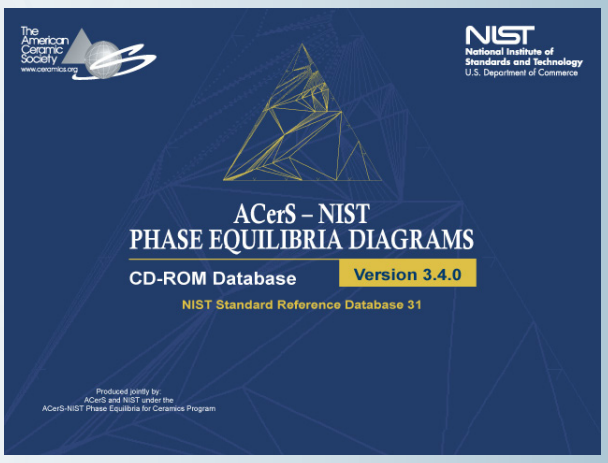
Phase Equilibrium Data

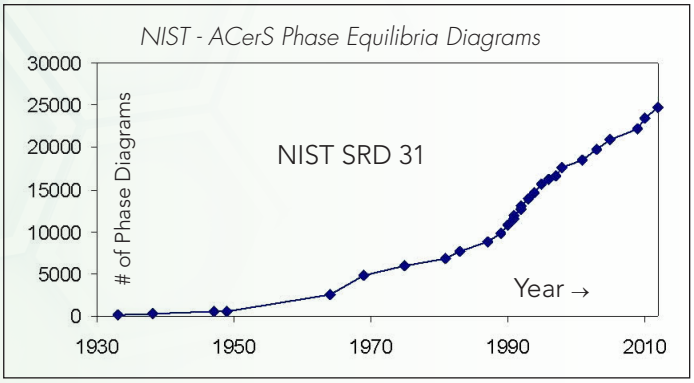
Objective

Advanced ceramics and inorganic materials are critically enabling elements in devices and systems across many technology sectors, such as telecommunications and energy. The objective of this project is to determine, compile and evaluate phase equilibrium data for NIST Standard Reference Database 31. The data, consisting of graphical diagrams, provide equilibrium chemical and structural behaviors. NIST Standard Reference Database 31 disseminates readily accessible and searchable data electronically in partnership with the American Ceramic Society (ACerS).



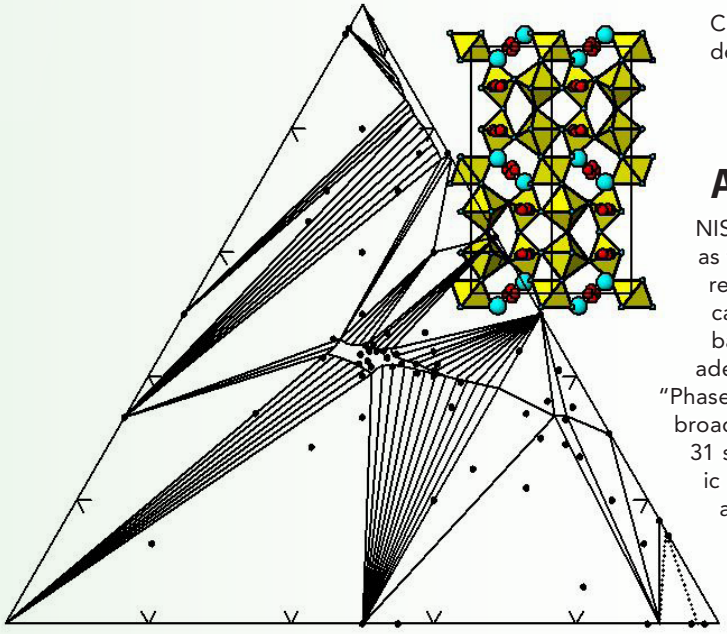
Impact and Customers

Every deliberate effort to develop a new ceramic or to improve processing begins with chemical composition and the conditions (temperature, pressure) under which pure compounds and their mixtures are stable (an equilibrium phase diagram). In the absence of such information, developers must resort to trial-and-error approaches or conduct their own studies to obtain the data.



