no detectable change in ultrasonic attenuation in any direction with compressional or with transverse waves. It was found, however, that there was a strong ultrasonic double refraction present as a result of the irradiation. The effect was observed in a pronounced way only at right angles to the direction of bombardment and was essentially absent when examined along the direction of bombardment. It is much easier to observe this double refraction effect than it is to measure the individual velocities to the required accuracy, especially when the effect is small.

The observation of ultrasonic double refraction obviously requires the use of transverse waves, and the following discussion is concerned entirely with transverse waves. With crystal faces identified as shown in Fig. 2, some of the experimental results are shown in Figs. 3 and 4. In Fig. 3(a) the pulse echo pattern is that

⁸ P. C. Waterman, "The effect of orientation on plane wave propagation in single crystals," Metals Research Laboratory, Brown University Technical Report (unpublished).



FIG. 4. Pulse echo patterns using face 2 shown in Fig. 2. (a) Pattern with double refraction present—polarization OA in Fig. 2. (b) Pattern with polarization OB or OC in Fig. 2. In each case the picture at the right shows an expanded time scale.

observed with the direction of propagation normal to the direction of bombardment and for all planes of polarization except those containing the direction of bombardment and the normal to the direction of bombardment. Figure 3(b) shows how the echo pattern becomes exponential for the two situations when the plane of polarization contains the direction of bombardment or the normal to the direction of bombardment. The results shown were observed with an ultrasonic frequency of 135 megacycles per second.

The nonexponential pattern of Fig. 3(a) contains dips or minima because the initial wave pulse entering the crystal from the transducer has a polarization OA[see Fig. 2(a)], and because the components OB and OC do not propagate with equal velocities. The resultant OA rotates as the component waves progress [see Fig. 2(b)] and as the phase relationship between them changes. At certain points the component waves OB and OC will have a phase difference such that their resultant will have rotated so that the transducer (still in original position OA) will see only a small part of the rotated resultant or none at all if OA is originally at 45° to *OB* and *OC*. The main result is similar in all principal features to optical double refraction if the single transducer is considered as both the polarizer and the analyzer; there are, as pointed out above, some differences because of the need to use pulses rather than continuous waves and because of the multiple reflections or echoes. The differences which arise as a result of the use of pulse methods are discussed in detail in reference 7.

Figure 2(a) shows schematically the orientation of the irradiated cube. Figure 3, just discussed, was obtained by coupling the transducer to face 3 of the crystal while Fig. 4 shows very much the same result when face 2 was used to couple the ultrasonic energy into the silicon crystal.

When, however, the quartz transducer was coupled to face 1 with the ultrasonic beam in the same direction as that of neutron bombardment, the results were quite different, as shown in Figs. 5(a) and 5(b) for two different polarization planes. There was in this case no plane of polarization where the echo pattern had the minima of Figs. 3 and 4 or where the echo pattern deviated appreciably from an exponential pattern. There was a small effect as the plane of polarization was rotated, and this should be expected since the fast neutrons were not really well collimated.

Two silicon single crystals were used and as mentioned above these were initially identical as far as all ultrasonic measurements were concerned. One crystal was not irradiated so that a comparison could be made with it at any time during the measurements. The echo pattern for the unirradiated crystal was a very



FIG. 5. Pulse echo patterns using face 1 shown in Fig. 2. (a) Pattern with polarization equivalent to OA on faces 2 and 3. (b) Pattern with polarization equivalent to OB or OC on faces 2 and 3. In each case here the echo pattern is nearly but not quite exponential, showing a slight effect caused by bombardment.

good exponential pattern for any of the three directions of propagation and for all planes of polarization.

Both from measurements of the unirradiated cube, and the irradiated cube prior to irradiation, one obtains the normal shear velocity V_0 and modulus G_0 for any plane of polarization of the incident wave. Consider next the difference in velocity between components of the shear waves *OB* parallel to, and *OC* (see Fig. 2), perpendicular to the long axis of the damaged regions. The shear modulus G_{II} is associated with a shear wave of velocity V_{II} , polarized in the *OB* direction, i.e., the long direction of the damage regions and is different from the shear modulus G_{I} , associated with a shear wave of velocity V_{I} , polarized on the *OC* direction, i.e., the short axis of the elongated damaged regions.

