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On the Phase $CrTe_3$

Short Communication

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A new phase $CrTe_3$ was discovered and its existence was confirmed by differential thermal analysis and X-ray investigations. Symmetry and lattice parameters as well as the temperature of its peritectic decomposition were determined.

(Keywords: Chalcogenide phases; Chromium-tellurium alloys; Crystallographic data; Differential thermal analysis)

Über die Phase $CrTe_3$

Eine neue Phase $CrTe_3$ wurde entdeckt und ihre Existenz wurde mittels differential-thermoanalytischer und röntgenographischer Untersuchungen sichergestellt. Es wurden Symmetrie und Gitterparameter sowie auch die Temperatur des peritektischen Zerfalls bestimmt.

During an investigation of the phase relationships in the chromium—tellurium system by means of differential thermal analysis and X-ray methods a hitherto unknown phase $CrTe_3$ was discovered.

All samples were prepared from chromium with 99.997% purity (referred to metallic impurities; AVIQUIPO-Corporation, USA) and from 99.99% tellurium (ASARCO, New York, USA). Weighed amounts of the pure materials were melted together in evacuated quartz capsules at about 1,300 K. The samples were then cooled in the furnace, the obtained ingots were crunched, finely ground and sealed again in quartz capsules. They were annealed at a temperature of about 675 K for at least four weeks.

For the DTA measurements a fully automated apparatus was used which has been described already in a previous paper[1]. The DTA heating curves of all samples between 75 and 95 at% Te showed an invariant thermal arrest at 718 ± 2 K and additionally one at

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753 ± 2K which was also found in the heating curve of a sample with 72.5 at % Te. These effects could be explained by the existence of a hitherto unknown phase which decomposes peritectically at 753 K, with the nonvariant reaction at 718 K corresponding to the eutectic between this phase and tellurium. The approximate stoichiometry of this new phase must be $CrTe_3$ because the invariant arrest at 753 K had its maximum intensity around 75 at % Te and the thermal effect at 718 K was not found anymore in the sample with 72.5 at % Te.

Powder diagrams were obtained in *Debye-Scherrer* cameras of 57.3 mm diameter using filtered CoK_α-radiation. A series of samples with compositions ranging from 62 to 90 at % Te was annealed at 675 K; it was compared to a corresponding series annealed at 850 K and quenched in ice water. The diffraction patterns of the latter samples showed the diffraction lines of the NiAs-type phase[2] with hexagonal symmetry and those of tellurium, with the strongest reflection of tellurium (101) already being observed at 62.5 at % Te. On the other hand, the diffraction patterns of the samples annealed at 675 K showed no tellurium reflections in the composition range between 62 and 75 at % Te, but the development of a new pattern, different from that of any chromium telluride described in lit.[2–6] could be observed. In a sample with 74.8 at % Te the strong (101)-reflection of the NiAs-type phase had disappeared completely, thus indicating the composition of the new phase to be $CrTe_3$ in accord with the conclusions drawn from the thermoanalytical investigations.

However, the powder pattern of the $CrTe_3$-phase was very complex and contained a large number of very weak lines so that indexing proved to be impossible. In order to determine symmetry and lattice parameters it was therefore necessary to investigate single crystals. Attempts to prepare single crystals from the melt or by halogen transport failed. Finally, small single crystals were obtained by annealing a sample with 74.8 at % Te for several weeks at about 730 K in the presence of $AlCl_3$ as a mineralizing agent. The crystals had the shape of tiny platelets with 20 to 40 μm diameter. Their composition was confirmed by comparison of rotating crystal photographs with the *Debye-Scherrer* pattern of the bulk $CrTe_3$-sample. Symmetry and lattice parameters were determined with the help of an automatic four cycle diffractometer (Philips PW 1100; MoK_α-radiation, graphite monochromator) which was also used to collect the intensity data. The crystallographic data obtained in this way are presented in Tab. 1.

The determination of the crystal structure of $CrTe_3$, presently being attempted, is of considerable interest, since this compound represents the first example of a tritelluride of a metal of the 3*d* transition series. Crystalline trichalcogenides of transition metals have so far been observed in group IV b and V b. With the exception of TiS_3[7] they are formed by the heavier elements in these groups. They are found to crystallize in one out of two structure types, either the $ZrSe_3$-[8] or the $TaSe_3$-type[9]. Both are layer structures which can be derived from the $NbAs_2$-structure type. Although the unit cell dimensions do not reveal a relationship of the $CrTe_3$-structure to either one, a layer structure is still very likely to be expected from morphological reasons.

Table 1. *Crystallographic data of* $CrTe_3$

Symmetry: simple monoclinic
Possible space groups: $P2/m$, $P2$, Pm
Cell content: 8 formula units
Lattice dimensions: $a = 10.370\ (1)$ Å
$b = 11.212\ (1)$ Å
$c = 7.896\ (1)$ Å
$\beta = 103.09\ (4)^\circ$
X-ray density: $\rho_x = 6.46\,\mathrm{g\,cm^{-3}}$

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