

rungen im Röntgendiagramm festzustellen. Erst bei 120 °C läuft die Rückumwandlung mit merklicher Geschwindigkeit ab. Sie führt direkt zur ursprünglichen Chalkopyritmodifikation zurück. Intermediäre Phasen mit Zinkblende-struktur und statistischer Kationenverteilung, wie sie beim AgInTe_2 ^{1,2} und AgInSe_2 ³ auftreten, wurden nicht beobachtet.

Über das Verhalten von AgGaS_2 , AgGaSe_2 und AgGaTe_2 bei hohen Drucken

KLAUS-JÜRGEN RANGEL, G. ENGERT und ARMIN WEISS

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(Z. Naturforschg., 24b, 1969—1969 [1969]); eingegangen am 6. Mai 1969

AgInS_2 ¹, AgInSe_2 ² und AgInTe_2 ^{3,4} gehen bei hohen Drucken aus der Chalkopyritstruktur mit tetraedrischer Koordination⁵ in dichtere Modifikationen über. Ähnliche Phasenumwandlungen erwarteten wir auch für AgGaS_2 , AgGaSe_2 und AgGaTe_2 , die bei Atmosphärendruck ebenfalls im Chalkopyritgitter kristallisieren⁶.

Die ternären Ausgangsphasen wurden durch Zusammenschmelzen stöchiometrischer Gemenge der binären Komponenten oder der Elemente in evakuierten Quarzampullen dargestellt. Aussehen und Gitterkonstanten der erhaltenen homogenen Produkte stimmten mit den Literaturangaben⁵ überein.

Die Durchführung der Druckversuche in einer Zweistempel-Apparatur wurde bereits an anderer Stelle beschrieben⁷. Abweichend davon wurden bei einigen Experimenten die Druckstempel während der Belastung oszillierend gegeneinander verdreht. Hierbei wirken neben dem hydrostatischen Druck Scherkräfte auf die Probe ein, welche die Umwandlungsgeschwindigkeit stark erhöhen. Die Gleichgewichtsdrücke werden dagegen durch Scherkräfte nur verhältnismäßig wenig beeinflusst⁸.

Nach der Druckbehandlung bei 50 kbar, 500 °C unter Scheren war das Chalkopyritgitter von AgGaS_2 und AgGaSe_2 unverändert erhalten geblieben. Die Pulverdiagramme waren bis auf eine geringfügige Linienverbreiterung mit denen der Ausgangsverbindungen identisch.

Bei AgGaTe_2 konnten im Druckbereich bis zu 50 kbar mehrere Hochdruckphasen beobachtet werden. Hierbei trat ein markanter Einfluß der Scherkräfte zutage. Ein statischer Druck von 20 kbar ohne Scherkräfte verändert bei einer Reaktionstemperatur von 300 °C die Ausgangsmodifikation (AgGaTe_2 -I) nicht. Geht man bei der gleichen Temperatur zu höheren Drucken über, wird das tetragonale Chalkopyritgitter des AgGaTe_2 deformiert. Die a -Achse verkürzt sich nach der Druckbehandlung bei 40 kbar,

Dem Leibniz-Rechenzentrum der Bayer. Akademie der Wissenschaften danken wir für die Bereitstellung von Rechenzeit.

¹ K.-J. RANGEL, G. ENGERT u. A. WEISS, Z. Naturforschg., 24b, 813 [1969].

² K.-J. RANGEL, J. ENGELS u. A. WEISS, Z. Naturforschg., 23b, 1262 [1968].

300 °C von 6,29₄ Å auf 6,20₂ Å, das Achsenverhältnis steigt von $c/a = 1,88_2$ auf $c/a = 1,53_2$ an. Das Elementarzellvolumen der neuen Phase (AgGaTe_2 -I') ist um 2,4% kleiner als das der Ausgangsphase. Tempert man AgGaTe_2 -I' bei Atmosphärendruck und > 200 °C, liegt nach 24 Stdn. wieder die ursprüngliche Chalkopyritphase vor.

Bei der Druckbehandlung unter Scheren wandelt sich AgGaTe_2 -I zwischen 20 und 50 kbar und Reaktionstemperaturen unter 250 °C in ein Gemenge zweier tetraedrisch koordinierter Phasen um. Neben der schon beschriebenen deformierten Chalkopyritphase AgGaTe_2 -I' tritt eine Phase mit Zinkblendenstruktur ($a = 3,94$ Å) auf, die sich bei Atmosphärendruck und 150 °C mit großer Geschwindigkeit in AgGaTe_2 -I' umwandelt. Die Ausgangsmodifikation wird bei > 200 °C zurückgehalten.

Bei einer Reaktionstemperatur von 300 °C unter Scheren tritt bei 10 kbar teilweise, bei 20 kbar vollständige Umwandlung des AgGaTe_2 -I in eine weitere Hochdruckphase ein, deren Röntgendiagramm sich kubisch mit 10,18₃ Å indizieren läßt (AgGaTe_2 -II; Tab. 1). Hier liegt ein bei

hkl	l	$\sin^2 \theta_{\text{beob.}}$	$\sin^2 \theta_{\text{ber.}}^*$
210	10	0,0290	0,0287
211	10	0,0345	0,0344
221, 300	100	0,0519	0,0516
310	50	0,0573	0,0573
311	70	0,0633	0,0631
321	35	0,0802	0,0802
411, 330	30	0,1028	0,1031
420	20	0,1144	0,1146
510, 431	70	0,1489	0,1490
520, 432	50	0,1664	0,1662
521	20	0,1710	0,1719
442, 600	15	0,2058	0,2063
611, 532	15	0,2172	0,2178
621, 540, 443	15	0,2349	0,2349
444	15	0,2723	0,2740
720, 641	15	0,3034	0,3037
721, 633, 552	15	0,3079	0,3094
731, 553	15	0,3368	0,3381
650, 643	20	0,3513	0,3496
653	15	0,4004	0,4011

Tab. 1. Röntgenographische Daten von AgGaTe_2 -II. * Für CuK_α -Strahlung mit $a = 10,18_3$ Å.

³ K.-J. RANGEL, M. KREUBLER u. A. WEISS, Z. Naturforschg., 24b, 1060 [1969].

⁴ K.-J. RANGEL u. A. WEISS, Z. Naturforschg., 23b, 1262 [1968]; K.-J. RANGEL, D. POPPINGER u. A. WEISS, Z. Naturforschg., 24b, 813 [1969].

⁵ K.-J. RANGEL, J. ENGELS u. A. WEISS, Z. Naturforschg., 23b, 1262 [1968]; K.-J. RANGEL, D. POPPINGER u. A. WEISS, Z. Naturforschg., 24b, 1061 [1969].

⁶ K.-J. RANGEL, G. ENGERT, J. ENGELS u. A. WEISS, Z. Naturforschg., 23b, 1008 [1968].

⁷ K.-J. RANGEL, G. ENGERT u. A. WEISS, Z. Naturforschg., 24b, 813 [1969].

⁸ H. HARN, G. FRANK, W. KLINGER, A. MEYER u. G. STRÖGER, Z. anorg. allg. Chem., 271, 153 [1953].

⁹ F. DACHILLE u. R. ROY, in: J. H. DE BOER (Hrsg.), Reactivity of solids, Elsevier, Amsterdam 1962, p. 502.

Notizen Über Das Verhalten Des Schnabelt

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