SIXTH INTERNATIONAL CONFERENCE ON HIGH TEMPERATURES—CHEMISTRY OF INORGANIC MATERIALS Gaithersburg, MD April 3–7, 1989

Report prepared by

J. W. Hastie

Metallurgy Division,
Materials Science and Engineering Laboratory,
National Institute of Standards and Technology,
Gaithersburg, MD 20899

This conference was the sixth of a series, sponsored by the International Union of Pure and Applied Chemistry (IUPAC) Commission II.3 on High Temperature and Solid State Chemistry, and which is held about every 3 years.

The NIST meeting represented only the second occasion that this conference series had been held in the U.S.A. Attendance, exceeding 170, included participants from 19 countries, and 130 papers were presented.

1. About the Conference

The conference program emphasized the basic chemical science and measurement issues underlying the characterization, processing, and performance of materials at high temperatures. Each of the major classes of materials was considered, including high performance alloys, ceramics, composites, and specialized forms such as films, coatings, clusters, powders, slags, fluxes, etc. in addition, individual substances, namely the elements and their compounds, were discussed in detail. Seven plenary lectures and 68 invited talks were given as well as 61 poster presentations and computer-based demonstrations. Also, Prof. Leo Brewer, one of the foremost pioneers of the field, gave an overview of the conference proceedings together with his perspective on the “Role of Chemistry in High-Temperature Materials Science and Technology.” During the conference sessions, many of the hot issues of the day were also discussed, including cold fusion, high-temperature superconductors, low pressure production of diamond films, etc.

Participation by the leading international researchers in the field was particularly strong in the materials-related areas of measurement techniques, thermochemistry and models, processing and synthesis, and performance under extreme environments. Of special interest were the topics on databases and phase equilibria models, processing—mainly from the vapor phase, and high power laser-materials interactions.

The conferees were welcomed by Dr. Lyle Schwartz, Director of the Institute for Materials Science and Engineering (IMSE) (now Materials Science and Engineering Laboratory), who also gave an overview of pertinent NIST and IMSE research activities. Prof. Jean Drowart of the Free University of Brussels, Belgium, addressed the meeting on behalf of IUPAC and gave a fascinating account of “7000 Years of High Temperature Materials Chemistry.”
A few representative technical highlights from each of the main conference sessions are given in the following discussion.

2. Advances in Measurement Techniques

Three areas were given special emphasis. These were spectroscopic probes, diffractometry, and physicochemical methods. The types of spectroscopic probes discussed included Raman and related laser spectroscopic methods for in situ molecular-level or phase-specific monitoring of hot surfaces. Examples were considered in the areas of corrosion, oxide superconductor processing, and in Raman imaging of ceramic crack suppression due to phase transformation toughening (see fig. 1). An interesting novel application of in situ optical emission spectroscopic analysis of molten steel, using a laser-induced plasma-forming technique, was also discussed (see fig. 2). These effectively nonintrusive methods also have potential as process monitoring probes for intelligent processing in addition to their utility in experimental systems.

In the area of diffractometry, in situ analysis of material structures at high temperatures, using x-ray and neutron sources, was described. Atom probe chemical analysis on alloy surfaces using field-ion microscopy was also discussed.

Physicochemical techniques have traditionally been key to the characterization of materials at high temperatures and significant recent advances have occurred in this area. Methods have been developed which effectively eliminate containment problems. For instance, with liquid metals, transient microsecond time scale techniques have been applied to accurate measurements of melting points and heat capacities at very high temperatures. For steady state measurements, electromagnetic levitation may be used as, for instance, with emissivity and optical constant measurements. Another transient technique that was discussed by a number of researchers throughout the conference is the pulsed laser-heating approach to the production of vapor species for mass and optical spectroscopic characterization.

4. Processing and Synthesis

The chemical basis for high temperature processing and synthesis of materials is a rapidly growing area of research and representative work in the field was discussed. An area of significant promise for the design of new or improved materials is that of molecular/atomic clusters. These species, with properties intermediate between molecular and bulk material, are key reaction intermediates to most deposition and condensation processes. They also serve as model structures for surfaces owing to their intrinsic high ratio of surface to bulk atoms. Their unique reactivity as a function of cluster size was indicated by several speakers (see fig. 5).