- 24,800 diagrams in 21 hard-copy volumes, CD-ROM versions and a new web platform; more than 60,000 units sold worldwide
- About 1,000 new diagrams released annually
- Annual sales of $200,000 to $300,000
- 300 to 400 website hits per month at http://www.nist.gov/srd/nist31.cfm
- 40 to 60 downloads per month of demonstration version from NIST Standard Reference Data website

Customers: Materials developers in industry, government and academia



Approach

NIST Standard Reference Database 31, published from 1964 to 1992 as the well-known Phase Diagrams for Ceramists "blue books," is the result of a long-standing collaboration between NIST and The American Ceramic Society to develop and maintain a state-of-the-art database of critically evaluated phase equilibria data for industrial and academic customers. In 1992, the title was changed to the more general "Phase Equilibria Diagrams" to emphasize that the data are useful to the broader inorganic materials community. Standard Reference Database 31 serves as the only "Mapquest" database for ceramic and inorganic systems: The diagrams provide maps of the equilibrium chemical and structural behaviors exhibited by materials and provide critical starting information for the rational design of materials-processing schemes and for quality assurance efforts and optimization of the physical and chemical properties of advanced materials.

Accomplishments

The 2012 release is the largest-ever update of Standard Reference Database 31 and includes 900 new figures with more than 1,400 new phase diagrams, in addition to all the information previously printed in the 21 hard-copy volumes of the series. The new content includes experimental and calculated data for an unprecedented range of non-organic material types, including phosphates (batteries, laser and other optoelectronic materials), chalcogenides (semiconducting sulfides, selenides and tellurides for thermoelectrics, optoelectronics and photovoltaics), pnictides (nitrides, phosphides, arsenides and antimonides for bandgap-engineered optical materials, electron-transport devices, sensors, detectors, photovoltaics and thermoelectrics), halides (nuclear applications, scintillation detectors), and oxide/metal+oxide systems (electrode processing, catalysis, electroceramics, magneto-resistors, thermistors, capacitors, nuclear fuel and waste, ionic conductors, and fuel-cell electrolytes).

By way of illustration, if one searches for phase diagrams containing: SrO, TiO_2 and Nb_2O_5, we uncover Fig. 11446, a phase diagram for the ternary system (at right). An extensive commentary details the experimental data presented within the diagram. This is just one example of the more than 24,000 diagrams present in SRD 31.

A free demonstration CD is available upon request or can be downloaded from the website:

http://www.nist.gov/srd/nist31.cfm.

Fig. 11446 — SrO-TiO_2-Nb_2O_5

Ternary subsolidus for the system SrO-TiO_2-Nb_2O_5 in air in the temperature range 1275°-1475°C.

T. A. Vanderah, A. R. Drews, R. S. Roth, J. M. Loezos, and I. Levin, National Institute of Standards and Technology, Gaithersburg, Maryland; private communication, 2005.

Approximately 110 specimens were prepared by solid-state reaction of high-purity $SrCO_3$, Nb_2O_5, and phosphate-free TiO_2. Samples were ground 15 min in agate mortars prior to each heating. Specimens were heated as pressed pellets placed on powder of the same composition to avoid container reactions. Three or four multiple-day heatings were typically required to attain equilibrium, assumed when no further changes could be detected in the details of X-ray powder diffraction patterns. Some samples along the $SrTiO_3$-$Sr_2Nb_2O_7$ composition line required more than ten heatings to equilibrate. The lowest solidus temperatures were observed at SrO-TiO_2-Nb_2O_5 compositions 0.25:0.25:0.50 (1300°C) and 0.040:0.625:0.335 (1350°C). Specimens were identified and characterized by X-ray powder diffraction.

