

SHORT COMMUNICATIONS

Nuclear Magnetic Resonance of Polyvinyl-alcohol (Transition Temperature)

By Kōji TANAKA, Kyōzō YAMAGATA,
Masako YOSHIDA and Minoru ODAJIMA

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In our previous communication¹⁾, the results of the measurements of the nuclear magnetic resonance absorption of protons in polyvinyl alcohol (P. V. A.) fibers from 10°C to 170°C were reported. After that, the measurements were done for the samples of P. V. A. powder and film from -50°C to 200°C. The P. V. A. powder was purified and fractionated. The P.V.A. film was cast from about 2.5% aqueous solution of P. V. A. on a glass plate placed horizontally in an air-thermostat at 50°C and dried at 50°C and 10⁻⁵ mmHg for several days.

The apparatus used is the same as mentioned before, except that the cryo-

stat²⁾ was used to cool the sample to below 20°C. The field intensity was about 3000 gauss.

The width between the points of the maximum slope of the adsorption curve is shown in Fig. 1 as the function of temperature. Below 20°C the widths are almost constant with the value of about 12 gauss, but in the range from 20°C to 50°C, the width decreases gradually with temperature and remains unchanged from

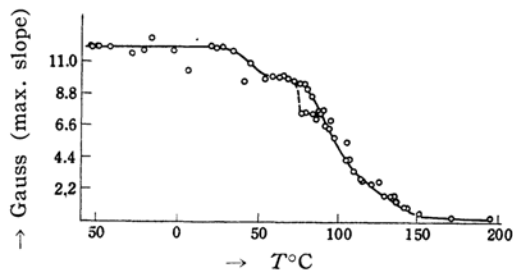


Fig. 1

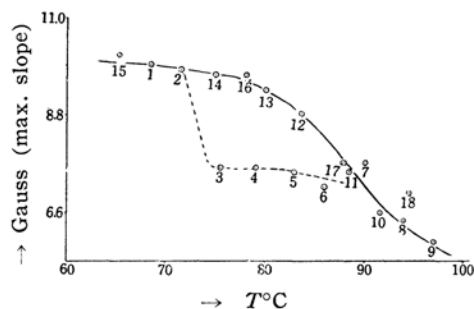


Fig. 2

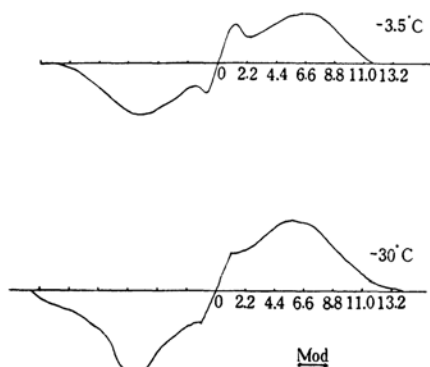


Fig. 3

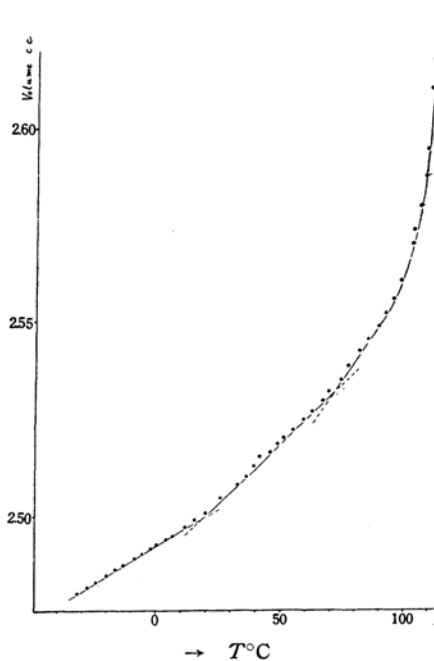


Fig. 4

1) Kōji Tanaka, Kyōzō Yamagata and Shigeyoshi Kittaka, *This Bulletin*, **29**, 843 (1956).

2) H. G. Gutowsky, L. H. Meyer and R. E. McClure, *Rev. Sci. Instr.* **24**, 644 (1953).

50°C to 70°C, then it decreases again in the range between 70°C and 120°C. With respect to this transition at around 70°C, an irreversible hysteresis is observed for the sample with smaller degree of crystallinity which has not been thermally pre-treated. The results are shown in Fig. 2 where the numbers in the figure denote the order of the treatments of the sample. For the sample, which is thermally pre-treated, this hysteresis is not observed and the decreasing of the line width occurs along the upper curve of the hysteresis curve in the figure.

The derivative curves of proton resonance at -3.5°C and -30°C are shown in Fig. 3 and at -50°C the central component is still observed though it is very weak.

In order to compare the results obtained by nuclear magnetic resonance method with those by dilatometric method, volume-temperature curve of P. V. A. film was obtained and its result is shown in Fig. 4.

It is interesting to note that the transition temperatures of about 15°C and 70°C ³⁾ obtained by the dilatometric method coincide with the starting temperatures of the transition obtained by the nuclear magnetic resonance method.

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*Tokyo College of Science,
Shinjuku-ku, Tokyo*

3) Y. Yano. *J. Chem. Soc. Japan*, **73**, 708 (1952).
