

SUPPLEMENTARY INFORMATION

PHASE PURE TiCoSb REFINEMENT (90-650 K)

Temperature	90 K	100 K	110 K	120 K
Exposure time (min)	5	5	5	5
# (Observation)	5699	5699	5699	5699
# (reflections)	190	190	190	190
# (parameters)	75	75	75	75
R _F	0.0104	0.0102	0.0104	0.0102
R _{Bragg}	0.0186	0.0185	0.0189	0.0195
R _p	0.0796	0.0803	0.0799	0.0799
R _{wp}	0.101	0.101	0.101	0.102
χ ²	14.5	14.58	14.41	14.5
Scale factor (TiCoSb)	0.0002583 (5)	0.0002590 (5)	0.0002564 (5)	0.0002548 (5)
Zero displament	-0.0122 (3)	-0.0091 (3)	-0.0070 (3)	-0.0062 (3)
a TiCoSb	5.87172 (6)	5.87207 (6)	5.87267 (6)	5.87322 (6)
U TiCoSb	0.014 (2)	0.013 (2)	0.012 (2)	0.012 (2)
V TiCoSb	0.0167 (7)	0.0173 (7)	0.0175 (7)	0.0174 (7)
W TiCoSb	0.00023 (4)	0.00019 (4)	0.00018 (4)	0.00018 (4)
Asym1 TiCoSb	0.0064 (1)	0.0070 (1)	0.0067 (1)	0.0071 (1)
Asym2 TiCoSb	0.0033 (2)	0.0034 (2)	0.0034 (2)	0.0035 (2)
U _{iso} Ti	0.0006 (1)	0.0007 (1)	0.0007 (1)	0.0009 (1)
U _{iso} Co	0.0003 (1)	0.0003 (1)	0.0004 (1)	0.0005 (1)
U _{iso} Sb	0.00019 (8)	0.00025 (8)	0.00035 (8)	0.00041 (8)

Temperature	130 K	140 K	150 K	160 K
Exposure time (min)	5	5	5	5
# (Observation)	5699	5699	5699	5699
# (reflections)	190	190	190	190
# (parameters)	75	75	75	75
R _F	0.0104	0.0104	0.0102	0.0102
R _{Bragg}	0.0191	0.0183	0.019	0.019
R _p	0.0797	0.0796	0.0793	0.0794
R _{wp}	0.101	0.101	0.101	0.101
χ ²	14.61	14.58	14.77	14.77
Scale factor (TiCoSb)	0.0002599 (5)	0.0002618 (5)	0.0002636 (5)	0.0002635 (5)
Zero displament	-0.0061 (3)	-0.0072 (3)	-0.0092 (3)	-0.0092 (3)
a TiCoSb	5.87381 (6)	5.87443 (6)	5.87511 (6)	5.87511 (6)
U TiCoSb	0.012 (2)	0.013 (2)	0.012 (2)	0.012 (2)

V TiCoSb	0.0174 (7)	0.0175 (7)	0.0174 (7)	0.0174 (7)
W TiCoSb	0.00018 (4)	0.00017 (4)	0.00018 (4)	0.00018 (4)
Asym1 TiCoSb	0.0069 (1)	0.0060 (1)	0.0057 (1)	0.0057 (1)
Asym2 TiCoSb	0.0034 (2)	0.0034 (2)	0.0033 (2)	0.0033 (2)
U _{iso} Ti	0.0009 (1)	0.0011 (1)	0.0011 (1)	0.0011 (1)
U _{iso} Co	0.0006 (1)	0.0007 (1)	0.0007 (1)	0.0007 (1)
U _{iso} Sb	0.00049 (8)	0.00058 (8)	0.00066 (8)	0.00066 (8)

Temperature	170 K	180 K	190 K	200 K
Exposure time (min)	5	5	5	5
# (Observation)	5699	5699	5699	5699
# (reflections)	190	190	190	190
# (parameters)	75	75	75	75
R _F	0.00997	0.0102	0.0103	0.00991
R _{Bragg}	0.0178	0.0181	0.0178	0.0171
R _p	0.0791	0.0798	0.0801	0.0795
R _{wp}	0.0995	0.100	0.100	0.0993
χ^2	14.15	14.44	14.55	14.39
Scale factor (TiCoSb)	0.0002633 (5)	0.0002656 (5)	0.0002701 (5)	0.0002724 (6)
Zero displant	-0.0054 (3)	-0.0088 (3)	-0.0123 (3)	-0.0162 (3)
a TiCoSb	5.87651 (6)	5.87723 (6)	5.87793 (6)	5.87863 (6)
U TiCoSb	0.011 (2)	0.012 (2)	0.011 (2)	0.011 (2)
V TiCoSb	0.0178 (7)	0.0175 (7)	0.0181 (7)	0.0179 (7)
W TiCoSb	0.00015 (4)	0.00017 (4)	0.00014 (4)	0.00014 (4)
Asym1 TiCoSb	0.0046 (1)	0.0040 (1)	0.0028 (1)	0.0027 (1)
Asym2 TiCoSb	0.0032 (2)	0.0032 (2)	0.0031 (2)	0.0030 (2)
U _{iso} Ti	0.0012 (1)	0.0013 (1)	0.0015 (1)	0.0016 (1)
U _{iso} Co	0.0009 (1)	0.0009 (1)	0.0011 (1)	0.0013 (1)
U _{iso} Sb	0.00086 (8)	0.00093 (8)	0.00108 (8)	0.00119 (8)