The session on CVD and other vapor phase-based processes was particularly exciting. Thermochemical, kinetic, transport models, whereby the processing of films (diamond, semiconductor, ceramic, alloy, etc.) could be optimized, were described (see fig. 6).

5. Performance Under Extreme Environments

The important related areas of hot and high temperature corrosion were discussed for both alloy and ceramic materials. In particular, the key role of chemical reaction and solubility was demonstrated (see fig. 7).

Another area where materials are subject to extreme conditions is that of laser-materials interactions. There are many areas of science and technology that require an improved understanding of this interaction, including design of laser resistant materials, laser deposition of films, laser etching for electronic devices, laser stimulated chemical processing, laser welding, and laser heating for containerless studies of thermochemistry at ultra-high temperatures. This latter case has special
significance to providing thermodynamic data for nuclear reactor excursions (see fig. 8) and for materials data for advanced aerospace applications.

6. Additional Information

A three volume proceedings (1350 pages) is being published by Humana Press, Clifton, NJ. Many of the conference presentations will appear in these volumes. Also included are a few articles, not presented at the conference, in order to provide a more complete coverage of certain topics. This will be the first generally available publication for this subject area and the proceedings should be of considerable interest to researchers, students, and others interested in the scientifically challenging, and technologically indispensable, interplay between materials and high temperatures.

The next meeting in the series is scheduled to be held in 1991 in Orleans, France and will be chaired by J. P. Coutures.
Figure 1. A map of the monoclinic phase fraction of a zirconia specimen subjected to an applied stress and crack growth. The stress history of the material is revealed in the extent and degree of transformation of the transformed zone. Large stresses induce a larger transformed zone around the crack tip that remains after the crack tip moves forward. (Taken from Rosenblatt et al., paper 4.)
Figure 2. Time-resolved emission spectra from a laser produced plasma plume generated off a specialty steel alloy target. Each trace represents a 20 ns exposure spectrum covering the spectral range of 1850 to 6200 Å. Each successive trace is delayed by 20 ns and the 50 traces shown cover the first 1 μs of the plume. The laser energy is 3.38 J and the ambient gas is argon at 0.015 Torr at room temperature. (Taken from Kim, paper 5.)
Figure 3. Enthalpy of mixing of the NaF–ZrF$_4$ system. Data points are experimental and line is calculated using an associated liquid model. (Taken from Gaune-Escard et al., paper 35.)

![Enthalpy of mixing](image)

Figure 4. Structure factors, $S(Q)$, for liquid KPb. Solid line: $S(Q)$ from diffraction measurements on SEPD; Points $S_0(Q) = -\Delta f^2 S(Q,E)$ from inelastic scattering measurements on LRMECS: (C)$\Delta = 40$ meV, (o)$\Delta = 5$ meV. (Taken from Saboungi et al., paper 27.)

![Structure factors](image)
Figure 5. Reaction rate of Pt with CH₄, normalized to Pt. (Taken from Kaldor et al., paper 77.)

Figure 6. Phase fields for deposition of Ge₃N₄ as a function of deposition temperature and the feed ratio \( P_{\text{NH}_3} / P_{\text{GeCl}_4} \) for the GeCl₄-NH₃-N₂ system. \( P = 1 \) atm and \( P_{\text{GeCl}_4} = 10^{-2} \) atm. (Taken from Anderson et al., paper 105.)

