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Evidence of a centre of symmetry: redetermination of $Ni_{2.60}Te_2$ from single-crystal data

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Key indicators: single-crystal X-ray study; T = 293 K; mean σ(Te–Ni) = 0.001 Å; disorder in main residue; R factor = 0.025; wR factor = 0.060; data-to-parameter ratio = 10.5.

The crystal structure of the title compound, nickel telluride, with composition $Ni_{2.60}Te_2$, has been the subject of a previous investigation based on X-ray powder data, when a slightly different composition of $Ni_{2.58}Te_2$ was determined [Gulay & Olekseyuk (2004). *J. Alloys Compd*, **376**, 131–138]. In contrast to the previous refinement in the space group $Pmc2_1$, the redetermination from single-crystal data reveals a centre of symmetry and the structure was refined in the space group *Pnma* with improved precision for the atomic coordinates and interatomic distances. The structure can be described as a $c \times a \times (3a)^{1/2}$ distorted orthorhombic variant of the hexagonal $Ni_{1.10}Se_{0.16}Te_{0.74}$ structure. All atoms are situated on mirror planes.

Related literature

For the previous structure refinement of the title compound from powder data, see: Gulay & Olekseyuk (2004). For the $Ni_{1.10}Se_{0.16}Te_{0.74}$ structure, see: Haugsten & Røst (1972). For crystallographic tools, see: Spek (2003).

Experimental

Crystal data

$Ni_{2.60}Te_2$	V = 333.91 (12) Å^3
M_r = 407.85	Z = 4
Orthorhombic, *Pnma*	Mo $K\alpha$ radiation
a = 12.380 (2) Å	μ = 31.39 mm^{-1}
b = 3.9192 (8) Å	T = 293 (2) K
c = 6.8818 (13) Å	0.14 × 0.09 × 0.04 mm

Data collection

Kuma KM-4 with CCD area-detector diffractometer	2788 measured reflections
Absorption correction: numerical *CrysAlis RED* (Oxford Diffraction, 2007)	335 independent reflections
T_{min} = 0.051, T_{max} = 0.323	329 reflections with $I > 2\sigma(I)$
	R_{int} = 0.068

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.025$	32 parameters
$wR(F^2) = 0.060$	$\Delta\rho_{max}$ = 1.23 e Å^{-3}
S = 1.10	$\Delta\rho_{min}$ = −1.45 e Å^{-3}
335 reflections	

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2007); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2007); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2005); software used to prepare material for publication: *publCIF* (Westrip, 2007).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2152).

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supplementary materials

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Evidence of a centre of symmetry: redetermination of $Ni_{2.60}Te_2$ from single-crystal data

L. D. Gulay, **M. Daszkiewicz** and **A. Pietraszko**

Comment

The crystal structure of the binary $Ni_{2.58}Te_2$ compound has been investigated recently using X-ray powder diffraction data (space group $Pmc2_1$, a = 3.9089 (2) Å, b = 6.8627 (3) Å, c = 12.3400 (6) Å; Gulay & Olekseyuk, 2004). We have now redetermined the crystal structure of this compound by means of single-crystal X-ray diffraction data and present the results here.

The composition $Ni_{2.60}Te_2$ and the unit-cell parameters of the single-crystal study are very similar to those of the powder refinement. However, the centrosymmetric space group *Pnma* was determined for the title compound in contrast to the non-centrosymmetric space group $Pmc2_1$ determined for the powder study. Nevertheless, the topologies and interatomic distances of both centrosymmetric and non-centrosymmetric models are very similar. The structure can be described as a close-packed arrangement of Te atoms with a stacking sequence of the layers as –ABAC–. The Ni atoms partially occupy octahedral and tetrahedral interstices of the Te sublattice. The unit cell and coordination polyhedra of the Ni atoms are shown in Fig. 1. In an alternative description, the structure of the compound can be viewed as a $c\times a \times (3a)^{1/2}$ distorted orthorhombic variant of the hexagonal $Ni_{1.10}Se_{0.16}Te_{0.74}$ structure (space group $P6_3/mmc$, a = 3.836 (1) Å, c = 12.24 (1) Å; Haugsten & Røst, 1972).

