was impractical to imbed thermocouples in the crystals for each run, the oven temperature was monitored, and later a calibration curve of crystal temperature *versus* oven temperature was obtained. Although this produces some uncertainty, the reproducibility of the data indicates that changes in thermal contacts were small and had little effect upon the data.

The thermocouple imbedded in the oven, which was of Pt *vs* Pt 10 percent Rh, was checked against a Leeds and Northrup optical pyrometer at 960°C. Using an emissivity of 0.37 for molybdenum and an estimated glass transmission factor of 90 percent, the two readings differed by only 4°C.

In the calibration experiments, the crystal temperature was found both by imbedding 4-mil thermocouples in holes in the crystal and also by using small platinum plates, in thermal contact with the crystal, to transfer heat to the thermocouples; the two methods gave similar results. The temperature gradient along the crystal was measured by two thermocouples, one at the end and one in the middle of the crystal. This gradient will produce an exponential conductivity gradient along the crystal, and the electric field in the crystal will hence be non-uniform, thus affecting the Hall voltage. An appropriate correction factor has been calculated and is plotted in Fig. 3.

PHYSICAL REVIEW VOLUME 87, NUMBER 3 AUGUST 1, 1952

The Coherent Neutron Scattering Cross Sections of Nitrogen and Vanadium*

S. W. PETERSON AND HENRI A. LEVY
Chemistry Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee

(Received April 16, 1952)

Neutron diffraction measurements on vanadium nitride, vanadium carbide, and potassium azide show the coherent neutron scattering cross section of nitrogen to be 11.1± 0.3 barns, positive phase and that of vanadium to be 0.029±0.01 barn, negative phase.

THE coherent neutron scattering cross sections of nitrogen and vanadium have been evaluated from neutron diffraction patterns of vanadium nitride VN and confirmed by similar data from potassium azide KN_3 and vanadium carbide VC. Vanadium nitride was prepared by the reaction of pure V_2O_5 with NH_3 gas at 1100°C for 6 to 8 hours. Neutron diffraction patterns with neutron wavelength 1.16A were obtained from samples sealed in aluminum cells shortly after preparation. The technique for making neutron diffraction measurements will be described elsewhere.

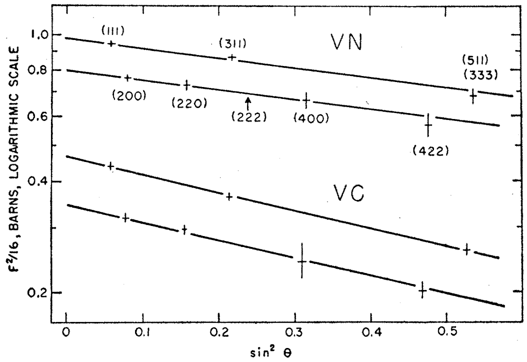

FIG. 1. A plot of observed neutron diffraction values of $F^2/16$ against $\sin^2\theta$ for vanadium nitride and vanadium carbide. The vertical lines indicate the estimated precision of the individual values.

VN crystallizes with the rocksalt structure, for which structure factors are of two types, corresponding to even-index and odd-index reflections. Integrated reflections of three odd-index and four even-index reflections were measured and converted to absolute structure-factor values by comparison with the (111) reflection from nickel powder, for which the coherent cross section was taken as 13.4 barns.[1] The (311) reflection was corrected for contamination by (222) by computing the intensity of the latter from its interpolated structure factor. The resulting values of $F^2/16$ are plotted in Fig. 1 against $\sin^2\theta$. Extrapolation of each family to zero angle yields values proportional to the squares of the structure factors corrected for the effect of temperature and zero-point motion in the crystal and equal, respectively, to the sum (even reflections) and difference (odd reflections) of the scattering amplitudes of V and N. It is immediately apparent that the amplitude of vanadium is of negative sign, opposite to that of nitrogen which is known to be positive.[1] Values of the coherent amplitudes f and cross sections σ yielded by the data are the following:

For nitrogen:

$$f_N = 0.940 \pm 0.015, \qquad \sigma_N = 11.1 \pm 0.3 \text{ barns.}$$

For vanadium:

$$f_V = -0.048 \pm 0.010, \qquad \sigma_V = 0.029 \pm 0.01 \text{ barn.}$$

---

* This work was performed for the AEC.

[1] C. G. Shull and E. O. Wollan, Phys. Rev. 81, 527 (1951).

Experimental uncertainties indicated are rough probable error estimates taking into consideration the statistical error in counting and the uncertainties in estimating the diffuse background, converting to absolute F^2 values, and extrapolation to zero angle, but not allowing for uncertainty in the nickel value used as standard.

