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Title:

SYNTHESIS AND DESIGN OF SILICIDE
INTERMETALLIC MATERIALS

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Submitted to:

High Temperature Structural Silicide
Conference
May 25-29, 1998 Hyannis, MA

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SYNTHESIS AND DESIGN OF SILICIDE INTERMETALLIC MATERIALS

DOE/AIM QUARTERLY PROGRESS REPORT January-March 1998

J.J. Petrovic, R.G. Castro, D.P. Butt, Y. Park, K.J. Hollis, H.H. Kung

Los Alamos National Laboratory
Los Alamos, New Mexico 87545

INTRODUCTION:

The overall objective of this program is to develop structural silicide-based materials with optimum combinations of elevated temperature strength/creep resistance, low temperature fracture toughness, and high temperature oxidation and corrosion resistance for applications of importance to the U.S. processing industry. A further objective is to develop silicide-based prototype industrial components. The ultimate aim of the program is to work with industry to transfer the structural silicide materials technology to the private sector in order to promote international competitiveness in the area of advanced high temperature materials and important applications in major energy-intensive U.S. processing industries.

The program presently has a number of developing industrial connections, including a CRADA with Johns Manville Corporation targeted at the area of MoSi_2 -based high temperature materials and components for fiberglass melting and processing applications. We are also developing an interaction with the Institute of Gas Technology (IGT) to develop silicides for high temperature radiant gas burner applications, for the glass and other industries. With Combustion Technology Inc., we are developing silicide-based periscope sight tubes for the direct observation of glass melts

High Temperature Structural Silicides Conference:

The Program for the Engineering Foundation High Temperature Structural Silicide Conference, to be held 25-29 May 1998 in Hyannis, Cape Cod, Massachusetts, has been finalized. The Final Program is shown below:

Final Program

High Temperature Structural Silicides Conference

**May 25-29, 1998
Tara Hyannis Hotel & Conference Center
West End Circle
Hyannis, Massachusetts 02601**

CO-CHAIRS:

J.J. Petrovic, Los Alamos National Laboratory
A.K. Vasudevan, Office of Naval Research
S.G. Fishman, Office of Naval Research
C.A. Sorrell, U.S. Department of Energy
M.V. Nathal, NASA-Lewis Research Center

Engineering Foundation Conferences
345 East 47th Street, Suite 303
New York, NY 10017
Tel: 1-212-705-7836; Fax: 1-212-705-7441
Email: engfnd@aol.com
WWW: <http://www.engfnd.org/8as.html>

Monday, 25 May 1998

4:00-6:00 pm Registration

7:00-9:00 pm Dinner followed by Opening Reception

Tuesday, 26 May 1998

7:00-8:30 am Breakfast

8:30-8:45 am Welcome
Conference Organizing Committee
Engineering Foundation

(1) 8:45-9:15 am Keynote Presentation
Key Developments in High Temperature Structural Silicides
J.J. Petrovic, Los Alamos National Laboratory, Los Alamos, New Mexico, USA
A.K. Vasudevan, Office of Naval Research, Arlington, Virginia, USA

Session: Materials I

Chair: J.J. Petrovic, Los Alamos National Laboratory

(2) 9:15-10:00 am Invited Presentation
Strong Monolithic and Composite MoSi₂ Materials by Nanostructure Design
K. Niihara and Y. Suzuki, Osaka University, Osaka, Japan

(3) 10:00-10:45 am Invited Presentation
Boron Doped Molybdenum Silicides for Structural Applications
M. Akinc, Iowa State University, Ames, Iowa, USA

10:45-11:00 am Coffee Break

(4) 11:00-11:45 am Invited Presentation
Development and Characterization of $\text{SiC}_7/\text{MoSi}_2\text{-Si}_3\text{N}_4$ Hybrid Composites
M.G. Hebsur, NASA Lewis Research Center, Cleveland, Ohio, USA

(5) 11:45-12:15 am Fabrication of Zirconium Silicide Intermetallic Compounds
With 16H-Type Crystal Structure
Y. Ikarashi, Tsuruoka Kogyo Koto Senmon Gakko, Yamagata, Japan
T. Nagai and K. Ishizaki, Nagaoka Gijyutsu-Kagaku Daigaku, Niigata, Japan

(43) 12:15-12:45 pm Mo_5Si_3 Single Crystals: Physical Properties and Mechanical Behavior
F. Chu, D.J. Thoma, K. McClellan, and P. Peralta, Los Alamos National Laboratory, Los Alamos, New Mexico, USA