Experimental

The sample with composition $Ni_{56.5}Te_{43.5}$ was prepared by fusion of the elemental constituents (Alfa Aesar, > 99.9 wt. %) in an evacuated silica ampoule. The synthesis was performed in a tube furnace with a heating rate of 30 K/h and a maximum temperature of about 1370 K. The sample was kept at this temperature for 4 h. Afterwards it was cooled slowly down to 850 K with a rate of 10 K/h and annealed at 850 K for another 240 h. Then the sample was quenched in cold water. The obtained black crystals had a prismatic habit and maximal lengths of 0.2 mm.

Refinement

The site occupancy factors for Ni2 and Ni3 were constrained (s.o.f. = 0.8) according to the employed composition of the sample. Results of single-crystal reinvestigation of $Ni_{2.60}Te_2$ agree well with those reported on the basis of the powder diffraction study, but with improved precision on atomic coordinates and interatomic distances. Space group *Pnma* was confirmed with *PLATON* (Spek, 2003) and no additional symmetry elements were found. The highest peak and the deepest hole in the final Fourier map are found 1.78 Å and 1.05 Å, respectively, from atom Te1.

Figures

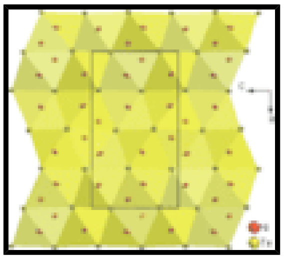

Fig. 1. The crystal structure of $Ni_{2.60}Te_2$ viewed along the *b* axis and displayed with displacement ellipsoids at the 50% probability level.

nickel telluride

Crystal data

$Ni_{2.60}Te_2$

$M_r = 407.85$

Orthorhombic, *Pnma*

Hall symbol: -P 2ac 2n

$a = 12.380$ (2) Å

$b = 3.9192$ (8) Å

$c = 6.8818$ (13) Å

$V = 333.91$ (12) Å^3

$Z = 4$

$F_{000} = 707$

$D_x = 8.113$ Mg m^{-3}

Mo *K*α radiation
λ = 0.71073 Å

Cell parameters from 329 reflections

θ = 3.4–25.0°

μ = 31.39 mm^{-1}

$T = 293$ (2) K

Prism, black

0.14 × 0.09 × 0.04 mm

Data collection

Kuma KM-4 with CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

Detector resolution: 1024x1024 with blocks 2x2, 33.133pixel/mm pixels mm^{-1}

$T = 293(2)$ K

ω scans

Absorption correction: numerical
CrysAlis Red (Oxford Diffraction, 2007)

$T_{min} = 0.051$, $T_{max} = 0.323$

2788 measured reflections

335 independent reflections

329 reflections with $I > 2\sigma(I)$

$R_{int} = 0.068$

$\theta_{max} = 25.0°$

$\theta_{min} = 3.4°$

$h = -14 \rightarrow 12$

$k = -4 \rightarrow 4$

$l = -8 \rightarrow 8$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.025$

Secondary atom site location: difference Fourier map

$w = 1/[\sigma^2(F_o^2) + (0.0225P)^2 + 4.6665P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$wR(F^2) = 0.060$

$S = 1.10$

335 reflections

32 parameters

Primary atom site location: structure-invariant direct methods

$\Delta\rho_{\max} = 1.23$ e Å^{-3}

$\Delta\rho_{\min} = -1.45$ e Å^{-3}

Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0020 (3)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	$U_{\rm iso}$*/$U_{\rm eq}$	Occ. (<1)
Ni1	0.05235 (10)	0.2500	0.4203 (2)	0.0152 (4)	
Ni2	0.14310 (14)	0.2500	0.0802 (3)	0.0185 (4)	0.80
Ni3	0.15349 (14)	−0.2500	0.5956 (3)	0.0191 (5)	0.80
Te1	0.25343 (5)	0.2500	0.42149 (10)	0.0131 (3)	
Te2	0.00373 (5)	0.2500	0.78163 (10)	0.0146 (3)	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0131 (7)	0.0139 (7)	0.0186 (8)	0.000	0.0020 (6)	0.000
Ni2	0.0139 (9)	0.0235 (9)	0.0182 (10)	0.000	0.0043 (7)	0.000
Ni3	0.0109 (9)	0.0182 (9)	0.0283 (11)	0.000	−0.0046 (8)	0.000
Te1	0.0095 (4)	0.0123 (4)	0.0174 (5)	0.000	−0.0001 (2)	0.000
Te2	0.0159 (5)	0.0117 (4)	0.0162 (4)	0.000	−0.0007 (3)	0.000

Geometric parameters (Å, °)