Figure 7. Trace of basicity and oxygen activity measured for preoxidized 99% Ni covered with a Na₂SO₄ film at 900 °C in 0.1% SO₂-O₂ gas atmosphere (preoxidized at 900 °C for 4 h in O₂). Numbers designate reaction time in hours except as indicated. Severe corrosion conditions. (Taken from Rapp, paper 115.)
Figure 8. Maximum UO$_2$$^+$ signals from the mass spectrometer for laser pulses of varying strength. $Q_p$ is the peak absorbed power density, and $T_{\text{max}}$ is the measured maximum surface temperature in the pulse. The scale designating the maximum number density in the ionizer of the mass spectrometer was calculated from measured ion intensities and the vapor pressure (Torr) is that of UO$_2$ at the peak surface temperature. The hatched area represents the range of results of the steady-state calibrations. (Taken from Olander, paper 123.)
7. List of Papers Presented at the Conference

AESRDANCES IN MEASUREMENT TECHNIQUES
Spectroscopic Probes

1. R. J. M. Anderson and J. C. Hamilton—(Sandia National Lab., United States) Nonlinear Optical Spectroscopy as a Probe of Processes and Processes at Surfaces and Interfaces

2. E. F. McNally, D. R. Bockroo, D. S. Glish, L. E. Vanturkin, and B. M. Gurtin—(Sandia National Lab., United States) High Temperature Oxidation of Silicon Coatings: A Raman Scattering Study

3. D. A. Ludden—(Sandia National Lab., United States) Temperature Measurements in Silica and Silicates: New Techniques for Temperature Measurements

4. G. R. Blackmore and D. E. Veirs—(Lawrence Berkeley Lab., United States) Recent Developments using Imaging Detectors for Raman Characterization of High Temperature Materials

5. Y. W. Kim—(Lehigh Univ., United States) Laser Plasma Plasma Analysis in High Temperature Environments

6. S. Scholz and K. Krasinski—(Tokyo Univ., Japan) InfraRed Spectrum of High Temperature Melts by Means of Emission Spectroscopy

7. L. J. Kucera, N. B. D. N. L. W. J. O. G. D. M. S. P. K. and N. A. Young—(Univ. Southampton, United Kingdom) EXAFS, Matrix Isolation and High Temperature Chemistry

Diffraclometry

8. H. F. Francis and S. J. Gim—(Johns Hopkins Univ., United States) High Temperatures for X-Ray and High Temperature X-Ray Diffraclometry of Ti-Al Alloy Phase Transitions

9. J. F. B. F. Jr. and R. L. H. Litterton—(Argonne National Lab., United States) High Temperature X-Ray Diffraction of Neutron Diffraction Studies of the Defect Structure of Neutron-doped Oxides

10. P. P. Chao—(NIST, United States) Field Ion Microscopy and Atomic Probe Chemical Analysis

Physico-Chemical Methods

11. A. C. Cambray—(NIST, United States) A Microsecond-Resolution Transient Technique for Thermophysical Measurements on Liquid Refractory Metals

12. R. H. Harben, W. A. Howard, and J. C. L. Margrave—(Rice Univ., United States) Emissivities and Optical Constants of Electromagnetically Levitated Liquid Metals as Functions of Temperature and Wavelength

13. M. Shamsudin, K. J. K. S. J. E. and M. L. S. R. R. A. S. Chemical and Physical Methods for Measurement of Thermodynamic Properties of Polycrystalline Materials

14. T. T. V. N. K. K. B. and L. N. S. K. L. A. New Technique for Measurement of Low-Q Partial Pressures

15. M. A. Pickel and E. A. Gass—(IBM Yorktown Heights, United States) Kinetics of Water Desorption from Glass Powders Studied by Knudsen Effusion Mass Spectrometry

16. R. J. Stirn, M. J. S. J. and M. S. G. H. A. H. A. H. Kinetics of Water Desorption from Glass Powders Studied by Knudsen Effusion Mass Spectrometry

17. J. T. Asela—(Dominion Univ., Canada) Thermodynamic Modeling of Solution Phases and Phase Diagram Calculations

18. A. B. Pelton and J. P. Bros, and B. M. Ruthardt—(Nuclear Research Centre, Federal Republic of Germany) Extended and Updated Data Bank on Thermodynamic Properties of Semiconductors

19. J. K. R. Weber—(Univ. of Toledo, United States) Vaporization Behavior of Molten Rare Earth Metals

20. M. Iwase, H. Af. Ondik—(QIST, United States) Determination of the Enthalpy of Dissociation of the Molecule CrPb by Faraday Effect