12:45-2:00 pm Lunch

Session: Materials II

Chair: M.V. Nathal, NASA-Lewis Research Center

(6) 2:00-2:45 pm Invited Presentation
The Development of Mo-Si-B Alloys for Aerospace Applications
D.M. Berczik and M.V. Garguilo, Pratt & Whitney, West Palm Beach, Florida, USA

(7) 2:45-3:30 pm Invited Presentation
A Comparison of MoSi_2 Composites With Other Silicon-Base Structural Materials
E.L. Courtright, Pacific Northwest National Laboratory, Richland, Washington, USA

(8) 3:30-4:15 pm Invited Presentation
An Overview of Ti-Based and Ni-Based Aluminides
L. Christodoulou and J.A. Christodoulou, Imperial College of Science, Technology and Medicine, London, UK
S.L. Kampe, Virginia Polytechnic Institute and State University, Blacksburg, Virginia, USA

(9) 4:15-4:45 pm Microstructure and Mechanical Properties of Mo- $\text{Mo}_3\text{Si-Mo}_5\text{SiB}_2$ Silicides
J.H. Schneibel, C.T. Lui, D.S. Easton, and C.A. Carmichael, Oak Ridge National Laboratory, Oak Ridge, Tennessee, USA

4:45-5:00 pm Coffee Break

(10) 5:00-5:30 pm Phase Structure Change of Mo-Si System During Mechanical Alloying
L. Shaojun, Q. Xuanghai, L. Zhijiang, and H. Baiyun, Central South University of Technology, Hunan Changsha, PR of China

(11) 5:30-6:00 pm Regularities in Solid Solutions of Transition Metal Disilicides
L.A. Dvorina, Institute for Problems of Materials, Ukrainian Academy of Sciences, Kiev, Ukraine

(12) 6:00-6:30 pm **MoSi₂-ZrO₂ Composites with Fine-Grain Matrix**
D. Yi and C. Li, Chalmers University of Technology, Gothenburg, Sweden

7:30-9:00 pm **Dinner**

9:00-10:00 pm **Social Hour**

Wednesday, 27 May 1998

7:00-8:30 am **Breakfast**

Session: Mechanical Behavior

Chair: L. Kabacoff, Office of Naval Research

(13) 8:30-9:15 am **Invited Presentation**
Brittle-Ductile Behavior of Single Crystals of Transition Metal Disilicides
M. Yamaguchi, Kyoto University, Kyoto, Japan

(14) 9:15-10:00 am **Invited Presentation**
Effect of Alloying on the Mechanical Properties of MoSi₂
T.E. Mitchell, Los Alamos National Laboratory, Los Alamos, New Mexico, USA

(15) 10:00-10:45 am **Invited Presentation**
Strength and Deformation Mechanism of C40-Based Single and Polycrystalline Silicides
Y. Umakoshi, T. Nakano, M. Kishimoto, D. Furuta, and K. Hagihara, Osaka University, Osaka, Japan

10:45-11:00 am **Coffee Break**

(16) 11:00-11:45 am **Invited Presentation**
Plasticity Enhancement Mechanisms in MoSi₂
R. Gibala, H. Chang, C.M. Czarnik, and J.P. Campbell, The University of Michigan, Ann Arbor, Michigan, USA

(17) 11:45-12:15 pm **Plastic Deformation of Transition-Metal Disilicides With C11_b and Its Derivative Structures**
H. Inui, M. Moriwaki, and M. Yamaguchi, Kyoto University, Kyoto, Japan

(18) 12:15-12:45 pm **In Situ Straining Experiments on MoSi₂ Single Crystals in an HVEM**
S. Guder, M. Bartsch, and U. Messerschmidt, Max Planck Institute of Microstructure Physics, Halle/Saale, Germany
M. Yamaguchi, Kyoto University, Kyoto, Japan

(45) 12:45-1:15 pm **Hot Hardness Testing of Single Crystal NbSi₂ and TaSi₂**
S.A. Maloy, J.J. Petrovic, and T.E. Mitchell, Los Alamos National Laboratory, Los Alamos, New Mexico, USA