Ni1—Te1	2.4894 (14)	Ni2—Te2$^{\rm vi}$	2.6831 (19)
Ni1—Te2$^{\rm i}$	2.5007 (10)	Ni2—Te1	2.717 (2)
Ni1—Te2$^{\rm ii}$	2.5007 (10)	Ni2—Te2$^{\rm i}$	2.8370 (13)
Ni1—Ni3$^{\rm i}$	2.551 (2)	Ni2—Te2$^{\rm ii}$	2.8370 (13)
Ni1—Te2	2.5582 (16)	Ni3—Ni2$^{\rm vii}$	2.521 (3)
Ni1—Ni1$^{\rm i}$	2.5928 (16)	Ni3—Te1$^{\rm vii}$	2.5214 (19)
Ni1—Ni1$^{\rm ii}$	2.5928 (16)	Ni3—Ni1$^{\rm i}$	2.551 (2)

Ni1—Ni2	2.596 (2)	Ni3—Te1	2.6090 (13)
Ni1—Ni3^{iii}	2.6197 (14)	Ni3—Te1^{viii}	2.6090 (13)
Ni1—Ni3	2.6197 (14)	Ni3—Ni1^{viii}	2.6197 (14)
Ni2—Ni3^{iv}	2.521 (3)	Ni3—Te2^{viii}	2.9860 (15)
Ni2—Te1^{v}	2.5834 (12)	Ni3—Te2	2.9860 (15)
Ni2—Te1^{iv}	2.5834 (12)		
Te1—Ni1—Te2^{i}	106.22 (4)	Ni2^{vii}—Ni3—Te1^{viii}	60.45 (4)
Te1—Ni1—Te2^{ii}	106.22 (4)	Te1^{vii}—Ni3—Te1^{viii}	101.05 (5)
Te2^{i}—Ni1—Te2^{ii}	103.18 (6)	Ni1^{i}—Ni3—Te1^{viii}	117.01 (5)
Te1—Ni1—Ni3^{i}	177.72 (8)	Te1—Ni3—Te1^{viii}	97.37 (6)
Te2^{i}—Ni1—Ni3^{i}	72.47 (4)	Ni2^{vii}—Ni3—Ni1^{viii}	117.27 (6)
Te2^{ii}—Ni1—Ni3^{i}	72.47 (4)	Te1^{vii}—Ni3—Ni1^{viii}	128.90 (4)
Te1—Ni1—Te2	103.43 (5)	Ni1^{i}—Ni3—Ni1^{viii}	60.18 (5)
Te2^{i}—Ni1—Te2	118.35 (4)	Te1—Ni3—Ni1^{viii}	125.24 (7)
Te2^{ii}—Ni1—Te2	118.35 (4)	Te1^{viii}—Ni3—Ni1^{viii}	56.86 (3)
Ni3^{i}—Ni1—Te2	78.85 (6)	Ni2^{vii}—Ni3—Ni1	117.27 (6)
Te1—Ni1—Ni1^{i}	119.91 (6)	Te1^{vii}—Ni3—Ni1	128.90 (4)
Te2^{i}—Ni1—Ni1^{i}	60.26 (4)	Ni1^{i}—Ni3—Ni1	60.18 (5)
Te2^{ii}—Ni1—Ni1^{i}	133.51 (8)	Te1—Ni3—Ni1	56.86 (3)
Ni3^{i}—Ni1—Ni1^{i}	61.23 (5)	Te1^{viii}—Ni3—Ni1	125.24 (7)
Te2—Ni1—Ni1^{i}	58.08 (5)	Ni1^{viii}—Ni3—Ni1	96.84 (7)
Te1—Ni1—Ni1^{ii}	119.91 (6)	Ni2^{vii}—Ni3—Te2^{viii}	129.67 (5)
Te2^{i}—Ni1—Ni1^{ii}	133.51 (8)	Te1^{vii}—Ni3—Te2^{viii}	84.40 (5)
Te2^{ii}—Ni1—Ni1^{ii}	60.26 (4)	Ni1^{i}—Ni3—Te2^{viii}	52.99 (4)
Ni3^{i}—Ni1—Ni1^{ii}	61.23 (5)	Te1—Ni3—Te2^{viii}	169.83 (6)
Te2—Ni1—Ni1^{ii}	58.08 (5)	Te1^{viii}—Ni3—Te2^{viii}	89.91 (2)
Ni1^{i}—Ni1—Ni1^{ii}	98.19 (8)	Ni1^{viii}—Ni3—Te2^{viii}	53.82 (4)
Te1—Ni1—Ni2	64.54 (5)	Ni1—Ni3—Te2^{viii}	113.05 (6)
Te2^{i}—Ni1—Ni2	67.61 (4)	Ni2^{vii}—Ni3—Te2	129.67 (5)
Te2^{ii}—Ni1—Ni2	67.61 (4)	Te1^{vii}—Ni3—Te2	84.40 (5)
Ni3^{i}—Ni1—Ni2	113.18 (8)	Ni1^{i}—Ni3—Te2	52.99 (4)
Te2—Ni1—Ni2	167.97 (7)	Te1—Ni3—Te2	89.91 (2)
Ni1^{i}—Ni1—Ni2	126.70 (6)	Te1^{viii}—Ni3—Te2	169.83 (6)
Ni1^{ii}—Ni1—Ni2	126.70 (6)	Ni1^{viii}—Ni3—Te2	113.05 (6)
Te1—Ni1—Ni3^{iii}	61.35 (4)	Ni1—Ni3—Te2	53.82 (4)
Te2^{i}—Ni1—Ni3^{iii}	167.10 (6)	Te2^{viii}—Ni3—Te2	82.03 (5)
Te2^{ii}—Ni1—Ni3^{iii}	78.60 (4)	Ni1—Te1—Ni3^{iv}	117.01 (6)
Ni3^{i}—Ni1—Ni3^{iii}	119.82 (5)	Ni1—Te1—Ni2^{ix}	119.82 (4)
Te2—Ni1—Ni3^{iii}	70.43 (5)	Ni3^{iv}—Te1—Ni2^{ix}	98.59 (5)
Ni1^{i}—Ni1—Ni3^{iii}	127.56 (9)	Ni1—Te1—Ni2^{vii}	119.82 (4)
Ni1^{ii}—Ni1—Ni3^{iii}	58.59 (5)	Ni3^{iv}—Te1—Ni2^{vii}	98.59 (5)

