Vapor Transport Investigations using the High-temperature Gas-balance

Robert Heinemann, Peer Schmidt

BTU Cottbus - Senftenberg / Faculty of Sciences / Inorganic Solids and Materials

peer.schmidt@b-tu.de

The High-temperature Gas-balance (HTGB[1]) has been established as a new method for investigations of heterogeneous phase equilibria with volatile components [1,2]. By using the High-temperature Gas-balance (HTGB), thermogravimetric analysis is realized in closed silica ampoules. Thus an equilibrium gas phase is formed in permanent contact with the solid phase. The measurement signal is caused by change of the leverage of the horizontal balance support during evaporation and condensation. The application of the HTGB allows the analysis of solid-gas equilibria in the working range from 0.01 till 15 bar at temperatures up to 1100°C.

This equipment is notably well suited to analyze *reversible heterogeneous equilibria* which occur during chemical vapor transport reactions (CVT[3]).Thereby, the relevant gas-solid equilibria are expressed by the mass change (Δm_{Gas}) in the gaseous phase during vaporization or condensation of substances in the regarded temperature range. For a vapor transport of germanium using iodine as transport addition the measured equilibria were proofed by thermodynamic calculations (Figure 1). The vaporization of GeI_4 as transport agent (I) is followed by formation (I→II) and decomposition (II) of GeI_2(s). Between 640 and 900 K an enrichment of germanium (III) in the gaseous phase has been found which represents the transport equilibrium.

$$Ge(s) + GeI_4(g) \rightleftharpoons 2\,GeI_2(g)$$

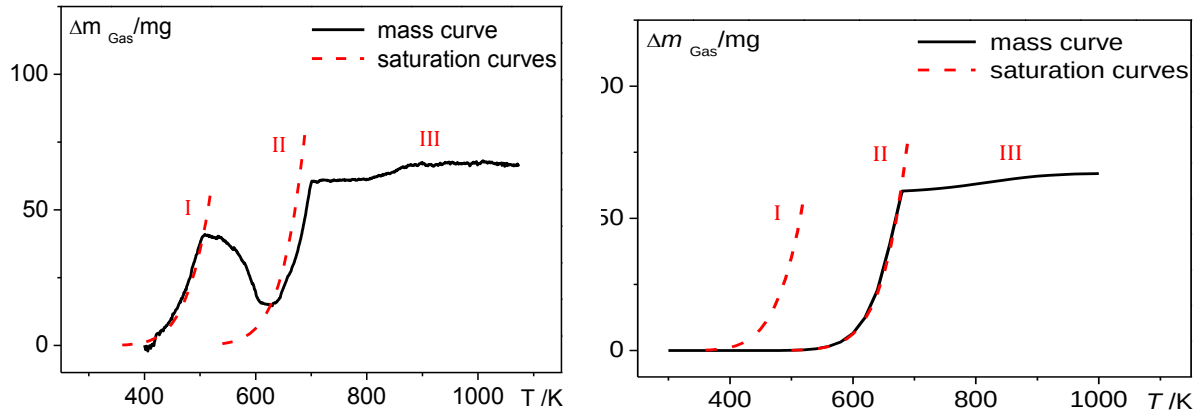

Figure 1: CVT of Ge-I; left: formation reaction by HTGB measurement, right: thermodynamic calculation of stationary equilibrium state

[1] P. Schmidt, M. Schöneich, M. Bawohl, T. Nilges, R. Weihrich, *J. Thermal Anal. Calorim.* **2012**, *110*, 1511.

[2] N. Eckstein, A. Hohmann, T. Nilges, P. Schmidt, *Z. Anorg. Allg. Chem*. **2013**, *639,* 2741.

[3] P. Schmidt, M. Binnewies, R. Glaum, M. Schmidt, Chemical Vapor Transport Reactions – Methods, Materials, Modeling, *in:* Advanced Topics on Crystal Growth, *InTech, Rijeka, **2013***