21. W. Gilles and G. F. Kessinger—(Univ. of Kansas, United States) The High Temperature VapORIZATION and Thermodynamics of the Magnesium Phases in the Calcium-Oxygen System

22. C. A. Atwood—(Univ. Notre Dame, United States) Strain Distribution in Oxide Ceramic Materials

23. R. Myers, R. W. Nichols, and B. S. Landford—(Martin Marietta Energy Systems, United States) Mechanistic Aspects of Metal Sulfate Decomposition Processes

24. D. W. Ronald and J. W. Hasie—(NIST, United States) A Predictive Slag Phase Diagram Model

25. H. M. Onisk—(NIST, United States) The NIST-Acem Ceramic Phase Diagram Data Base

26. G. G. E. K. P. B. N. C. and G. Hagen—(Univ. de Frevence, France) Thermodynamic Modelling of High Temperature Melts and Phase Diagram Calculations

27. M. G. Rusk and G. Hasch—(Univ. de Frevence, France) Thermodynamic Modelling of High Temperature Melts and Phase Diagram Calculations

28. F. P. Gillies and M. A. W. Williams—(Univ. of Kansas, United States) Vaporization Chemistry of the Vanadium Oxides

29. R. Martin, C. D. Childs, and R. J. Weber—(Univ. of Toledo, United States) Vaporization Behavior of Molten Rare Earth Metals

30. J. C. Hamilton and J. K. R. Weber—(Univ. of Toledo, United States) Vaporization Chemistry in the CaO-SiO2 System

31. G. Balducci, G. De Maria, G. Gigli, and M. Guido—(Univ. di Roma 'La Sapienza', Italy) Vaporization Behavior of Molten Rare Earth Metals

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44. C. A. Atwood—(Univ. Notre Dame, United States) Strain Distribution in Oxide Ceramic Materials

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46. D. W. Ronald and J. W. Hasie—(NIST, United States) A Predictive Slag Phase Diagram Model

47. H. M. Onisk—(NIST, United States) The NIST-Acem Ceramic Phase Diagram Data Base

48. G. G. E. K. P. B. N. C. and G. Hagen—(Univ. de Frevence, France) Thermodynamic Modelling of High Temperature Melts and Phase Diagram Calculations

49. F. P. Gillies and M. A. W. Williams—(Univ. of Kansas, United States) Vaporization Chemistry of the Vanadium Oxides

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67. P. D. Kleinachschmidt and K. Aaker—(Los Alamos National Lab., United States) Activity and Free Energy of Formation of the Compound CaOCl₃
68. R. F. Gurdine, R. A. Schifman, and J. K. R. Weber—(Intercontec, Inc., United States) Direct Laser Ablation of Boron
69. G. N. Papathodorou and L. Nalbandian—(Institute of Chemical Engineering and High Temperature Chemical Processes, Greece) Raman Spectra and Vibrational Analysis of the F1+2, F1C₄, A₃C₄, and A₄C₄ Vapor Molecules
70. M. Shumalovin and A. Naur—(Bashkirian Hanu Univ., India) Thermodynamic Properties of Calcium Telluride
71. F. R. Shively, T. Y. Archak, and M. M. Shanks—(Institute of Silicate Chemistry of the Academy of Sciences, U.S.S.R.) High Temperature Mass Spectrometric and Infrared Study of the Thermodynamic Properties of Boron Cermets Systems
72. M. E. Joint and W. E. Thompson—(NIST, United States) The Production and Spectroscopy of Small Polyatomic Molecular Ions Isolated in Solid Neon
73. M. Shumalovin, A. Naur, and V. B. Tsur—(Bashkirian Hanu Univ., India) Electrical Conductivity and Defect Structure of Calcium Telluride
74. M. Guert-Escard and A. Bogan—(Univ. de Provence, France) Cetallimetric Invaration of NiOCl and of Ni₂O₃-3Cl₂ Mixtures
75. J. P. Bok, D. El Alain, M. Gaust-Escard, and E. Hay—(Univ. de Provence, France) Halothane Formation of Ni- and Pd-Based Ternary Alloys
76. C. B. Coughman, T. J. Anderson, and J. J. Egbe—(Univ. of Florida, United States) Thermodynamic Investigations of the Al₂S₃ and Al₂S₆ Systems by Solid State Electrochemistry