The accuracy of the above values, particularly those for N, is of course dependent on the purity of the VN preparation. Spectrographic examination showed the absence of all but faint traces of common impurities, and similar examination of a rare-earth concentrate coprecipitated with yttrium showed that these elements, if present, total less than 0.02 percent by weight. An x-ray diffraction pattern from the sample immediately after preparation showed only the lines of VN. A straight-forward chemical analysis for vanadium yielded the theoretical content. Since, however, VN preparations are capable of containing VO in isomorphous solid solution, attempts were made to establish the nitrogen content directly. This proved to be difficult and only partially satisfactory because of the extremely slow rate of solution of VN in suitable reagents. A modified Kjeldahl analysis gave a nitrogen content 95 percent of theoretical; this is regarded as only a lower limit because of the possibility of incomplete solution and loss of nitrogen by oxidation during the long digestion of the sample with concentrated H_2SO_4. Further, this analysis was carried out about 3 weeks after preparation, and it was later found that VN samples exposed to air slowly take up oxygen with the formation of V_2O_3. Perhaps the best evidence for purity is the following: (1) The value found for σ_N is essentially the maximum value consistent with the total scattering cross section for nitrogen, 10–11 barns.[1,2] (2) The x-ray spacing of 4.129±0.001A requires a VN content greater than 98 percent, based on previously determined spacings[3] of VN and VO and Vegard's rule. (3) The method of preparation is reported to yield VN of at least 99 percent purity.[4] The values of the scattering amplitudes and cross sections reported above assume a purity of 100 percent.

The sign and magnitude of f_V were confirmed by examination of the neutron pattern from a sample of VC. These data, shown also in Fig. 1 and treated similarly to those of VN, yielded $f_V = -0.046$ in satisfactory agreement. The corresponding value of σ_C is 5.05 barns, in reasonable agreement with previous measurements at this Laboratory.[1] Since the origin and purity of the VC samples are not known, these values are regarded as less trustworthy than those from VN.

TABLE I. Neutron diffraction data[a] from potassium azide.

hkl	obs	calc	*hkl*	obs	calc
110	121	124	211	445	436
002	158	159	202	22	18
200	72	71	220	93	94
112	489	493	310	21	20

[a] The quantities listed are the square of the structure factor multiplied by the multiplicity of the reflection, jF^2. The parameters involved in the calculated values are: $f_K = 0.35$, $f_N = 0.940$, $x_N = 0.133$, $B_K = 1.25A^2$, $B_N = 1.4A^2$ (for all N), and $B_N' = 1.0$ (for end N). For a detailed description of the model see reference 6.

Confirmation of the nitrogen value was obtained by examination of diffraction pattern of KN_3. The crystal structure is well known and involves one structural parameter x_N whose value has been given from x-ray studies[5] as 0.133; it is isotypic with KHF_2 which has recently received a detailed neutron diffraction study with single crystals.[6] Table I presents a comparison of observed and calculated structure factors. To achieve satisfactory agreement, it was found necessary to assign separate temperature factors to K and N and to assign an asymmetric component of temperature motion to the two end nitrogen atoms of the N_3^- group. The same treatment was necessary in KHF_2. Like KHF_2, KN_3 powder samples displayed a strong tendency toward preferential orientation with the *c* axes aligned; this difficulty was overcome for the final data by packing the sample loosely in a rectangular type cell and shaking for some time before the diffraction measurements.

The value 11.1 barns for nitrogen finds confirmation also in a single-crystal neutron diffraction study of the crystal structure of NH_4Cl, which has been reported separately.[7] The value is appreciably larger than one of 9.1 barns given by previous neutron diffraction work[8] on zirconium nitride ZrN, and also by preliminary examination[1] of KN_3 at this Laboratory in which temperature corrections were neglected; however, it is in better agreement with recently reported[9] values obtained by neutron refraction in nitrogen gas (11 and 9.7 barns).

The negative sign found for the amplitude of vanadium is in conflict with observations of total reflection of neutrons from vanadium.[10] A probable source of the discrepancy is an impurity with positive amplitude in the vanadium mirror, possibly an oxide film. A Breit-Wigner type calculation by Hamermesh and Muehlhause[11] from data on the scattering resonance in vanadium at ~2700 ev yields a thermal neutron scattering amplitude of negative sign in good agreement with our value.

---

[2] *Nuclear Data*, National Bureau of Standards Circular No. 499 (1950).

[3] R. W. G. Wyckoff, *Crystal Structures* (Interscience Publishers, Inc., New York, 1951), Vol. I, Chapter III, Table, page 14a.

[4] V. Epelbaum and A. K. Breger, Acta Physicochim. U.R.S.S. **13**, 595 (1940).

[5] L. K. Frevel, J. Am. Chem. Soc. **58**, 779 (1936).

[6] S. W. Peterson and H. A. Levy, J. Chem. Phys. **20**, 704 (1952).

[7] H. A. Levy and S. W. Peterson, Phys. Rev. **86**, 766 (1952).

[8] G. H. Goldschmidt and D. G. Hurst, Phys. Rev. **83**, 88 (1951).

[9] A. W. McReynolds, Phys. Rev. **84**, 969 (1951).

[10] A. W. McReynolds and R. J. Weiss, Phys. Rev. **83**, 171 (1951).

[11] M. Hamermesh and C. O. Muehlhause, Phys. Rev. **78**, 175 (1950).