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X-ray diffraction studies of $NbTe_2$ single crystal

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Abstract. **$NbTe_2$ is a member of transition metal dichalcogenide (TMDC) group. Single crystals of niobium ditelluride ($NbTe_2$) have been grown by a chemical vapour transport technique using iodine as transporting agent. The composition of the grown crystals was confirmed on the basis of energy dispersive analysis by X-ray (EDAX) and remaining structural characterization was also accomplished by X-ray diffraction (XRD) studies. Lattice parameters, volume and X-ray density have been carried out for the grown crystals. The particle size for a number of reflections has been calculated using Scherrer's formula.**

Keywords. **Transition metal dichalcogenides; $NbTe_2$ single crystal; EDAX; XRD.**

1. Introduction

$NbTe_2$ is a member of transition metal dichalcogenides group, which possesses layered structure. The TMDC group attracted many research workers on account of the interesting properties of the compounds of this family (Dimigen *et al* 1979; Christy 1980; Spalvins 1982; Fleischauer 1987). The structural and bonding properties of transition metal tellurides have become important in recent solid-state research (Canadell *et al* 1992; Jobic *et al* 1992a,b; Rouxel and Evain 1994; Van der Lee *et al* 1994; Carteaux *et al* 1995).

The layered structure compound, $NbTe_2$, is one of the typical materials which lead to charge density waves (Van Landuyt *et al* 1975, 1976). The experimental value of electrical resistivity and diamagnetic susceptibility have been reported for high quality single crystals of $NbTe_2$ (Shoichi Nagata *et al* 1993). Raman scattering measurements on the layered structure compound, $NbTe_2$, are also reported with no evidence for a phase transition found for temperatures between 80 K and 420 K (Erdogam and Kirby 1989). The Raman spectra of $NbTe_2$ has also been recorded which shows complicated structure (Hangyo and Nakashima 1984). The single crystals of $NbTe_2$ has been grown using an iodine carrier with suitable gradient between hot and cold ends of the evacuated quartz tube (Nitsche *et al* 1961; Brown 1966). Single crystals of $NbTe_2$ were also grown by the vapour phase transport method with iodine as the transporting agent. The resulting crystals were thin elongated hexagons of typical dimensions $3 \times 5 \times 0{\cdot}1$ mm^3 which could be cleaved using cellophane tape (Levy 1977; Erdogam and Kirby 1989). An attempt has, therefore, been made in the present work to carry out X-ray diffraction study of $NbTe_2$ single crystal.

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2. Experimental

Single crystals of $NbTe_2$ were grown by a chemical vapour transport technique using iodine as transporting agent. A 5 g mixture of Nb (purity: 99·95%, make: Reachim, USSR) and Te (purity: 99·99%, make: Chiti-chem corporation, Baroda, India) was filled in the dried ampoule. Iodine of quantity 5 mg/cc of the ampoule volume was sealed in the thin capillaries and placed in the ampoule as transporting agent. Then the ampoule was sealed at the pressure of 10^{-5} torr. The sealed ampoule was introduced into a two-zone furnace at a constant reaction temperature to obtain the charge of $NbTe_2$. The charge so prepared was rigorously shaken to ensure proper mixing of the constituents and kept in the furnace again, under appropriate condition to obtain single crystals of $NbTe_2$. The charge compositions and growth conditions are summarized in table 1. The chemical composition of the grown samples was confirmed by carrying out EDAX analysis as shown in table 2.

For X-ray diffraction work, several small crystals were finely ground with the help of an agate mortar and filtered through 106 μm sieve to obtain grains of nearly equal size. X-ray powder patterns were recorded on X-ray diffractometer (Phillips, Holland and X"Pert model) using CuK_a radiation.

3. Results and discussion

The shining black opaque single crystals of $NbTe_2$ were obtained by chemical vapour transport (CVT) technique. The crystal structure of $NbTe_2$ is monoclinic with the space group $C2/m$. The X-ray diffractograms obtained for $NbTe_2$ is shown in figure 1. The pattern consists of well-defined sharp diffraction lines, indicating good crystallinity of the specimen. Figure 2 shows the energy dispersive spectra of $NbTe_2$ single crystal. The values of lattice

Table 1. Growth parameters of $NbTe_2$ single crystals using chemical vapour transport technique.

Initial material			Ampoule dimension		Temperature distribution		Physical characteristics of crystals			
Source	Weight (g)	Transporting agent	Length (mm)	ID (mm)	Hot zone (K)	Cold zone (K)	Growth time (h)	Plate area (mm^2)	Thickness (mm)	Colour
$NbTe_2$	10	I_2	250	22	1173	1123	216	6	0·02	Black opaque

Table 2. Weight % of elements taken for growth and obtained from EDAX analysis.

	$NbTe_2$	
Weight (%) of elements	Nb	Te
Taken	26·7	73·3
from EDAX	30·3	69·7

Table 3. Crystallographic data of $NbTe_2$ single crystals grown using chemical vapour transport technique.

