

of flaws in the theory of either or both instruments may be worth investigating. We are in good agreement with Kao and Kobayashi³ for N₂ and slightly higher for He, while our discrepancies with Michels *et al.*^{19,30,36} are not consistent (both of these being capillary measurements). There are no other dense-gas data sufficiently accurate for useful comparison, but it should be noted that our 1-atm viscosities (based on Table II), except for He, are consistently an average of about 0.5% lower than the bulk of previous work¹⁰; since we deliberately

extended our work to lower densities than before, this can no longer be attributed to extrapolation error. The weight of the evidence suggests the possibility of systematic errors, but is not sufficiently consistent to be conclusive.

ACKNOWLEDGMENTS

We thank Professor J. Kestin and Professor E. F. Greene for helpful discussions, and Mr. B. Pilz for assistance with some of the measurements.

THE JOURNAL OF CHEMICAL PHYSICS VOLUME 51, NUMBER 9 1 NOVEMBER 1969

Crystal Structures of Cubic and Trigonal Yttrium Hypocarbides; A Dimorphically Interphased Single-Crystal*

MASAO ATOJI AND M. KIKUCHI

Chemistry Division, Argonne National Laboratory, Argonne, Illinois 60439

(Received 4 June 1969)

X-ray structure determinations on the cubic YC_x ($x \approx 0.3-0.7$, stable above about 900°C) and the trigonal Y₂C (stable below 900°C) have been carried out using a quenched "single crystal" in which a transient state in the midst of the cubic-to-trigonal transition has been arrested. Our "single crystal" is composed of one cubic and four trigonal single crystals, the trigonal *c* axis being parallel to one of four cubic body-diagonal axes. The cubic structure is of NaCl type with random holes in the carbon-atom sites and with $a = 5.115 \pm (2)$ Å for YC_{0.44}. The ordering of the carbon atoms creates the anti-CdCl₂-type trigonal Y₂C with the yttrium positional parameter $z = 0.2585 \pm (3)$ in the hexagonal-based unit cell, $a = 3.617 \pm (2)$ and $c = 17.96 \pm (1)$ Å, containing three Y₂C formula units. Mo K α x-ray was employed, and consequently the dispersion corrections on the atomic scattering factor of Y amount to 10%-20% of the nondispersion value and are considerably larger than the carbon-scattering factor. The structure analyses with and without the dispersion corrections demonstrate that even these unusually large corrections do not modulate the Y and C positional parameters significantly. The x-ray single-crystal results were further substantiated by the neutron-powder analysis. The present work confirmed unambiguously not only the reported x-ray powder results as well as the neutron-powder data of the isostructural Ho₂C, but also yielded the structural parameters with a much higher accuracy, hence providing detailed insights into the chemical-bonding correlation in the order-disorder transition.

INTRODUCTION

Spedding *et al.*¹ have established the existence of the hypocarbides of heavy rare earths and have assigned a cubic NaCl-type structure with disordered carbon atoms to yttrium hypocarbide based on the x-ray power-diffraction data. The present study was initiated in order to verify the x-ray result using the neutron powder-diffraction technique. The neutron powder pattern exhibited several prominent extraneous reflections, most of which appeared to be indexable by doubling the x-ray unit-cell dimension. A number of structure models were tried out, but none rendered a satisfactory result. Later, accidentally, quite a few good "single crystals" were found in the arc-melted buttons. These crystals were too small for neutron

diffraction, but just right in size for the x-ray experiment. The "single crystal" x-ray diffraction patterns were, however, unusually complex and required extensive data acquisition and processing. Concurrently, Bacchella *et al.*² reported the crystal structure of holmium hypocarbide (Ho₂C) using the neutron-powder method. Their structure, trigonal and ordered, did not immediately explain our x-ray patterns. Eventually, it was found that our "single crystal" consists of both the cubic and trigonal hypocarbides with intricate interphasing axial relations. Based on this two-phase structure, the neutron-powder pattern was successfully interpreted. The powder method does not always lead to a unique solution. The present single-crystal analysis has confirmed not only the reported powder results unambiguously, but also provided the structure parameters with a much higher accuracy.

* Based on work performed under the auspices of the U.S. Atomic Energy Commission.

¹ F. H. Spedding, K. A. Gschneidner, Jr., and A. H. Daane, *J. Am. Chem. Soc.* **80**, 4499 (1958); our terminology "rare-earth" includes yttrium.

² G. L. Bacchella, P. Mériel, M. Pinot, and R. Lallement, *Bull. Soc. Franc. Mineral. Crist.* **89**, 226 (1966); R. Lallement, Centre d'Etudes Nucléaires de Fontenay-Aux-Roses Rapport, CEA-R3043, 1966.