1:30-2:30 pm **Lunch**

2:30-6:30 pm **Ad hoc discussions/free time**

6:30-8:00 pm **Dinner**

Session: Atomic Mechanisms

Chair: P. Angelini, Oak Ridge National Laboratory

(19) 8:00-8:45 pm Invited Presentation

Microalloying for Ductility in Molybdenum Disilicide

M. Duesbery, Fairfax Materials Research Inc., Springfield, Virginia, USA

U. Waghmare and E. Kaxiras, Harvard University, Cambridge, Massachusetts, USA

(20) 8:45-9:15 pm Directional Atomic Bonds in MoSi₂

K. Tanaka, H. Inui, M. Yamaguchi, and M. Koiwa, Kyoto University, Kyoto, Japan

(21) 9:15-9:45 pm Atomistic Potentials for the Molybdenum-Silicon System

M.I. Baskes, Sandia National Laboratories, Livermore, California, USA

10:00-11:00 pm **Social Hour**

Thursday, 28 May 1998

7:00-8:30 am **Breakfast**

Session: Fabrication

Chair: K. Jata, Wright-Patterson AFB

(22) 8:30-9:15 am Invited Presentation

An Overview of Powder Processing of Silicides and Their Composites

N.S. Stoloff, Rensselaer Polytechnic Institute, Troy, New York, USA

(23) 9:15-10:00 am Invited Presentation

Phase Identification in Reactively Sintered Molybdenum Disilicide Composites

K.K. Chawla, University of Alabama at Birmingham, Birmingham, Alabama, USA

J. Alba Jr., New Mexico Tech, Socorro, New Mexico, USA

J.J. Petrovic, Los Alamos National Laboratory, Los Alamos, New Mexico, USA

R.L. Hexemer, Advanced Refractory Technologies, Lockport, New York, USA

**(24) 10:00-10:30 am The Improvement in Mechanical Properties of MoSi₂ Through
In Situ Crystallization of Grain Boundary Silica Glass by the Additions of Refractory
Oxides**

Y. Suzuki and K. Niihara, Osaka University, Osaka, Japan

P.E.D. Morgan, Rockwell Science Center, Thousand Oaks, California, USA

10:30-10:45 am **Coffee Break**

(25) 10:45-11:15 am Preliminary Characterization of Plasma Sprayed MoSi₂/Sigma SiC Fibre Monotapes

A.M. Baker and P.S. Grant, Oxford University, Oxford, UK

R. Castro and H. Kung, Los Alamos National Laboratory, Los Alamos, New Mexico, USA

(26) 11:15-11:45 am Fabrication of Silicide Materials and Their Composites by Reaction Sintering

R. Scholl, A. Bohm, and B. Kieback, Fraunhofer Institute for Applied Materials Research, Dresden, Germany

(27) 11:45-12:15 pm Evaluation of Vapor Deposited Nb/Nb₅Si₃ Microlaminates

A.J. Gavens, D. Van Heerden, and T.P. Weihs, The Johns Hopkins University, Baltimore, Maryland, USA

T. Foecke, National Institute of Standards and Technology, Gaithersburg, Maryland, USA

(28) 12:15-12:45 pm Self-Propagating Formation Reactions in Nb/Si Multilayers

M.E. Reiss, C.M. Esber, D. Van Heerden, A.J. Gavens, and T.P. Weihs, The Johns Hopkins University, Baltimore, Maryland, USA

1:00-2:00 pm **Lunch**

Session: Properties

Chair: W. Simmons, Army Research Office

(29) 2:00-2:45 pm Invited Presentation

Interdiffusion and Diffusion Structure Development in Selected Refractory Metal Silicides

M.A. Dayananda and P.C. Tortorici, Purdue University, West Lafayette, Indiana, USA

(30) 2:45-3:30 pm Invited Presentation

Review of Creep and Fatigue Behavior of Silicides and Their Composites

K. Sadananda and C.R. Feng, Naval Research Laboratory, Washington, DC, USA

R. Mitra, DMRL, Hyderabad, India

S.C. Deevi, Phillip Morris Research Center, Richmond, Virginia, USA

(31) 3:30-4:00 pm Low Temperature Oxidation Behavior of a MoSi₂ Composite

J. Chen, C. Li, Z. Fu, and X. Tu, Chalmers University of Technology, Goteborg, Sweden

H. Arwin, Linkoping Institute of Technology, Linkoping, Sweden

M. Sundberg, Kanthal AB, Hallstahammar, Sweden

R. Pompe, Swedish Ceramic Institute, Goteborg, Sweden

(32) 4:00-4:30 pm Wear of MoSi₂ and MoSi₂-Base Composites by Hard Particles at Room and Elevated Temperatures

D.E. Alman, J.H. Tylczak, and J.A. Hawk, U.S. Department of Energy Albany Research Center, Albany, Oregon, USA

