whisker axis.[8] We have used an improved technique[9] in which the lattice twists are determined from the tilt of equatorial Laue spots obtained by utilizing x-rays from a microfocus tube, and recorded on a cylindrical camera coaxial with the whisker. The twist is given by $\alpha = k \tan\rho$, where ρ is the tilt angle of the Laue diffraction image of the whisker and k is a constant determined by camera geometry which is so arranged that the Laue spots are distorted only by twisting of the diffraction planes in the whisker. Effective precautions are taken to avoid any deformation during handling, and motion, misalignment, or strain during exposure.

Our most common observation with whiskers of zinc, copper, iron nickel, manganese, and sometimes silver and palladium grown by chemical reaction[3] is the lack of any detectable lattice twist. Any twist more than 0.01 that expected of a prime axial screw dislocation would have been observed. This does not eliminate a dislocation mechanism for growth of these whiskers because there may be (1) an even number of parallel cooperating screw dislocations with equal magnitudes of Burgers vectors of each sign, or (2) a screw dislocation that climbs or is forced out through the lateral surface of the whiskers.

On the other hand, nine sapphire (α-Al_2O_3) whiskers[9,10] with $\langle 00\cdot 1\rangle$ growth axes were each found without exception to contain an axial screw dislocation with a Burgers vector equal to an integral multiple of the prime Burgers vector in the $\langle 00\cdot 1\rangle$ direction. These dislocations are clearly shown by axial pores about 0.1 to 1.0 μ in diameter as predicted by Frank.[11] The x-ray measurement of the lattice twist used to compute b was confirmed by optical observation of the twist of the $(10\cdot 0)$ facets on the lateral surfaces of the whiskers.

Numerous whiskers of silver, palladium, and copper grown by chemical reaction appeared as single crystals in the form of both helices and nominally straight but twisted prisms. A nominally straight silver whisker and a helical palladium whisker,[12] both with low-index growth axes, showed large axial twists corresponding to screw dislocations with large Burgers vectors. The observed lattice twists could be accounted for by an axial dislocation (or group of dislocations) with a net Burgers vector equal to an integral multiple of atom periodicities in the direction of the growth axis. A single-crystal helical copper whisker showed no lattice twist. Several straight palladium whiskers in the form of tightly twisted prisms showed high-index growth directions close to the $\langle 211\rangle$ direction and lattice twists corresponding to Burgers vectors from zero to unmeasurably large values.

We conjecture that the contortions of these whiskers are due to climb of the dislocation inside the tip of the whisker during growth due to condensation of $<0.1\%$ vacancies that are trapped in the lattice at the tip of the whisker during growth. This forces the point of intersection of the dislocation with the surface to prescribe a helical path which successively crosses the several facets comprising the growing whisker tip, thus periodically changing the effective growth direction. The observed growth forms can be accounted for by variations of the ratio of the radius of gyration of the dislocation terminus to the radius of the whisker, the ratio of the rate of gyration to the rate of growth, and the orientation of the net Burgers vector.

In most of our tin whiskers grown by stress activation no lattice twist was detected, but two had uniform twists corresponding to calculated Burgers vectors for axial dislocations of about 0.2 and 0.4 A. Previously, Treuting[4] reported small lattice twists in tin whiskers that may have been plastically deformed, while Hirsch[5] reported that no twists were detectable.

The hypothesized presence of screw dislocations in some whiskers has been unequivocally confirmed, and some complexities of their behavior have been noted. Further details of our observations will be published later.

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High Negative Nuclear Polarizations in a Liquid*,†

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UPON partially saturating the electron spin resonance (ESR) in a solution of sodium and naphthalene in 1,2-dimethoxyethane, we have observed by means of a simultaneous nuclear magnetic resonance (NMR) experiment, negative nuclear polarizations of the proton spins of up to sixty-five times the equilibrium value. We call this an inverted Overhauser effect.[1,2]

The naphthalene in the solution is ionized by the addition of an electron forming an ionic free radical.[3]