The expression for the fractional velocity change when the double refraction effect is induced in the solid is given by

$$\left(\frac{\Delta V}{V}\right)_{\rm d.r.} = \left(\frac{V_{\rm II} - V_{\perp}}{V_0}\right) = \frac{1}{2} \left(\frac{G_{\rm II} - G_{\perp}}{G_0}\right), \qquad (1)$$

where

$$V = \left(\frac{G}{\rho}\right)^{\frac{1}{2}}, \qquad \frac{\Delta V}{V} = \frac{1}{2}\frac{\Delta G}{G}.$$
 (2)

One can also express $(\Delta V/V)_{d.r.}$ as follows:

$$\left(\frac{\Delta V}{V}\right)_{\rm d.r.} = \left\{\frac{\Delta V_{\rm II}}{V_0} - \frac{\Delta V_{\perp}}{V_0}\right\} = \frac{1}{2} \left\{\frac{\Delta G_{\rm II}}{G_0} - \frac{\Delta G_{\perp}}{G_0}\right\}, \quad (3)$$

where

$$\frac{\Delta V_{11}}{V_0} = \left(\frac{V_{11} - V_0}{V_0}\right) = \frac{1}{2} \left(\frac{\Delta G_{11}}{G_0}\right),$$

$$\frac{\Delta V_{\perp}}{V_0} = \left(\frac{V_{\perp} - V_0}{V_0}\right) = \frac{1}{2} \left(\frac{\Delta G_{\perp}}{G_0}\right).$$
(4)

It can be shown (Appendix A and reference 2) that $(\Delta V/V)_{\rm d.r.}$ can also be obtained from the double refraction measurements by means of the relation

$$(\Delta V/V)_{\rm d.r.} = 1/(2\nu t_T), \tag{5}$$

where ν is the ultrasonic frequency and t_T the time to the first true node (see Appendix A) which is obtained by the measurement of a time t_A to the first apparent node as observed in pulse echo patterns such as those in Figs. 3(a) and 4(a). The values of $(\Delta V/V)_{\rm d.r.}$ determined in this way may be compared with the values obtained from the direct velocity measurements by use of Eq. (3).

Using the measured values of ν and t_T in the last equation, one finds

$$(\Delta V/V)_{\rm d.r.}\cong 10^{-2},\tag{6}$$

when properly polarized shear waves are propagated in a direction normal to the bombardment direction.

Such a value of $(\Delta V/V)_{d.r.}$ is relatively easy to measure and since values as low as $10^{-4} - 10^{-5}$ can be measured, smaller radiation effects than present in this silicon cube can presumably be detected by this method.

In addition to measurements of $(\Delta V/V)_{d.r.}$ as discussed above, separate and independent precision velocity measurements were made of V_0 , V_{11} , and V_{\perp} . The values measured are:

$$V_{0} = 5.810 \times 10^{5} \text{ cm/sec},$$

$$V_{11} = 5.719 \times 10^{5} \text{ cm/sec},$$

$$V_{1} = 5.770 \times 10^{5} \text{ cm/sec}.$$
(7)

The corresponding moduli are calculated directly from the above velocities from $G = \rho V^2$ using $\rho =$ density of silicon = 2.328 g/cm^3 .

$$G_0 = 7.858 \times 10^{11} \text{ dynes/cm}^2,$$

$$G_{\perp} = 7.750 \times 10^{11} \text{ dynes/cm}^2,$$
 (8)

$$G_{II} = 7.614 \times 10^{11} \text{ dynes/cm}^2.$$

The velocities of the component transverse waves are less than V_0 , and the moduli of the irradiated silicon are both smaller than the modulus of the original unirradiated silicon. A check on the value measured for $(\Delta V/V)_{\rm d.r.}$ is given by these velocity values:

$$\left(\frac{\Delta V}{V}\right)_{\rm d.r.} = \left(\frac{V_{\rm H} - V_{\perp}}{V_0}\right) = 0.88 \times 10^{-2}, \qquad (9)$$

in excellent agreement with the independently measured value of 10^{-2} from the double refraction measurement. The accuracy of the direct velocity measurements is about 0.01% and the accuracy of the double refraction measurements is probably not better than five or ten percent depending strongly on the range of values of $(\Delta V/V)_{d,r}$ being measured. On the other hand, the double refraction technique can be used to measure values of $(\Delta V/V)_{\rm d.r.}$ down to 10^{-5} and perhaps 10^{-6} while the direct velocity measurement is not of any use below 10⁻³ or 10⁻⁴.