Parameter	Brown (1966)	Present work
a (Å)	19·39	19·24
b (Å)	3·642	3·69
c (Å)	9·375	9·30
Unit cell volume $(Å)^3$	–	473·95
X-ray density (g/cm^3)	–	4·867

parameters *a*, *b* and *c*, unit cell volume (*V*) and X-ray density (*r*) determined from the X-ray diffractograms in figure 1 are presented in table 3. Values obtained by Brown (1966) have also been listed for comparison. The X-ray data for $NbTe_2$ was used for the estimation of particle size using Scherrer's formula (Srivastava and Avasthi 1986),

$$t = \frac{K l}{b \cos q},$$

where *t* is the crystallite size as measured perpendicular to the reflecting plane, *K* the Scherrer constant whose value is taken to be unity assuming the particles to be spherical, *l* the wavelength of X-ray radiation, *b* the half intensity width measured in radians and *q* the Bragg angle. (*h k l*) values corresponding to prominent reflections, *d*-values, half width, peak intensities and particle size for $NbTe_2$ single crystal are shown in table 4.

4. Conclusions

Single crystals of $NbTe_2$ were grown by chemical vapour transport (CVT) technique. The EDAX and XRD studies give confirmation that $NbTe_2$ single crystals are stoichiometrically perfect and from XRD analysis it is concluded that structure of $NbTe_2$ is monoclinic with space group *C*2/*m*. The values of parameters *a*, *b* and *c* are very well matched with the similar work done by Brown.

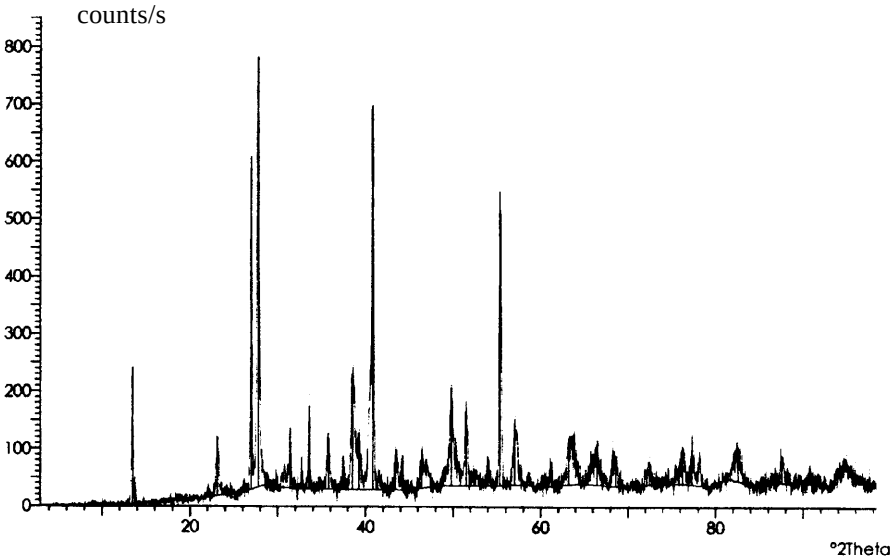

Figure 1. X-ray diffractogram of $NbTe_2$ single crystal.

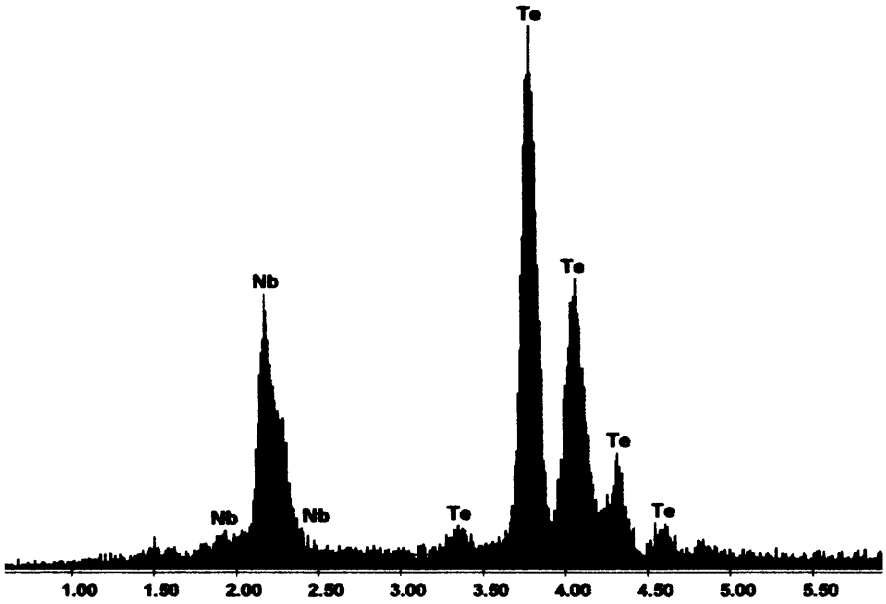

Figure 2. Energy dispersive spectra of $NbTe_2$ single crystal.

Table 4. X-ray diffraction data for $NbTe_2$ single crystal.

(*h k l*)	*d*-spacing (Å)	Peak width (°2*q*)	Relative intensity (%)	Peak intensity (counts/s)	Particle size (Å)
(200)	6·6020	0·12	31·84	239·14	740·62
(002)	3·3123	0·12	77·42	581·50	756·29
(210)	3·2171	0·24	100·00	751·13	378·79
(420)	1·6151	0·24	14·32	107·54	418·43
(610)	1·9569	0·36	6·77	50·87	266·71
(004)	1·6595	0·12	68·56	514·96	830·43
(014)	1·5131	0·30	5·07	38·12	341·83

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