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Acta Cryst. (1987). **B43**, 415-416

On the Structure and Twinning of Monoclinic β -V₂H*

BY R. E. MARSH

*Arthur Amos Noyes Laboratory of Chemical Physics, California Institute of Technology,
Pasadena, California 91125, USA*

(Received 13 February 1987; accepted 28 April 1987)

Abstract

The structure of the monoclinic [$a = 4.4566$ (5), $b = 3.0022$ (2), $c = 4.4760$ (5) Å, $\beta = 95.609$ (8)°, $Z = 2$] form of β -V₂H, formerly described in space group *Cm* [Noda, Masumoto, Koike, Suzuki & Sato (1986). *Acta Cryst.* **B42**, 529-533] is properly described in *C2/m*. The crystal used for the original intensity measurements was surely twinned, across the *ab* plane, so that all reflections of the type $5n, k, l$ were composite; the twin component was about 55% as large as the main crystal. When the intensities were corrected for this twinning, refinement in space group *C2/m* led to an *R* of 0.023 for 668 reflections, compared to 0.080 for the earlier investigation. Despite the improvement in refinement, the H atom could not be located from the X-ray data.

Introduction

The structure of the monoclinic (unstressed) form of β -V₂H was described (Noda, Masumoto, Koike, Suzuki & Sato, 1986; NMKSS) in space group *Cm* with two formula units per cell [$a = 4.4566$ (5), $b = 3.0022$ (2), $c = 4.4760$ (5) Å, $\beta = 95.609$ (8)°]. In this description, the V atoms were placed in two independent sites on a mirror plane and the H atoms were ignored. In terms of atom positions, such an arrangement can be equally well described in *C2/m* with a twofold axis midway between the V atoms. However, NMKSS reported that space groups *C2/m* and *C2* could be ruled out 'on the basis of Hamilton's test' and their reported structure showed very large differences in the U_{ij} terms of the two V atoms which

could, in principle, lower the symmetry to *Cm*. But such differences seemed very surprising, especially in view of the simplicity of the structure. Accordingly, I have reinvestigated the structure and shown that it is properly described in *C2/m*. During the process, it became clear that the crystal used for intensity measurements included a twin component that contributed in a systematic way to many reflections; when the intensities were corrected for this twinning, refinement led to an *R* of 0.023 compared to the 0.080 reported by NMKSS for the *Cm* description.

Experimental

Values of F_o (corrected for extinction) and $\sigma(F_o)$ for 672 reflections were recovered from Supplementary Publication No. SUP 42832, and the starting model in *C2/m* was quickly derived by averaging the coordinates and U_{ij} values reported in Table 3 of NMKSS. Preliminary least-squares refinement led to an *R* of 0.074 - already better than the 0.080 of NMKSS. However, reflections with $h = 5n$ were clearly aberrant: *R* for these 145 reflections was 0.18, compared to 0.04 for the remaining reflections, and their values of $F(\text{obs.})$ averaged about 22% larger than $F(\text{cal.})$. That this effect could be due to twinning was confirmed by the fact that the reciprocal lattice maps onto itself, with an 'index' of 5, if it is reflected across the *ab* plane (or, alternatively, rotated by 180° about the *a* axis): reflections of the type $5n, k, l$ of one lattice fall very nearly on top of $5n, k$ (or $-k$), $-l - n$ of the other.

In order to correct for this apparent twinning, I first refined the structure (including the scale factor) on the basis of the reflections with $h \neq 5n$; *R* for these reflections became 0.0202 and the goodness-of-fit was 1.85. The intensities for those with $h = 5n$ were then adjusted by dividing $F^2(\text{obs.})$ by 1.55 for $h = 0$ and,

* Contribution No. 7554 from the A. A. Noyes Laboratory of Chemical Physics. I thank the National Institutes of Health for financial support (Grant No. GM 16966).

for $h=5$ or 10 , by subtracting $0.55 F^2(\text{cal.})$ of the twin component ($5n, k, -l-n$). The twinning factor of 0.55 was arrived at by trial and error; I estimate its standard uncertainty at about 0.02 . After correction, two reflections showed slightly negative values of F_o^2 ; these were included in the refinement. The weights of the adjusted reflections were reduced by the factor $\frac{1}{4}$ for $h=0$ and by $\frac{1}{16}$ for $h=5$ or 10 - unnecessarily, probably, since the final model shows nearly as good agreement for the adjusted reflections as for the others.

Final least-squares refinement led to an R of 0.0232 for 668 reflections (two had been removed because their twin mates were not recorded). During the last cycles, the 44 reflections with $\sin^2 \theta / \lambda^2 \geq 1.78$ ($\theta \geq 71.5^\circ$ for Mo radiation) were given zero weight since several of them showed large residuals suggesting that the θ - 2θ scan range had not encompassed the entire $K\alpha_1$ - $K\alpha_2$ doublet; if these reflections are deleted from the R index, it becomes 0.0217 . The GOF for 626 reflections is 1.63 , but is artificially small because of the reduced weights assigned to the twinned reflections. Refinement was by full-matrix minimization of $\sum w(F_o^2 - F_c^2)^2$, with $w = [2F_o\sigma(F_o)]^{-2}$.

Attempts were made to locate the H atom, with no success: difference maps (with various $\sin \theta / \lambda$ cut-offs) showed no clear peak, and refinement of an isotropic B for an H atom placed in its logical site at $0, 0, \frac{1}{2}$ (NMKSS) led to an increase to over 10.0 . NMKSS reported similar difficulties in locating the H atom. The problem of locating the H was probably exacerbated by rather severe extinction effects (see SUP 42832).

Acta Cryst. (1987). B43, 416-422

The Further Geometry of Grain Boundaries in Hexagonal Close-Packed Metals

BY FU-RONG CHEN* AND A. H. KING

Department of Materials Science and Engineering, State University of New York at Stony Brook, Stony Brook, NY 11794-2275, USA

(Received 16 January 1987; accepted 28 April 1987)

Abstract

A technique is given for finding partial DSC vectors appropriate to crystals with more than one atom per lattice site. The DSC lattice is made up of vectors that represent displacements of one crystal with

* Now at Department of Materials Science and Engineering, Massachusetts Institute of Technology, Cambridge, MA 02139, USA.

Table 1. *Parameters of the V atom, space group C2/m*

The U_{ij} 's are matrix elements, with units \AA^2 .			
x	0.26608 (3)	U_{11}	0.00607 (5)
y	0	U_{22}	0.00601 (4)
z	0.23373 (3)	U_{33}	0.00582 (4)
		U_{13}	0.00053 (3)

The final parameters are given in Table 1.* They are more precise, by factors of 5 to 10, than those reported by NMKSS for the Cm model; moreover, the U_{ij} values are not only more isotropic than those of NMKSS but, of course, are identical for all V atoms. The general description of the structure is unchanged.

NMKSS noted that their refinement of this structure was probably hampered by the presence of twins and suggested that these twins were 'less than $1 \mu\text{m}$ in width'. The sample had been ground into a sphere 0.178 (3) mm in diameter; that the twins could have survived intact this grinding is evidence that the twinning was very intimate. It is somewhat surprising, then, that the twin component was a minor - rather than an equal - contributor to the diffraction pattern.

* A list of observed and calculated structure factors has been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43937 (4 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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respect to the other that leave the boundary structure shifted, but not complete. A new, rapid method for finding the step vectors associated with perfect DSC dislocations is described. Partial DSC vectors and step vectors for perfect DSC dislocations in hexagonal close-packed crystals are determined. The availability of reactions between lattice partial dislocations and grain boundaries in hexagonal close-packed crystals is also assessed.