Rough calculations indicate that there are 11×10^{15} recoils per cm³ producing "oriented" regions and 4.3 ×10¹⁵ recoils per cm³ producing "random" regions of damage. Together this should amount to approximately 1.5×10^{18} displaced atoms as calculated from the treatments of Kinchin and Pease9 and Seitz and Koehler.10

Such damage regions, if present in sufficient numbers, will increase the attenuation, and this has been observed in previous experiments where, with considerably greater damage, the attenuation increased by a factor of two or more. The present evidence is that the observed increase in attenuation was a scattering effect rather than a dislocation effect. In the experiments with much longer irradiation and larger damage, it is possible that both dislocation effects and scattering effects are present.

As mentioned above, the velocity changes discussed were not accompanied by any detectable attentuation change. On the basis of the dislocation damping picture, this fact indicates that the effect is not a dislocation effect. Ultrasonic attenuation by dislocation damping has been studied in detail, and one form of the theory¹¹ which has had some success, especially at high frequencies, relates the attenuation α to the elastic modulus G_{el} , the dislocation loop length L, and the dislocation density Λ :

$$\alpha \sim \Lambda L^4 G_{\rm el}.\tag{10}$$

(11)

The same theory relates the difference between the purely elastic velocity and the modified velocity when vibrating dislocations are present.

and

$$(\Delta V/V) \sim \Lambda L^2 G_{\rm el},$$
 (11)

$$(\Delta V/V) = \frac{1}{2} (\Delta G/G_{\rm el}), \qquad (12)$$

where here ΔG is the deviation of the modulus G from the purely elastic modulus G_{el} caused by the presence of dislocations.

The irradiation used in this experiment cannot be expected to alter the dislocation density Λ . Consequently, if there is any dislocation damping change, it must come from a change in the loop length L; and if there is any apparent change in L, it must affect the attenuation α as L^4 while $(\Delta V/V)$ depends only on L^2 . If then there is an easily measurable change in velocity and no detectable attenuation change, one must conclude that this effect is not a dislocation effect. The argument for this conclusion is quite strong if one accepts the theory¹¹ used here. By using values of attenuation known for silicon, it can be shown that for values of Λ and L needed to give a value of attenuation equal to the measured values one cannot get a value of $\Delta V/V$ anywhere near the value measured in this experiment. Even if the entire value of α measured for silicon were due to dislocations, the corresponding value of $\Delta V/V$ would be not more than 0.01% (a factor of 100 smaller than the measured change). Consequently, any change in α of an amount smaller than the total measured α would require a value of $\Delta V/V$ less than 10⁻⁴. Details of a similar argument for germanium are given by Granato and Truell.12

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⁹G. H. Kinchin and R. S. Pease, Repts. Progr. Phys. 18, 1

^{(1955).} ¹⁰ F. Seitz and J. S. Koehler, *Solid State Physics* (Academic Press, Inc., New York, 1956), Vol. 2, p. 307.

 ¹¹ A. Granato and K. Lücke, J. Appl. Phys. 27, 583 (1956).
 ¹² A. Granato and R. Truell, J. Appl. Phys. 27, 1219 (1956).

SUMMARY AND CONCLUSIONS

It has been found that irradiating a single crystal of silicon by roughly collimated fast neutrons produces anisotropic effects which are clearly detectable by ultrasonic double refraction measurements. These effects are elastic modulus changes which do not appear to be associated with dislocations. These modulus changes are observable when properly polarized transverse waves are propagated in a direction normal to the bombardment direction. The magnitude of the double refraction velocity difference observed in this experiment is $(\Delta V/V) \sim 10^{-2}$. Independent precision velocity measurements give a fully consistent velocity change (decrease) together with velocity values and modulus values which the double refraction measurements alone do not give. The sensitivity of the double refraction method to changes is, however, considerably greater than are direct velocity measurements. The above effect was observed with a "collimated" fast flux of approximately $9 \times 10^{16} nvt$, which should produce 11 $\times 10^{15}$ oriented recoils per cm³, superimposed on a randomly oriented flux of $4 \times 10^{16} nvt$ which would give rise to 4.3×10^{15} random recoils per cm³. The experiment shows that there is a pronounced directional effect resulting from the directional bombardment by fast neutrons. Such a directional effect can be attributed to elongated regions of radiation damage having properties different from the surrounding material.