Ni2—Ni1—Ni3^{iii}	102.02 (6)	Ni2^{ix}—Te1—Ni2^{vii}	98.67 (6)
Te1—Ni1—Ni3	61.35 (4)	Ni1—Te1—Ni3^{iii}	61.78 (4)
Te2^{i}—Ni1—Ni3	78.60 (4)	Ni3^{iv}—Te1—Ni3^{iii}	128.70 (4)
Te2^{ii}—Ni1—Ni3	167.10 (6)	Ni2^{ix}—Te1—Ni3^{iii}	58.08 (6)
Ni3^{i}—Ni1—Ni3	119.82 (5)	Ni2^{vii}—Te1—Ni3^{iii}	127.64 (6)
Te2—Ni1—Ni3	70.43 (5)	Ni1—Te1—Ni3	61.78 (4)
Ni1^{i}—Ni1—Ni3	58.59 (5)	Ni3^{iv}—Te1—Ni3	128.70 (4)
Ni1^{ii}—Ni1—Ni3	127.56 (9)	Ni2^{ix}—Te1—Ni3	127.64 (6)
Ni2—Ni1—Ni3	102.02 (6)	Ni2^{vii}—Te1—Ni3	58.08 (6)
Ni3^{iii}—Ni1—Ni3	96.84 (7)	Ni3^{iii}—Te1—Ni3	97.37 (6)
Ni3^{iv}—Ni2—Te1^{v}	61.47 (4)	Ni1—Te1—Ni2	59.64 (5)
Ni3^{iv}—Ni2—Te1^{iv}	61.47 (4)	Ni3^{iv}—Te1—Ni2	57.38 (6)
Te1^{v}—Ni2—Te1^{iv}	98.67 (6)	Ni2^{ix}—Te1—Ni2	127.93 (4)
Ni3^{iv}—Ni2—Ni1	113.23 (8)	Ni2^{vii}—Te1—Ni2	127.93 (4)
Te1^{v}—Ni2—Ni1	126.56 (4)	Ni3^{iii}—Te1—Ni2	99.12 (5)
Te1^{iv}—Ni2—Ni1	126.56 (4)	Ni3—Te1—Ni2	99.12 (5)
Ni3^{iv}—Ni2—Te2^{vi}	132.43 (8)	Ni1^{i}—Te2—Ni1^{ii}	103.18 (6)
Te1^{v}—Ni2—Te2^{vi}	89.72 (5)	Ni1^{i}—Te2—Ni1	61.65 (4)
Te1^{iv}—Ni2—Te2^{vi}	89.72 (5)	Ni1^{ii}—Te2—Ni1	61.65 (4)
Ni1—Ni2—Te2^{vi}	114.34 (7)	Ni1^{i}—Te2—Ni2^{x}	127.17 (3)
Ni3^{iv}—Ni2—Te1	57.41 (6)	Ni1^{ii}—Te2—Ni2^{x}	127.17 (3)
Te1^{v}—Ni2—Te1	96.67 (5)	Ni1—Te2—Ni2^{x}	126.37 (5)
Te1^{iv}—Ni2—Te1	96.67 (5)	Ni1^{i}—Te2—Ni2^{i}	57.80 (4)
Ni1—Ni2—Te1	55.82 (5)	Ni1^{ii}—Te2—Ni2^{i}	123.34 (5)
Te2^{vi}—Ni2—Te1	170.16 (8)	Ni1—Te2—Ni2^{i}	118.46 (4)
