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High Pressure Phase Transition Studies by Scanning of Nuclear γ-Resonance Absorption

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Untersuchung von Phasenübergängen bei hohen Drucken durch Bestimmung der Resonanzabsorption von γ-Strahlen

Eine Technik, die Kern-γ-Resonanz-Absorption für Phasenübergangsuntersuchungen unter hohen Drucken benutzt, wird beschrieben. Als typische Anwendung werden Curie-Temperatur-Messungen an einer Legierung von 3% Eisen in Palladium bei Drucken von 0 und 60 kbar besprochen.

A technique using nuclear γ-resonance absorption for phase transition studies on solids under high pressures is described. Results of Curie point determinations in a dilute alloy of iron in palladium at pressures of 0 and 60 kbar are given as a typical application.

An earlier paper [1] has demonstrated the value of the thermal scanning technique of nuclear γ resonance absorbtion for high-pressure phase transition studies. In the present paper, improvements upon the technique are described extending the temperature range down to helium temperatures and increasing the accuracy through the use of a constant-velocity motion device and a two-channel digital ratemeter.

The mechanical features of the system are depicted in Fig. 1. Details of the cryostat and of the high-pressure cell are described elsewhere [2]. The sample *1*, which is subjected to high pressures by the mechanical device *2*, is a radioactive source. It contains an isotope which emits γ-rays suitable for nuclear γ-resonance studies of the particular transition. Twin absorbers *3* are mounted on a movable carriage at room temperature. The mode of operation of the system is to drive the carriage back and forth with a constant velocity, v, which corresponds to a γ-resonance line characeristic of one of the phases. The intensity of this absorbtion line, as a function of temperature, pressure or time, is then recorded by the associated electronics and used as an indication of the phase change. The symmetric arrangement reduces the counting time by a factor of two in that it allows measurement at positive and negative velocities simultaneously.

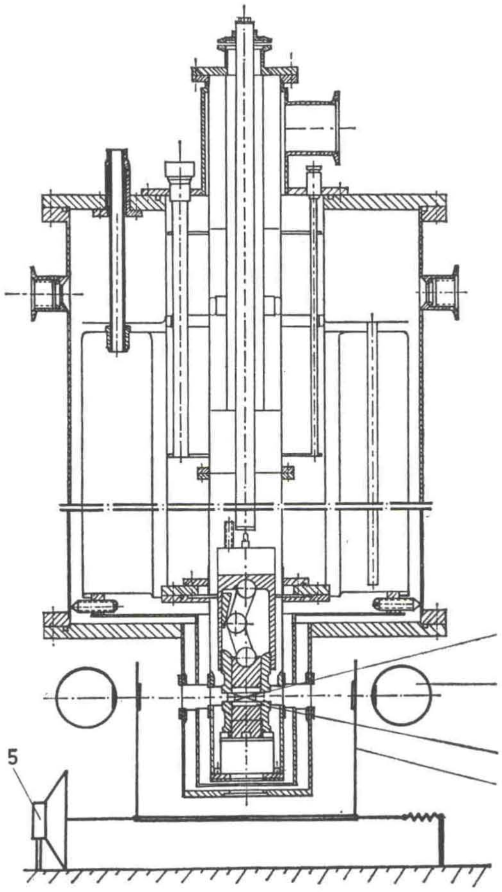

Fig. 1. Nuclear γ-resonance apparatus with high pressure cell. *1* Sample, *2* Anvils, *3* Absorber, *4* Detector, *5* Drive

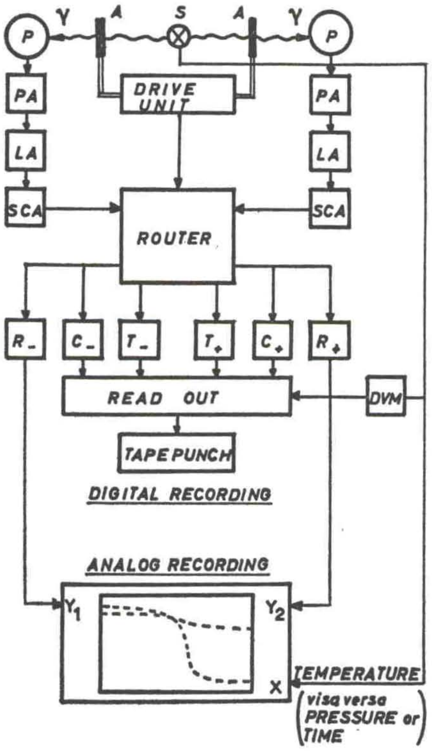

Fig. 2. Block diagram for the scanning technique. For details see text

Fig. 2 shows a block diagram of the electronic components. The γ-rays are emitted from the source, S, pass through the absorbers, A, and are detected by the proportional counters, P. The pulses thus produced are preamplified, PA, amplified, LA, and fed into the single channel analysers, SCA, which select the pulses corresponding to the appropriate nuclear decay. The pulses, now of uniform size and shape, enter a *Router* which channels them into a plus (or minus) count register, C_+ (or C_-), when the absorber in front of the detector is mowing toward (or away from) the source. Information on the relative motion is provided for the *Router* by the loudspeaker *Drive Unit* [3] which controls the motion of the carriage. The *Router* also contains an adjustable dead-time to blank the pulses during the discontinuous change of the velocity. This dead-time is typically less then 5% of the live-time. The live-time is measured by two timers, T_+ and T_-, which insure that the two counters, C_+ and C_-, are open for equal times. At the end of a preselected counting period (typically of the order of 100 seconds), a *Readout* unit reads the totals accumulated in the counters as well as the output of a digital voltmeter, DVM, which monitors the variable coordinate, e.g. temperature. The digital information is recorded in punched tape. Parallel to the digital output is an analog output in the form of an X-Y-recorder which plots the counting rates from the ratemeters, R_+ and R_-, as a function of the variable parameter.