PROCESSING AND SYNTHESIS Clusters as Reaction Intermediates and Model Structures

77. A. Kadar—(Exxon Research and Engineering, United States) Clusters as Intermediates for New Materials
78. F. W. Freben, T. M. Chandrasekhar, and J. Kolenda—(Freie Univ. Berlin, Federal Republic of Germany) Cluster Formation in Laser-Induced Material Interaction With Optical Spectroscopic Characterization
79. M. Voly, T. M. Chandrasekhar, and A. Szczepanski—(Univ. of Florida, United States) Vapor Pressure and Structure of Small Carbon Clusters
80. K. G. Well and A. Hartman—(Technische Hochschule Darmstadt, Federal Republic of Germany) Mechanism of Cluster Formation During Evaporation of Aluminum
81. E. Hillert and D. Kast—(Research Center, Federal Republic of Germany) Investigations of Small Alkal Metal Clusters by Kausty Ellulon Mass Spectrometry using Broad Band Photolization
82. T. C. D'Vera and J. L. Gole—(James Madison Univ., United States) Oxidation of Small Metal Clusters
83. R. S. Berry, H.-P. Cheng, and J. Rose—(Univ. of Chicago, United States) Freezing and Melting of Metallic and Salt-Like Clusters
84. E. Blustrupiter and M. Nyholm—(NIST, United States) Thermal Fragmentation of Small Carbon Clusters
85. K. A. Gingerich, J. E. Kleczek, and J. I. Shinn—(Texas A&M Univ., United States) Band Resonance and Nature of Bonding in Small Transition Metal Solid-Conductor Clusters
86. R. J. Ficorillo and J. H. Hawley—(Univ. of Florida, United States) Heterogeneous Formation of Aluminum Vapor Clusters

Nucleation and Growth of Small Particles

87. J. Schoonman, R. A. Bau, and J. G. M. Recht—(Delf University, The Netherlands) Laser-Carboh Vapor Precipitation of Ultrafine Carbon Powders: Si and Si₃N₄
88. N. Shima and K. Yoshitake—(Nihon Kosan Central Research Labs., Japan) Laser Production of Metallic Plates from Organometallic Compounds
89. J. L. Katz and M. D. Donohue—(Johns Hopkins Univ., United States) Nucleation with Stimulation Chemical Reaction
90. T. D. Kunz, R. F. Menefee, and L. G. Fredin—(Rice Univ., United States) Laser-Vaporization of Uranium Dioxide and Other Refractory Materials
91. D. A. Cremers, R. D. Dixon, R. C. Estler, G. K. Lewis, R. E. Dufay, and D. R. Oland—(Univ. of California, United States) Direct Laser/Interactions Laser Ablation of Supercritical Materials and Laser Welding
92. J. M. Berry, T. D. Kruz, R. F. Memfiske, and L. G. Fredin—(Rice Univ., United States) Laser Ablation of Supercritical Materials and Laser Welding
93. Y. Nishina—(Univ. of California Berkeley, United States) A Conference Overview with a Personal Perspective on the Role of Chemistry in High Temperature Materials Science and Technology

CONFERENCE WRAP-UP

102. J. S. Horwitz and M. C. Lim—(U.S. Naval Research Labs., United States) Laser and Mass Spectrometric Studies of the Mechanism of Silica Single Crystal Etching Reactions
103. R. A. Mener—(Univ. of California Davis, United States) The Utilization of Combustion Processes for the Synthesis of High Temperature Materials
104. K. L. Romerik and H. Blaha—(Institute of Inorganic Chemistry, Austria) The Reduction of Silica With Graphite
105. T. J. Anderson, J. L. Puntawier, and F. Deoort—(Univ. of Florida, United States) Thermodynamic Analysis of Ge₅N₄ Chemical Vapor Deposition
106. Z. H. Xuelf and R. S. Pong—(Naval Research Lab., United States) The Activation of the CH₂-Bond of Alkane by Ground State Atomic Iron
107. T. C. DeVore, M. L. Smith, C. P. Fagri—(James Madison Univ., United States) Chemical Vapor Transport Species Resulting from the Oxidation of Hot W. Mo Filmst by NO₂, O₂, POCl₃, and KClO₃