Ni3^{iv}—Ni2—Te2^{i}	128.76 (5)	Ni2^{x}—Te2—Ni2^{i}	98.94 (5)
Te1^{v}—Ni2—Te2^{i}	169.54 (7)	Ni1^{i}—Te2—Ni2^{ii}	123.34 (5)
Te1^{iv}—Ni2—Te2^{i}	86.30 (2)	Ni1^{ii}—Te2—Ni2^{ii}	57.80 (4)
Ni1—Ni2—Te2^{i}	54.59 (3)	Ni1—Te2—Ni2^{ii}	118.46 (4)
Te2^{vi}—Ni2—Te2^{i}	81.06 (5)	Ni2^{x}—Te2—Ni2^{ii}	98.94 (5)
Te1—Ni2—Te2^{i}	91.86 (5)	Ni2^{i}—Te2—Ni2^{ii}	87.38 (5)
Ni3^{iv}—Ni2—Te2^{ii}	128.76 (5)	Ni1^{i}—Te2—Ni3^{iii}	116.64 (5)
Te1^{v}—Ni2—Te2^{ii}	86.30 (2)	Ni1^{ii}—Te2—Ni3^{iii}	54.54 (4)
Te1^{iv}—Ni2—Te2^{ii}	169.54 (7)	Ni1—Te2—Ni3^{iii}	55.75 (4)
Ni1—Ni2—Te2^{ii}	54.59 (3)	Ni2^{x}—Te2—Ni3^{iii}	85.93 (5)
Te2^{vi}—Ni2—Te2^{ii}	81.06 (5)	Ni2^{i}—Te2—Ni3^{iii}	174.17 (5)
Te1—Ni2—Te2^{ii}	91.86 (5)	Ni2^{ii}—Te2—Ni3^{iii}	95.06 (4)
Te2^{i}—Ni2—Te2^{ii}	87.38 (5)	Ni1^{i}—Te2—Ni3	54.54 (4)
Ni2^{vii}—Ni3—Te1^{vii}	65.21 (6)	Ni1^{ii}—Te2—Ni3	116.64 (5)
Ni2^{vii}—Ni3—Ni1^{i}	175.12 (10)	Ni1—Te2—Ni3	55.75 (4)
Te1^{vii}—Ni3—Ni1^{i}	119.66 (8)	Ni2^{x}—Te2—Ni3	85.93 (5)

$Ni2^{vii}$—Ni3—Te1	60.45 (4)	$Ni2^{i}$—Te2—Ni3	95.06 (4)
$Te1^{vii}$—Ni3—Te1	101.05 (5)	$Ni2^{ii}$—Te2—Ni3	174.17 (5)
$Ni1^{i}$—Ni3—Te1	117.01 (5)	$Ni3^{iii}$—Te2—Ni3	82.03 (5)

Symmetry codes: (i) −*x*, −*y*, −*z*+1; (ii) −*x*, −*y*+1, −*z*+1; (iii) *x*, *y*+1, *z*; (iv) −*x*+1/2, −*y*, *z*−1/2; (v) −*x*+1/2, −*y*+1, *z*−1/2; (vi) *x*, *y*, *z*−1; (vii) −*x*+1/2, −*y*, *z*+1/2; (viii) *x*, *y*−1, *z*; (ix) −*x*+1/2, −*y*+1, *z*+1/2; (x) *x*, *y*, *z*+1.

Fig. 1