(33) 4:30-5:00 pm Effect of Microstructure on the Indentation Response of MoSi₂

A. Newman, S. Sampath, and H. Herman, State University of New York at Stony Brook, Stony Brook, New York, USA

(34) 5:00-5:30 pm The 1100 °C Isothermal Section of the Ti-Ni-Si Ternary Systems

H.X. Xiaohua, C. Guoliang, N. Kequan, and L. Yimin, University of Science and Technology, Beijing, PR of China

(44) 5:30-6:00 pm Residual Thermal Stresses in MoSi₂-Mo₅Si₃ In-Situ Composites

P. Peralta, R. Dickerson, J.R. Michael, K.J. McClellan, F. Chu, and T.E. Mitchell, Los Alamos National Laboratory, Los Alamos, New Mexico, USA

7:00-8:30 pm Dinner

Friday, 29 May 1998

7:00-8:30 am Breakfast

Session: Applications

Chair: C.A. Sorrell, Department of Energy

(35) 8:30-9:15 am Invited Presentation

Improvement of Mechanical Properties of Si₃N₄/MoSi₂ and Si₃N₄/SiC Composites by Controlling Structure

K. Yamada and N. Kamiya, Toyota Central R&D Laboratory, Aichi-ken, Japan

(36) 9:15-9:45 am Durability of Molybdenum Silicide Materials in Molten Alkali Borosilicate Glass

Y.S. Park, R.G. Castro, K. Hollis, J.J. Petrovic, and D.P. Butt, Los Alamos National Laboratory, Los Alamos, New Mexico, USA

(37) 9:45-10:15 am HSR/EPM Combustor Materials Development Program

D. Brewer, NASA Lewis Research Center, Cleveland, Ohio, USA

10:15-10:30 am Coffee Break

(38) 10:30-11:00 am Oxidation and Hot Corrosion of MoSi₂-Si₃N₄ Base Composites

R.W. Kowalik, Naval Air Warfare Center Aircraft Division, Patuxent River, Maryland, USA

M.G. Hebsur, NASA Lewis Research Center, Cleveland, Ohio, USA

(39) 11:00-11:30 am Low Temperature Oxidation of Fully Dense and Porous MoSi₂

K. Kurokawa, H. Houzumi, and H. Takahashi, Hokkaido University, Sapporo, Japan

(40) 11:30-12:00 am Applications of Molybdenum Disilicide Composites Produced by Plasma Spraying

R.G. Castro, H. Kung, K.J. Hollis, and J.J. Petrovic, Los Alamos National Laboratory, Los Alamos, New Mexico, USA

(41) 12:00-12:30 pm Silicide and Silicide Coatings: Some Specific Features and Application Prospects

M.S. Tsirlin, Ben-Gurion University of the Negev, Beer-Sheva, Israel

**(42) 12:30-1:00 pm Processing and Characterization of Composites Based on
Molysilicides**

S.C. Deevi and A.C. Lilly, Phillip Morris Research Center, Richmond, Virginia, USA

1:00-2:00 pm Lunch

Session: General Discussion

Chair: A.K. Vasudevan, Office of Naval Research

**2:00-3:30 pm General group discussion of needs and directions for high
temperature structural silicides**

3:30 pm Conference Adjournment

**The Proceedings for this Conference will be published in the refereed journal - Materials
Science & Engineering.**

This is a major international conference on Silicides, which Los Alamos has played a central role in organizing. Details of the Conference can be obtained from the following Engineering Foundation Web Site: <http://www.engfnd.org/8as.html>.

IGT Interaction:

**Performance Testing of Silicide and Aluminide Based Materials in a Combustion
Environment - IGT Project No. 65941**

A meeting was held on March 5, 1998 at the IGT Energy Development Center with Richard Castro (LANL), Brian Bartram (LANL), Walter Kunc (IGT), Sreenath Gupta (IGT) and Harry Kurek (IGT) to discuss the performance testing of the U-tube which contained (9) different samples placed on the inside and outside of the U-tube. The following testing methodology was discussed:

1. The radiant U-tube will be installed and mounted 180° to the original mounting plan in order to shorten T/C length within the furnace.
2. A constant furnace temperature operation would be one of the objectives in the test. To simulate a typical operating environment inside of the furnace a number of operating schemes were discussed which included periodically opening of the charge door once or twice per day in order to cycle the burner tube in both a high and lower fire mode.
3. The following operating plan was adopted for the test:
 - the furnace would be operated in an endothermic atmosphere for ten hours a day (Mon thru Fri).
 - the furnace would be operated in a nitrogen atmosphere for fourteen hour during the off shifts (Mon thru Fri).
 - the furnace would operate in a nitrogen atmosphere for forty-eight hours during weekends (Sat through Sun).

