

## HIGH-PRESSURE SYNTHESIS OF $\beta$ -W-TYPE $\text{Nb}_3\text{Te}$

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**Abstract-** High-pressure, high-temperature techniques were used to synthesize  $\beta$ -W-type  $\text{Nb}_3\text{Te}$  with a lattice parameter  $a = 5.261 \text{ \AA}$ . This compound represents the first reported  $\beta$ -W-type chalcogenide. The superconducting transition is no higher than  $2.5^\circ\text{K}$ .

### 1. INTRODUCTION

The  $\beta$ -W structure is assumed by certain  $\text{A}_3\text{B}$  compounds where A is an element from groups IVB-VIB and B is an element from groups VIII, IB, IIB or IIIA-VA. About 56 of these compounds have been reported. In three cases ( $\text{Nb}_3\text{In}$ ,  $\text{Nb}_3\text{Bi}$ ,  $\text{Mo}_3\text{Sn}$ ) it was necessary to apply high pressure to obtain the  $\beta$ -W structure. An examination of the periodic chart revealed that there were many  $\text{A}_3\text{B}$  combinations for which no  $\beta$ -W-type compound exists. In particular, it was interesting that there were no  $\beta$ -W-type compounds involving either group VIIB or group VIA elements.

The suggestion has been made [4] that it might be interesting to attempt the synthesis of a  $\beta$ -W-type chalcogenide (group VIA) such as  $\text{Nb}_3\text{Te}$ . Attempts to make such compounds by conventional high-temperature techniques had not been successful. High-pressure methods seemed appropriate in view of the large differences in melting temperatures of the constituent elements. Also, rough calculations showed that (for  $\text{Nb}_3\text{Te}$ ) a more favorable radius ratio would be attained with increasing, pressure.

### 2. EXPERIMENTAL

The high-pressure experiments were made in a tetrahedral-anvil apparatus designed by Hall [5,6]. A graphite tube was used as an electrical resistance heater. Inside the graphite tube was a boron nitride sleeve which contained the sample. A detailed account of the experimental procedure has been published elsewhere [7].

The reaction mixture was prepared by combining three parts Nb powder with one part Te powder. The Nb (99.9 per cent pure) was obtained in powder form from United Mineral and Chemical Corporation and the Te (99.99+ per cent pure) in lump form from the American Smelting and Refining Company. The Te was crushed by mortar and pestle, and that portion passing a 200-mesh sieve was used.

X-ray work was done on a G.E. XRD-5 powder diffraction unit with Ni-filtered Cu radiation. Debye-Scherrer photographs were made with a 143.2 mm dia. camera. The Nelson-Riley extrapolation [8] was used to correct for absorption.

### 3. RESULTS

Beta-W-type  $\text{Nb}_3\text{Te}$  was prepared at pressures above about 59 kbar with temperatures in the range  $1330\text{-}1430^\circ\text{C}$ . A rather substantial amount of  $\text{Nb}_5\text{Te}_4$  (tetragonal with  $a = 10.274 \pm 0.002$  and  $c = 3.696 \pm 0.001 \text{ \AA}$ ) was present with the  $\text{Nb}_3\text{Te}$  in each case. This material ( $\text{Nb}_5\text{Te}_4$ ) has been reported previously [9] and our identification is based on data from the cited paper. There was also present in each preparation a small amount of b.c.c. material with a lattice parameter of about  $3.338 \text{ \AA}$ . This material could possibly be excess Nb which has incorporated into its lattice some impurity from the high-pressure environment.‡

Table I. X-ray data for Nb<sub>3</sub>Te

hkl	d <sub>obs</sub> (Å)	d <sub>cal</sub> (Å)	I <sub>obs</sub>	I <sub>cal</sub>
200	2.624	2.631	w	46
210	2.360*	2.353‡	vs	100
211	2.146	2.148	m	100
320	1.453†	1.459‡	m	20
321	1.405	1.406	m	47
400	1.316†	1.316‡	s	16
420	1.179*	1.77‡	m- w	13
421	1.147	1.148	m- w	18
332	1.122	1.122	vw	11
520	0.9760	0.9771	m	19
521	0.9599	0.9607	vw	17
440	0.9293†	0.9302	m	14
600	0.8774†	0.8770	m	11
610	0.8645	0.8651	vw	7
611	0.8534	0.8536	m	28
622	0.7932	0.7933	vw	8
630	0.7843	0.7844	m	45
631	0.7759	0.7759	m	65

\*Interference from b.c.c. material.

† Interference from Nb<sub>5</sub>Te<sub>4</sub> material.

‡ Not used in determination of lattice parameter.

Best results were obtained from experiments at about 67 kbar (maximum pressure for the particular apparatus in use) and 1430°C for 20 min. X-ray data for the β-W phase from such an experiment are given in Table I. The lattice parameter for Nb<sub>3</sub>Te is  $a = 5.261 \pm 0.002$  Å. The lattice parameters of Nb<sub>3</sub>Te from experiments involving other conditions of pressure and temperature each fall within a standard deviation of the above reported value. This indicates that Nb<sub>3</sub>Te (whatever the exact stoichiometry) does not vary in composition.

Magnetization measurements showed that samples containing Nb<sub>3</sub>Te became superconducting at about 2.5°K. Because of the b.c.c. impurity known to be present, no comment on this result is possible at this time.

## REFERENCES

1. Banus M. D., Reed T. B., Gatos H. C., Lavine M. C. and Kafalas J. A., *J. Phys. Chem. Solids* **23**, 971 (1962).
2. Killpatrick D. H., *J. Phys. Chem. Solids* **25**, 1213 (1964).
3. Killpatrick D. H., *J. Phys. Chem. Solids* **25**, 1499 (1964).
4. Matthias B. T., private communication.
5. Hall H. T., *Rev. scient. Instrum.* **29**, 267 (1958).
6. Hall H. T., *Rev. scient. Instrum.* **33**, 1278 (1962).

7. Cannon J. F. and Hall H. T., *Inorg. Chem.* **9**, 1629 (1970).
8. Nelson J. B. and Riley D. P., *Proc. Phys. Soc. (Lond.)* **57**, 160 (1945).
9. Selta K. and Kjekshus A., *Acta Chem. Scand.* **17**, 2560 (1963).

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‡ High-pressure experiments involving only powdered Nb as a sample showed that high temperature runs caused the Nb lattice to expand. Neither the cause of this expansion nor the quantitative relation of amount of expansion versus experimental conditions has been investigated. The expansion was found to occur regardless of the material used to contain the Nb (graphite, BN and Ta were each used).