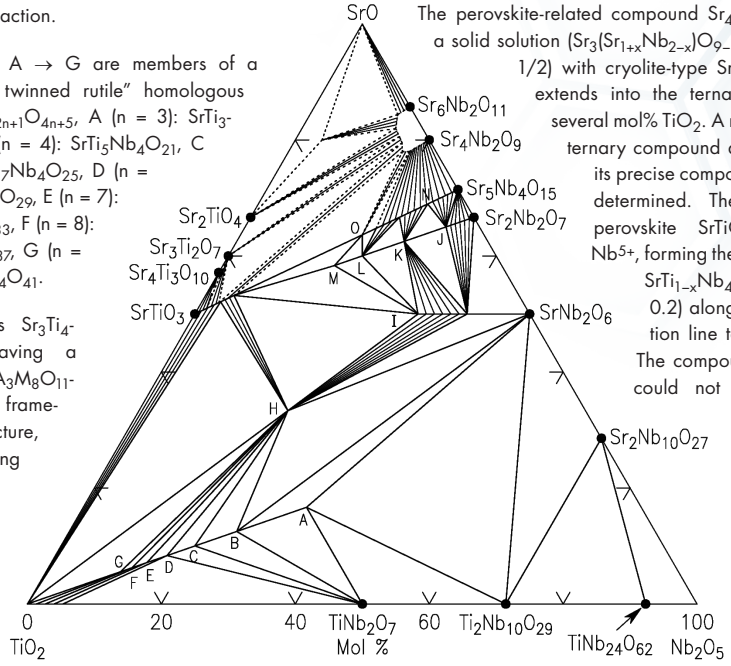
Compounds A → G are members of a "chemically twinned rutile" homologous series, $SrM_{2n+1}O_{4n+5}$, A (n = 3): $SrTi_3Nb_4O_{17}$, B (n = 4): $SrTi_5Nb_4O_{21}$, C (n = 5): $SrTi_7Nb_4O_{25}$, D (n = 6): $SrTi_9Nb_4O_{29}$, E (n = 7): $SrTi_{11}Nb_4O_{33}$, F (n = 8): $SrTi_{13}Nb_4O_{37}$, G (n = 9): $SrTi_{15}Nb_4O_{41}$.

Phase H is $Sr_3Ti_4Nb_4O_{21}$ having a hexagonal $A_3M_8O_{11}$-type open framework structure, and showing no detectable solid solution formation in contrast to the BaO-TiO_2-Nb_2O_5 system (Figs. EC-444, 9529). Phase I is a tetragonal tungsten bronze system, $Sr_{6-x}Ti_{2-2x}Nb_{8+2x}O_{30}$, with the value of x ranging from 0 to 0.5.

Phases J → M (and $Sr_2Nb_2O_7$) are members of the incommensurately modulated series $A_nB_nO_{3n+2}$, with structures built from slabs of perovskite, cut along the cubic (110) plane, with widths of n octahedra. The compounds share a basic orthorhombic subcell with a long dimension that increases with the width of the slabs. $Sr_2Nb_2O_7$ (= $Sr_4Nb_4O_{14}$) is the n = 4 member with perovskite slabs four [NbO_6] octahedra in width. J = $Sr_{4.33}Ti_{0.33}Nb_4O_{15}$ (n = 4,5), K = $Sr_5TiNb_4O_{17}$ (n = 5),5 L = $Sr_6Ti_2Nb_4O_{20}$ (n = 6),6 M = $Sr_7Ti_3Nb_4O_{23}$ (n = 7). The solid solution shown between phase J and $Sr_2Nb_2O_7$ consists of intergrowths.

Another perovskite-slab series, $A_nB_{n-1}O_{3n}$, forms along the line between $Sr_5Nb_4O_{15}$ and $SrTiO_3$. These hexagonal or rhombohedral phases feature slabs n–1 octahedra thick cut along the (111) parent perovskite planes. $Sr_5Nb_4O_{15}$ is the n = 5 member. Compound N (n = 6) is $Sr_6TiNb_4O_{18}$. The indicated "solid solution" between members consists of intergrowths among them.

The perovskite-related compound $Sr_4Nb_2O_9$ forms a solid solution ($Sr_3(Sr_{1+x}Nb_{2-x})O_{9-3x/2}$, x = 0 → 1/2) with cryolite-type $Sr_6Nb_2O_{11}$ that extends into the ternary, dissolving several mol% TiO_2. A new, high-SrO ternary compound also forms but its precise composition was not determined. The well-known perovskite $SrTiO_3$ dissolves Nb^{5+}, forming the solid solution $SrTi_{1-x}Nb_{4x/5}O_3$ (x ≤ 0.2) along the composition line to $Sr_5Nb_4O_{15}$. The compound $Sr_4Ti_3O_{10}$ could not be obtained as a single-phase in the present study.



Learn More

Terrell A. Vanderah,
Editor-in-Chief

301-975-5785
terrell.vanderah@nist.gov

http://www.nist.gov/srd/nist31.cfm

Publications

Phase Equilibria Diagrams CDROM v.3.4, NIST Standard Reference Database 31, The National Institute of Standards and Technology and The American Ceramic Society, 2012.

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