Temperature	210 K	220 K	240 K	260 K
Exposure time (min)	5	5	5	5
# (Observation)	5699	5699	5699	5699
# (reflections)	190	191	191	191
# (parameters)	75	75	75	75
R _F	0.00987	0.00981	0.00952	0.00916
R _{Bragg}	0.0169	0.0158	0.016	0.0157
R _p	0.0798	0.0798	0.0803	0.0799

R _{wp}	0.0992	0.0991	0.0989	0.0988
χ^2	14.5	14.26	14.11	14.09
Scale factor (TiCoSb)	0.0002760 (6)	0.0002736 (6)	0.0002742 (6)	0.0002766 (6)
Zero displacement	-0.0109 (3)	-0.0076 (3)	-0.0051 (3)	-0.0138 (3)
<i>a</i> TiCoSb	5.87939 (6)	5.88009 (6)	5.88123 (6)	5.88237 (6)
U TiCoSb	0.012 (2)	0.012 (2)	0.011 (2)	0.013 (2)
V TiCoSb	0.0175 (7)	0.0175 (7)	0.0178 (7)	0.0173 (7)
W TiCoSb	0.00018 (4)	0.00018 (4)	0.00015 (4)	0.00018 (4)
Asym1 TiCoSb	0.0030 (1)	0.0027 (1)	0.0028 (1)	0.0023 (1)
Asym2 TiCoSb	0.0031 (2)	0.0030 (2)	0.0031 (2)	0.0030 (2)
U _{iso} Ti	0.0017 (1)	0.0018 (1)	0.0021 (1)	0.0023 (1)
U _{iso} Co	0.0013 (1)	0.0014 (1)	0.0018 (1)	0.0020 (1)
U _{iso} Sb	0.00134 (8)	0.00148 (8)	0.00174 (8)	0.00197 (9)

Temperature	280 K	300 K	550 K	600 K
Exposure time (min)	5	5	5	5
# (Observation)	5699	5699	5699	5699
# (reflections)	191	191	191	191
# (parameters)	75	75	75	75
R _F	0.00953	0.00899	0.00967	0.00915
R _{Bragg}	0.017	0.0113	0.0142	0.013
R _p	0.0812	0.086	0.0969	0.097
R _{wp}	0.0993	0.106	0.108	0.112
χ^2	14.31	65.91	9.067	9.63
Scale factor (TiCoSb)	0.0002806 (6)	0.001135 (2)	0.0001689 (4)	0.0001678 (4)
Zero displacement	-0.0136 (3)	-0.0182 (3)	-0.0032 (3)	-0.0069 (3)
<i>a</i> TiCoSb	5.88354 (6)	5.88246 (6)	5.89356 (7)	5.89593 (7)
U TiCoSb	0.011 (2)	0.010 (2)	0.022 (2)	0.026 (2)
V TiCoSb	0.0180 (7)	0.0182 (7)	0.0098 (7)	0.0081 (7)
W TiCoSb	0.00014 (4)	0.00011 (5)	0.00043 (4)	0.00047 (4)
Asym1 TiCoSb	0.0023 (1)	-0.0011 (1)	0.0024 (1)	0.0002 (1)
Asym2 TiCoSb	0.0029 (2)	0.0032 (2)	0.0033 (2)	0.0031 (2)
U _{iso} Ti	0.0026 (2)	0.0028 (2)	0.0054 (2)	0.0058 (2)
U _{iso} Co	0.0022 (1)	0.0024 (1)	0.0048 (2)	0.0053 (2)
U _{iso} Sb	0.00219 (9)	0.0025 (1)	0.0047 (1)	0.0049 (1)

Temperature	650 K
Exposure time (min)	5
# (Observation)	5699

# (reflections)	191
# (parameters)	75
R _F	0.00856
R _{Bragg}	0.0151
R _p	0.100
R _{wp}	0.118
χ^2	10.73
Scale factor (TiCoSb)	0.0001699 (4)
Zero displacement	-0.0102 (3)
<i>a</i> TiCoSb	5.89838 (7)
U TiCoSb	0.029 (3)
V TiCoSb	0.0062 (7)
W TiCoSb	0.00053 (4)
Asym1 TiCoSb	-0.0006 (1)
Asym2 TiCoSb	0.0031 (2)
U _{iso} Ti	0.0062 (2)
U _{iso} Co	0.0056 (2)
U _{iso} Sb	0.0051 (1)