ACKNOWLEDGMENTS

We are very grateful to the Bell Telephone Laboratories and particularly to Dr. David Kleinman for providing us with the silicon cubes used in this experiment. We are indebted to Professor John deKlerk who designed and built the rather special velocity equipment used for the precision velocity measurements. We also acknowledge the valuable cooperation of Dr. G. J. Dienes of Brookhaven National Laboratory for many contributions to this work.

APPENDIX A

Waterman has shown^{7,8} that when two component transverse ultrasonic waves have propagated a distance x in a medium, the resultant displacement resolved in the direction of the transducer vibration is

$$u = \cos\left(\frac{\pi}{\lambda} \frac{\Delta V}{V}x\right) e^{-\alpha x},\tag{13}$$

and this function has zeros at

$$x = \frac{(2n+1)}{2(\Delta V/V)}, \quad n = 0, 1, 2, \cdots.$$
 (14)



FIG. 6. The apparent, i.e., observed, and the true envelope of the ultrasonic pulse echo pattern with the transducer operating both as polarizer and analyzer.

When pulse echo methods are used, it is not in general possible to observe these zeros or nodes directly because the pulses are not sufficiently dense; hence the pulse amplitude envelope can only be observed at a finite number of equally spaced stations on the x axis. Since this spacing is given by the round-trip distance of the pulse in the medium, the entire envelope is not observed experimentally. It turns out, however, that one can get from the observed or apparent envelope of the pulses enough information to determine the true envelope or the envelope which would be observed with many pulses. Figure 6 shows the true and apparent envelopes together with a particular pulse echo pattern. In the figure, S_0 is the distance between observation stations, S_T is the true distance to the first zero or node, and S_A is the apparent distance to the first node. Now

$$\Delta V/V = 1/(2\nu t_T), \tag{15}$$

where t_T is the time of travel corresponding to the distance S_T ; $t_T = S_T/V$. The time t_T is obtained from the times of travel t_A and t_0 corresponding to the distances S_A and S_0 , by means of the relation

$$t_T = \frac{2t_0 t_A}{(2m+1)t_A + (-1)^m (2t_0 - t_A)},$$
(16)

where m is a positive integer (or zero) selected so as to satisfy the relationship

$$\frac{t_0}{(m+1)} \le t_T \le \frac{t_0}{m}.$$
(17)

The times t_0 and t_A are measured experimentally, and t_T is obtained from Eq. (16). Usually several values of m satisfy Eq. (17). The determination of appropriate values of m is carried out by determining those frequencies for which values of $\Delta V/V = 1/(2\nu t_T)$ are constant. For a series of frequencies, it will be found that ν and the corresponding t_T are such that νt_T remains constant; the lowest value of ν for which these relations remain true for m=1 will determine the correct branch or, in other words, the correct t_T .



(b)

FIG. 3. Pulse echo patterns using face 3 shown in Fig. 2. (a) Pattern with double refraction present—polarization OA in Fig. 2.
(b) Pattern with polarization OB or OC in Fig. 2. In each case the picture at the right shows an expanded time scale.



FIG. 4. Pulse echo patterns using face 2 shown in Fig. 2. (a) Pattern with double refraction present—polarization OA in Fig. 2.
(b) Pattern with polarization OB or OC in Fig. 2. In each case the picture at the right shows an expanded time scale.



FIG. 5. Pulse echo patterns using face 1 shown in Fig. 2. (a) Pattern with polarization equivalent to OA on faces 2 and 3. (b) Pattern with polarization equivalent to OB or OC on faces 2 and 3. In each case here the echo pattern is nearly but not quite exponential, showing a slight effect caused by bombardment.