Refinement of the crystal structure of palladium pentagallide, PdGa₅

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Received June 27, 1996, transferred to 2nd update of database ICSD in 1997, CSD-No. 402535

Source of material: The sample with the nominal composition Pd₅Ga₉₅ was prepared by arc melting of the metals (Ga - 99.999%, Pd - 99.99 %). The sample was remelted in glass carbon crucible in an evacuated quartz tube at 575 K and cooled down slowly to 425 K. According to the phase diagram, the intermetallic phase PdGa₅ coexists with liquid gallium below 453 K. Thus after cooling, the remaining gallium was dissolved in diluted HCl. The binary phase PdGa₅ was identified first by Schubert and coworkers (see ref. 1) and the structure was derived from Weissenberg photographs without refinement (see ref. 2): space group *I4/mcm*, *a* = 6.448 Å, *c* = 10.003 Å, *Z* = 4.

The tetragonal *I4/mcm* structure is a three-dimensional system of bicapped tetragonal antiprisms [PdGa₁₀] (see Fig.) condensed via common corners to columns along [001] and via common edges to layers parallel (001). Eight Ga(1) atoms form the tetragonal antiprismatic fragment of the coordination polyhedron around the Pd atom. Two Ga(2) atoms cap this fragment along [001], forming linear -Ga-Pd-Ga- chains. The important interatomic intra-cluster distances are: *d*(Pd-Ga) = 2.496 Å (2 Ga(2)), 2.697 Å (8 Ga(1)); *d*(Ga(1)-Ga) = 2.828 Å (8 Ga(2)), 2.898 Å (4 Ga(1)), 2.903 Å (4 Ga(1)), 3.480 Å (8 Ga(1)). The shortest intercluster distances in the Ga₄ square are *d*(Ga(1)-Ga(1)) = 2.669 Å, 2.787 Å.

Ga₅Pd, tetragonal, *I4/mcm* (No. 140), *a* = 6.436(6) Å, *c* = 9.990(8) Å, *V* = 413.8 Å³, *Z* = 4, *R*(*F*) = 0.041, *R*_w(*F*) = 0.041.

Table 1. Parameters used for the X-ray data collection

Crystal:	metallic lustre, prismatic, size 0.03 x 0.05 x 0.07 mm
Wavelength:	Mo K _α radiation (0.71073 Å)
μ:	377.93 cm ⁻¹
Diffractometer:	Siemens-Nicolet
Scan mode:	2θ/ω
T _{measurement} :	293 K
2θ _{max} :	65°
N(<i>hkl</i>) _{unique} :	220
Criterion for <i>F</i> _o :	<i>F</i> _o > 4 σ(<i>F</i> _o)
N(<i>param</i>) _{refined} :	12
Program:	CSD

Table 2. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
Pd	4a	0	0	1/4	0.0115(6)	<i>U</i> ₁₁	0.0088(8)	0	0	0
Ga(1)	16f	0.1467(2)	<i>x</i> +1/2	0.1395(2)	0.0175(6)	<i>U</i> ₁₁	0.0158(8)	0.0016(5)	-0.0004(5)	<i>U</i> ₁₃
Ga(2)	4c	0	0	0	0.034(1)	<i>U</i> ₁₁	0.008(1)	0	0	0

References

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