REFINEMENT INCLUDING CoSb (700-1000 K)

Temperature	700 K	750 K	800 K	850 K
Exposure time (min)	5	5	5	5
# (observation)	5699	5699	5699	5699
# (reflections)	692	690	684	686
# (reflections)	78	78	78	78
R _F (TiCoSb)	0.00753	0.00826	0.00842	0.0105
R _{Bragg} (TiCoSb)	0.0129	0.0133	0.0139	0.0155
R _p	0.0978	0.11	0.122	0.123
R _{wp}	0.119	0.128	0.139	0.142
χ^2	10.64	11.92	13.6	13.72
Zero displacement	-0.0130 (3)	-0.0065 (3)	-0.0113 (3)	-0.0074 (3)
scale factor (TiCoSb)	0.0001674 (4)	0.0001627 (4)	0.0001565 (4)	0.0001470 (4)
scale factor (CoSb)	0.0000034 (5)	0.0000132 (7)	0.0000373 (9)	0.000100 (2)
<i>a</i> TiCoSb	5.90103 (7)	5.90379 (7)	5.90621 (8)	5.90866 (8)
U TiCoSb	0.001 (2)	0.001 (2)	0.001 (2)	0.001 (2)
V TiCoSb	0.0308 (6)	0.0314 (6)	0.0328 (7)	0.0322 (7)
W TiCoSb	0.00483 (4)	0.00350 (4)	0.00196 (4)	0.00090 (4)
Asym1 TiCoSb	0.000 (1)	0.000 (1)	-0.001 (1)	0.000 (1)
Asym2 TiCoSb	0.0031 (2)	0.0029 (2)	0.0027 (2)	0.0024 (2)

U_{iso} Ti (TiCoSb)	0.0067 (2)	0.0071 (2)	0.0076 (3)	0.0080 (3)
U_{iso} Co (TiCoSb)	0.0062 (2)	0.0066 (2)	0.0071 (2)	0.0076 (3)
U_{iso} Sb (TiCoSb)	0.0055 (1)	0.0059 (1)	0.0062 (2)	0.0066 (2)
a CoSb	3.89269 (2)	3.90770 (6)	3.91904 (3)	3.92312 (1)
c CoSb	5.33324 (2)	5.31003 (7)	5.21553 (3)	5.21811 (1)
Weight fraction (TiCoSb)	99.7 (3)	98.9 (4)	96.9 (4)	91.7 (4)
Weight fraction (CoSb)	0.28 (4)	1.08 (6)	3.07 (8)	8.3 (2)

Temperature	900 K	950 K	1000 K
Exposure time (min)	5	5	5
# (observation)	5699	5699	5699
# (reflections)	689	689	693
# (reflections)	78	78	78
R_{F} (TiCoSb)	0.0104	0.0113	0.0134
R_{Bragg} (TiCoSb)	0.0167	0.0187	0.0205
R_{p}	0.128	0.136	0.143
R_{wp}	0.148	0.155	0.161
χ^2	14.79	16.11	17.32
Zero displacement	-0.0050 (3)	-0.0132 (3)	-0.0120 (3)
scale factor (TiCoSb)	0.0001426 (4)	0.0001402 (5)	0.0001371 (5)
scale factor (CoSb)	0.000132 (2)	0.000158 (2)	0.000197 (3)
a TiCoSb	5.91129 (8)	5.91350 (9)	5.91561 (9)
U TiCoSb	0.001 (2)	0.001 (3)	0.001 (3)
V TiCoSb	0.0316 (7)	0.0338 (7)	0.0348 (7)
W TiCoSb	0.00031 (4)	-0.00034 (4)	-0.00032 (4)
Asym1 TiCoSb	0.000 (1)	0.000 (2)	0.002 (2)
Asym2 TiCoSb	0.0024 (2)	0.0024 (3)	0.0026 (3)
U_{iso} Ti (TiCoSb)	0.0086 (3)	0.0091 (4)	0.0095 (4)
U_{iso} Co (TiCoSb)	0.0082 (3)	0.0086 (3)	0.0092 (3)
U_{iso} Sb (TiCoSb)	0.0070 (2)	0.0072 (2)	0.0075 (2)
a CoSb	3.92844 (1)	3.93249 (1)	3.93742 (1)
c CoSb	5.22127 (1)	5.22508 (1)	5.22849 (1)
Weight fraction (TiCoSb)	89.0 (4)	86.9 (4)	83.8 (4)
Weight fraction (CoSb)	11.0 (2)	13.1 (2)	16.2 (2)