We have detected the ESR of this free radical by means of an amplitude bridge[4] at 50 Mc/sec in a steady field of 17.8 oersteds. The width between maximum and minimum slopes was 2.6 oersteds. In the same steady field, we observed the proton resonance of the solvent ether at 76.8 kc/sec using a modified Pound-Watkins spectrometer. (In the approximately $0.1N$ solution we used, less than one percent of the observed NMR is contributed by the protons of the naphthalene.) Small sine-wave modulation of the steady field at 200 cycles/sec provided the derivative of the absorption curve which was monitored by a phase-sensitive lock-in detector, the output of which is proportional to the nuclear polarization. Simultaneously, the ESR is partially saturated by applying a large 50-Mc/sec voltage across a two-turn coil, within which is located both the nuclear resonance coil and the free-radical sample. The 50-Mc/sec magnetic field H_1 is proportional to the voltage; hence the saturation factor s ($s=\gamma_e^2 H_1^2 T_1 T_2$) is proportional to the square of the voltage. Voltages (V) from zero to 400 volts rms were used corresponding to values of s from zero to about one-quarter. A plot of the experimental values of nuclear polarization as a function of V^2 is shown in Fig. 1. A represents the signal on the lock-in detector; A_0 the value of A with zero 50-Mc/sec voltage applied. A/A_0 is the amount of nuclear polarization achieved. The effect of a very small degree of saturation of the ESR is to depolarize the nuclei. Higher saturation levels of the ESR cause the proton polarization to increase in the negative direction, corresponding to a larger number of nuclei in the upper energy state. This situation has some of the characteristics of a negative absolute temperature. However, as has been pointed out by several observers,[5] the Overhauser effect is not an equilibrium process and hence the concept of negative temperature is not thermodynamically applicable to it.

When negative polarization of the protons is achieved, the detection of the NMR is not by absorption but by stimulated emission, suggesting the possibility that the Overhauser effect could be used for a low-noise molecular amplifier.

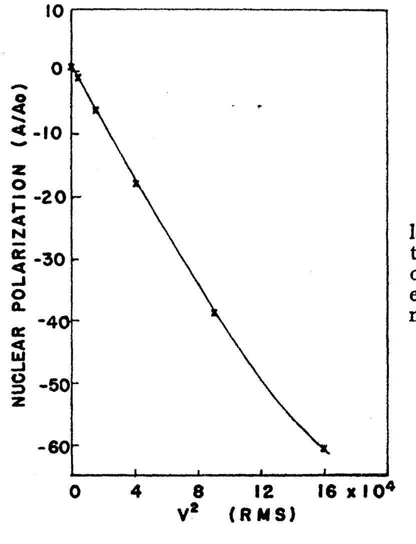

FIG. 1. Nuclear polarization as a function of the degree of saturation of the electron spin resonance.

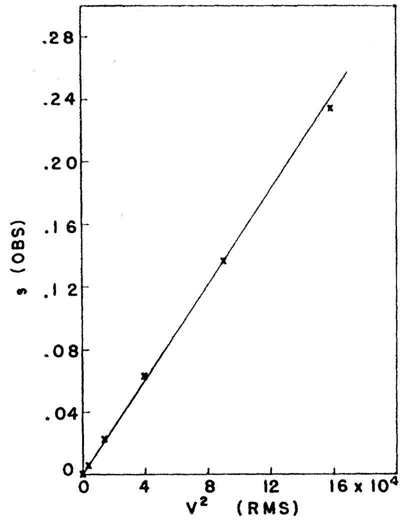

FIG. 2. Observed saturation factor *vs* square of voltage across electron spin resonance coil.

The nuclear polarization expected is

$$\frac{A}{A_0}=\frac{1}{2}\frac{\gamma_e}{\gamma_n}\frac{s}{1+s}. \tag{1}$$

We call s(obs) the values of s obtained from Eq. (1) when the experimental values of A/A_0 are inserted. A plot of s(obs) against V^2 (Fig. 2) yields a straight line with slope 1.51×10^{-6}. If we assume $T_1=T_2$ for the electrons and insert the value of H_1 at the center of the ESR coil computed from the geometry as a function of V, we get $s=1.58\times10^{-6}\ V^2$, in good agreement with Fig. 2. The fact that the effect is substantially complete for each degree of saturation is in good accord with the assumption that the protons can only relax to the lattice via their magnetic dipolar interaction with the "free" electron. If, for example, an $\mathbf{I}\cdot\mathbf{S}$ interaction dominates, the normal Overhauser effect would be observed. This was the case for sodium dissolved in liquid ammonia, which is the only example of an Overhauser effect in a liquid previously reported.[6]

We expect to make a study of the concentration dependence of the effect.

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† Since submission of this letter, we have learned of somewhat similar results obtained in a different material by Abragam, Cambrisson, and Solomon, Compt. rend. **245**, 157 (1957).

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