The basic advantage of the two channel "scanning" technique, compared with the taking of full spectra, is the greatly reduced measuring time.

The performance of the system is demonstrated by the determination of the Curie-temperature in an iron-palladium alloy under pressure. The magnetic hyperfine interaction of the ^{57}Fe nuclei with the internal magnetic field of the alloy is reflected in a splitting of the Mössbauer-spectra. At a temperature, T_1, above the Curie-temperature, T_C, the sample is paramagnetic and the spectrum is a single line, Fig. 3. At T_3, well below the transition temperature, the spectrum is split into a resolved sixline pattern. The maximum change in the relative absorption is observed at a velocity v_-, were as at v_+ only minor changes are observed as shown in the right half of Fig. 3. Close to the transition, the splitting collapses and both phases may be present as indicated by the spectrum for T_2.

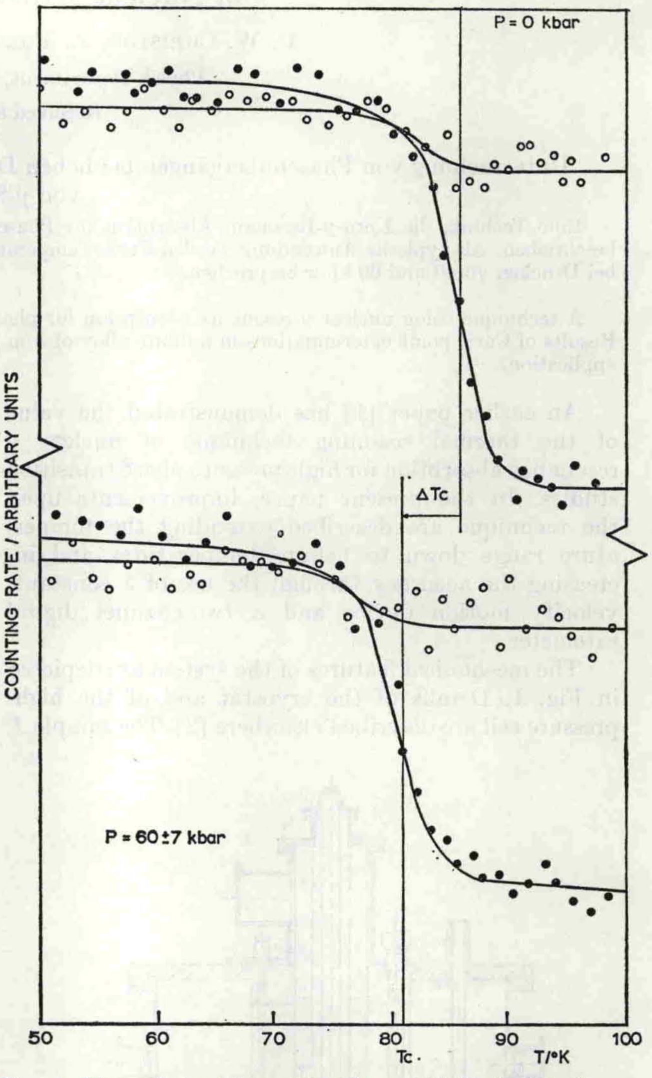

Fig. 4. Thermal scanning curves for a 3% Fe in Pd alloy at 0 and 60 kbar

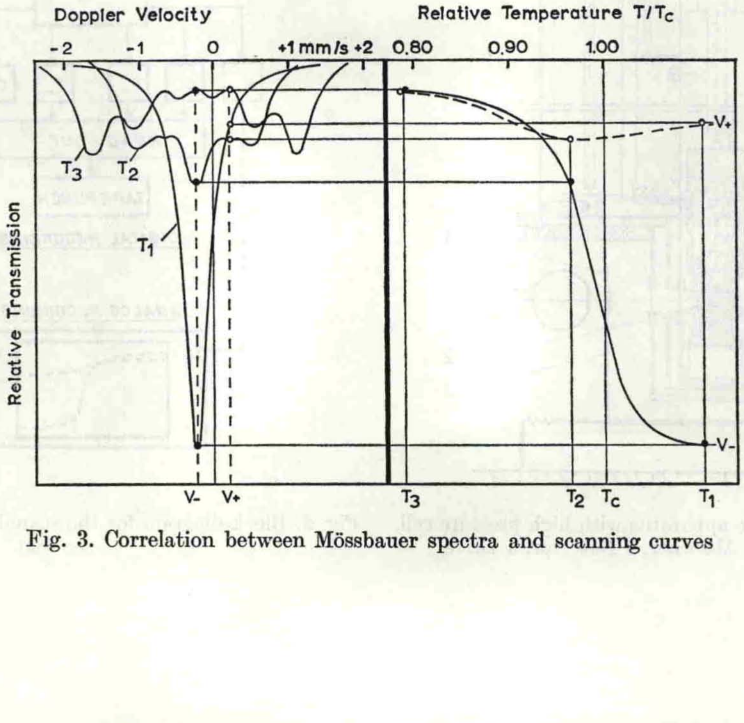

Fig. 3. Correlation between Mössbauer spectra and scanning curves