Process Models and Materials by Design

108. P. J. Spencer and H. Holle—(Lehrstuhl fur Theoretische Htittenkunde, Federal Republic of Germany) Application of a Thermochimical Data Bank System to the Calculation of Metastable Phase Formation During PVD of Carbides, Nitrides and Borides Coatings
109. P. R. Streett and O.-M. Chwe—(Univ. of Connecticut, United States) Ultrasonic Composite Synthesis by Laser-Induced Reactive Evaporation and Rapid Condensation
110. J. W. Mitchell and G. Cadet—(AT&T Bell Labs, United States) Microprobe Discharge Synthesis and Characterization of Materials
111. R. W. Frobert, W. G. Ingo, A. Mazzarano, S. Sturlic—(Centro Sviluppo Materiali SpA, Italy) High Temperature Stability of Ge₂O₅:Yα Stabilized Zirconia Plasma Spray Powders: XPS and DIA Investigations
112. N. Zechetti and M. G. Ingo—(Centro Sviluppo Materiali SpA, Italy) XPS Investigations on the Chemical Structure and Growth Model of Amorphous Silicon Nitride (a-SiNₓ)

PERFORMANCE UNDER EXTREME ENVIRONMENTS Hot Corrosion

113. R. A. Rey—(Ohio State Univ., United States) Hot Corrosion of Materials
114. R. L. Jones—(Naval Research Labs., United States) Zinc-Oxide-Acid Reactions in Ceramic Corrosion
115. N. S. Johnson, I. M. Morra, E. R. Kreidler, and M. J. McAllan—(NASA Lewis Research Center, United States) High Temperature Reactions of Ceramics and Metals with Chlorine and Oxygen
116. N. Birk, D. L. Bishel, and F. S. Petit—(Univ. of Pittsburg, United States) Erosion and Corrosion of Metals in Sulfurous Atmosphere
117. I. Tomszuka, H. Hunska, H. Herada, Y. Kozumi, and M. Yamasaki—(National Research Institute for Metals, Japan) Effects in Processing History and Minor Element Contents on Hot-Corrosion Behavior of a Power-Metaiurgically Prepared Nickel-Based Superalloy
118. B. H. Roepma-Klein and J. Kästker—(FOM-Institut for Atomic and Molecular Physics, The Netherlands) Material Transport at the Interface of a Graphite Wall and a CO₂ Gas/Liquid Mixture
119. T. Pincaut, M. Kubel, and B. Emnott—(Association EURATOM-ENEA sulla Fusione Centro Ricerca Energia Frascati, Italy) Deuterium Implantation in C-SiC and CSi309P09 Materials
120. P. L. Kodaisy, P. S. Kiley, and V. J. Shemet—(Institute of Superhard Materials, Academy of Sciences, U.S.S.R.) High Temperature Oxidation of Molybdenum Aluminides

High Power Laser-Materials Interactions

121. D. R. Olmedo, S. K. Yaguihi, and C. H. Tait—(Univ. of California, United States) Laser-Pulse-Vaporization of Uranium Dioxide and Other Refractory Materials
122. J. L. Lyman, D. A. Creners, R. D. Dixon, R. C. Estler, G. L. Lewis, R. E. Murekhausen, N. S. Nogar, M. Pitch—(Los Alamos National Lab., United States) Direct Laser/Materials Interactions Laser Ablation of Supercritical Materials and Laser Welding
123. J. M. Berry, T. D. Kruz, R. F. Memfiske, and L. G. Fredin—(Rice Univ., United States) Laser Ablation of Supercritical Materials and Laser Welding
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