# NUCLEAR SPIN-LATTICE RELAXATION TIME IN TIN

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# NUCLEAR SPIN-LATTICE RELAXATION TIME IN TIN

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Résumé.- Les temps de relaxation nucléaire spin-réseau de $^{119}$Sn et $^{117}$Sn on été mesurés entre 20 et 50 mK à l'aide d'un magnétomètre à SQUID en suivant la restauration de l'aimantation longitudinale après une impulsion R. F. La température a été déterminée à l'aide d'un thermomètre à bruit couplé à un SQUID. Les constantes de Korringa ont été estimées d'après les valeurs de $\tau_1$. Nous discutons les possibilités d'application de cette méthode a la thermométrie.

Abstract.- The nuclear spin-lattice relaxation times for $^{119}$Sn and $^{117}$Sn have been measured in the temperature range between 20 and 50 mK with a SQUID magnetometer by watching the recovery of the longitudinal magnetization of the nulear spins after the application of an rf-pulse. The temperature was determined using a SQUID noise thermometer. The Korringa constants were estimated from the $\tau_1$-values. Application of the method for thermometry is discussed.

The standard method for thermometry in the temperature range below 50 mK is presently pulsed NMR on platinum. The NMR thermometer is selfcalibrating through the Korringa relation, which gives the temperature from a measurement of the spin-lattice relaxation time. In small fields at low temperatures this method is, however, affected by magnetic impurities in the platinum, which shorten the relaxation. As an alternative to Pt tin has been suggested /1/. Because of the wide line width ordinary NMR gives a bad signal to noise ratio. A more promising method is SQUID NMR, the usefulness of which we have tested. During this experiment $\tau_1$ in $^{119}$Sn and $^{117}$Sn where measured.

The experimental arrangement is shown in figure 1A. The sample consists of 0.33 g of tin powder, of 99.5 % purity and 6 µm particle size. The toroidal coil form, which contains the sample, is made of.paper and GE varnish. The rf-coil consists of 25 turns of 0.1 mm Cu-wire wound on the paper form. The inductance is 0.85 µH. The steady magnetic field is produced by a superconducting solenoid made of Nb-wire and compensated at both ends to provide a good homogeneity. The current of 3.5 A gives a field of 64 mT, which is well above the critical magnetic field of tin, 30.9 mT. A superconducting switch is used to persist the current in the field coil.

The changes in the flux due to the sample are transferred to the SQUID magnetometer by a flux transformer made of 750 µm diameter Nb-wire. The astatic pair consists of two coils, each of 60 turns, wound on a thin-wall silver tube which acts as a low-pass filter, $f_{3dB}$ = 300 Hz. The tube eliminates rf-coupling and the magnetometer responds only to the nuclear magnetization of the sample.

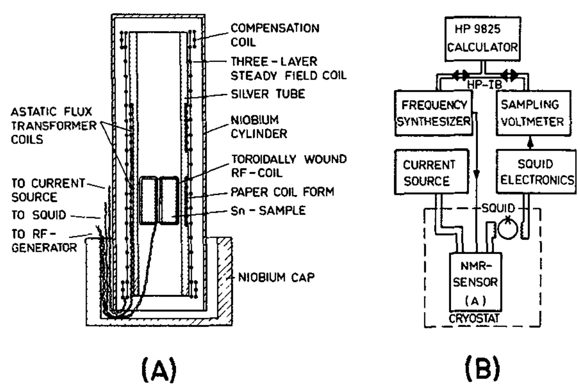


Fig. 1 : The NMR sensor (A) and the block diagram (B).

The sensor is placed in a niobium cylinder that eliminates interference from the surroundings. The cylinder is in the mixing chamber of a dilution refrigerator. The SQUID and the persistent-mode switch of the field solenoid are in the liquid $^4$He bath 15 cm above the mixing chamber.

The block diagram of the complete system is shown in figure 1B. The measurement of $\tau_1$ is controlled by the HP 9825 calculator, which is connected via the HP-IB-interface bus to a sampling voltmeter and a frequency synthesizer. The SQUID is used in the feedback mode which gives a voltage proportional to the flux change.

At the beginning of the measurement the rf-signal is set to the resonant frequency of the sample, 1011 kHz for $^{119}$Sn and 965 for $^{117}$Sn, respectively. After the stabilization of the nuclear magnetization the frequency is shifted up by 20 kHz and the recovery of the magnetization is registered by the magnetometer. This eliminates changes in the electronic background magnetization during the measurement. The output is measured with the voltmeter and the results are transferred to the calculator which fits an exponential through the points. At the end of each measurement the frequency is shifted back to resonance. The procedure is repeated ten times at each temperature. The mean value and the deviation of the ten resulting $\tau_1$-values are calculated and printed. The entire procedure is completely automatized. The Korringa law $\tau_1 T = K$ relates the spin-lattice relaxation time to the temperature which is measured using a SQUID noise thermometer.

The spin-lattice times of $^{119}$Sn and $^{117}$Sn are plotted in figure 2 as functions of the inverse temperature. The open circles represent the averaged experimental data. The solid line is the best fit through the experimental points. The slopes of the lines, 54 msK and 58 msK (±2%), are the Korringa constants of $^{119}$Sn and $^{117}$Sn, respectively. These figures are some 50 % larger than the result of earlier measurements by Asyama and Itaho between 2.0 and 77 K /2/.

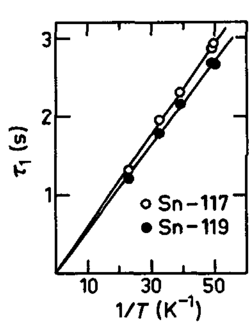


Fig. 2 : $\tau_1$ versus the inverse temperature.

The usefulness of the method for thermometry is determined by three factors : Background noise, background signal, and rf-heating. The background noise is caused mainly by vibrations and flux creep. The noise in the frequency region of the interest was at best $2 \times 10^{-3}\ \phi_o/\sqrt{Hz}$ at 1 Hz. The $^{119}$Sn NMR signal was 0.18 $\phi_o$ at 20 mK. The background signal was mainly caused by electronic impurities in the sample and in the insulation of the copper wire, which gave about 460 $\phi_o$ between 20 and 200 mK. The contribution of the empty magnetometer in the same temperature interval was about 14 $\phi_o$. The rf-heating was caused by eddy current in the tin. The rf-current, which was necessary at 20 mK to completely destroy the nuclear magnetization caused a 5 mK temperature increase in the sample. The $\tau_1$- measurements therefore had to be made at rf power level 20 dB below this value. Even at this level, however, the deviation of the ten $\tau_1$-values measured was only 1 % at 20 mK. Still better results could be obtained by using a finer powder size to cut down the rf-heating.

## References

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/2/ Asyama, K. and Itoh, J., J. Phys. Soc. Japan 17 (1962) 1065.