Multiple Knudsen Cell Configuration
Improved for Alloy Activity Studies

Knudsen effusion mass spectrometry (KEMS) allows the simultaneous determination of the identity and pressure of vapor species in equilibrium with a condensed phase as a function of temperature (ref. 1). This information can be used to determine the thermodynamic properties of materials. The partial pressure of species $j$ in the cell is related to the measured intensity of the ion $k$ formed from $j$, $I_j^k$, and the absolute temperature $T$, where $S_{jk}$ is the sensitivity factor.

$$p_j = \frac{(I_j^k + T)}{S_{jk}}$$

Measurement of absolute pressures requires the determination of $S_{jk}$, which is a difficult problem in KEMS. The inclusion of multiple effusion cells in the isothermal zone of a furnace allows direct comparison of the partial pressure of species in equilibrium with various condensed samples and can remove the need to determine $S_{jk}$. This provides a method to directly determine the thermodynamic activity of solution components at elevated temperatures (refs. 2 to 4), where $A$ and $R$ represent measurements from the alloy and reference, respectively.

$$a_j = \frac{P_j(A)}{P_j(R)} = \frac{[I_j^k(A) + T]}{[I_j^k(R) + T]} \cdot \frac{S_{jk}(R)}{S_{jk}(A)} = \frac{[I_j^k(A)]}{[I_j^k(R)]}$$

Despite its theoretical simplicity, it is based on $S_{jk}$, remaining constant, which introduces a range of challenging experimental conditions. All these issues have been resolved in a comprehensive redesign of the multiple-cell configuration of KEMS at the NASA Glenn Research Center that now allows accurate activity measurements to be made in a range of important metallic and ceramic systems. The following is a brief summary of the major changes.
Schematic of the fixed field and source apertures that attenuate a fixed molecular beam. The effusate is sampled by accurately aligning each cell with the fixed apertures.

Two fixed apertures--field and source apertures--were added between the effusion cell and the ion source (shown above). These apertures define a fixed molecular beam and maintain a constant ionization volume, while an accurate positioning mechanism (automated X-Y table, shown below) ensures that the same portion of the effusate distribution is sampled from all cells (refs. 5 to 9). The relative difference in effusate distribution, due to variation in orifice shape, is measured by loading a reference material into all cells and comparing the ion intensities (refs. 7 to 9). With these changes, the factors affecting the measured ion intensity of species effusing from a cell are limited to the composition and temperature of the condensed sample.
Effusion cell furnace and the X-Y table that is used to accurately position the effusion cells.

This technique relies on the ability to sample the effusate from all cells at one temperature and, therefore, a furnace that can maintain a constant temperature with time and cell position and has an isothermal zone large enough to contain all effusion cells. A resistance furnace was constructed by placing three effusion cells, with radial symmetry (ref. 10) in a molybdenum block at the center, surrounded by a cylindrical tungsten sheet-heating element (25-μm-thick) and a seven-layer tantalum heat-shield pack. A sheet-heating element provides the most uniform radiation heat transfer, while conduction in the
molybdenum block further reduces any thermal gradients. The required temperature stability was achieved after separating the water-cooling circuit from the electrical power circuit. The temperature of each effusion cell is measured independently with B-type thermocouples and a single-color, disappearing filament optical pyrometer.

These changes have created a world-class facility for the study of the thermodynamics of metallic and ceramic systems. This type of fundamental information is required to understand the behavior of materials used at high temperatures. Current studies are focused on Ti- and Ni-based alloys.

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Find out more about high-temperature mass spectroscopy at Glenn http://www.grc.nasa.gov/WWW/EDB/Facilities/mass_spectrometer_facility.htm.

References


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