4. In the event there is a T/C problem (major temperature difference) at a sample location, a furnace shutdown for T/C replacement will be considered at that time.
5. Several "grab samples" of the endothermic gas at the output of the generator and several "grab samples" of the endothermic gas within the furnace will be taken (simultaneously) once a stable operation has been reached.
6. Manual sampling and analysis of the test tube P.O.C. will be done for the test tube (NO_x , xsO_2 , CO_2 , CO and THC) during low fire and high fire conditions to establish the mean values of emissions.
7. Thermal input into each burner will be determined by gas flow orifice measurement and air flow by calculations from P.O.C.
8. Raw data and reduced data will be part of the report which will be delivered to LANL upon completion of the test. The format is to be a spread sheet format with all parameters/data associated with the test.
9. An assessment of the test and test results will be made at approximately 200 cumulative hours of endothermic atmosphere operation. At this time the inside T/C's will have had 672 hours of exposure to P.O.C. and the outside T/C's will have had 210 hours of endothermic atmosphere and 462 hours of nitrogen atmosphere all at 1800 °F furnace operating temperature.

The IGT radiant tube test was initiated on March 10, 1998 and was completed on March 27, 1998. Results of the test will be reported in the next quarterly report. The radiant tube with the sample holders/samples inside of the IGT furnace is shown in the following figures.

Oxford University Collaboration:

The results of the following investigation "Preliminary characterization of a plasma sprayed MoSi₂/Sigma SiC fiber monotape" which has been done collaboratively with Materials Department at Oxford University, will be presented at the High Temperature Structural Silicides Conference in May, 1998. The following is the abstract of this investigation:

Abstract:

A composite of unidirectional SiC fibers in a MoSi₂ matrix has potential for use in aeroengine components which operate above 1000 °C. Above this temperature the MoSi₂ acts like a ductile metal matrix with good stiffness and strength because of the SiC fiber reinforcement. UK Defense and Evaluation Research Agency Sigma C-coated SiC fibers of 107µm diameter, were cylindrically wound onto a 160 mm diameter mild steel ring at a fibers spacing of 0.2µm. Air plasma spraying (APS) was then used to spray 10-40 µm droplets of molten MoSi₂ onto the rotating fibers preform to give a final monotape thickness of 350-400 µm. Monotapes were removed from the substrate using an acid bath. Cross section samples have been polished and the composite microstructure examined using scanning electron microscopy (SEM) and transmission Electron Microscopy (TEM). The microstructure consists of splat quenched and unmelted MoSi₂ particles, a reaction layer between the fiber C-coating and the MoSi₂ matrix as well as some cracking at the point of minimum separation between adjacent fibers.

Fibers have been extracted from the matrix material by heating in air at 500 °C for 2 hr which caused the matrix to crumble by pest oxidation, an intermediate temperature effect in MoSi₂. SEM examination of these fibers showed that in general APS damage was minimal although isolated regions of the fibers showed coating damage, with a maximum penetration depth of ~ 1µm. Subsequent uniaxial tensile and bend strain to failure testing of individual SiC fibers showed that the bend strain to failure fell by ~12% during APS and subsequent oxidation removal of the matrix, whereas mean tensile strength increased by ~8%. For both bend and tensile data the Weibull modulus, or spread of values, fell from ~25 to ~10 indicating an increase population of damaged fibers produced by APS. Hypotheses to explain the relationship between APS deposition conditions, fiber extraction methods, possible interfacial chemical reactions, considerations of thermal residual stresses and fiber property changes have been developed. The following is a figure of the MoSi₂/Sigma SiC fiber monotape produced by plasma spraying.

Combustion Tech Inc. (CTI):

Testing of the plasma spray-formed periscope site-tube has been delayed and is expected to be performed during May. The performance test will require the insertion of the MoSi_2 periscope site tube into the glass melter which will cause a thermal shock of the tube from room temperature to approximately 3000 °C in a 30 second time period. Removal of the site tube, which can result over a 5 second time period can also thermal shock the site tube. The following is a figure showing the spray-formed periscope site tube and its industrial use for close-circuit monitoring of glass melters.

Closed-Circuit Monitoring Systems for Glass Melters

MoSi₂-based Periscope Sight Tubes:

- Combustion Tec Inc.,
- Enhanced lens cooling system
- Heat resistant monitoring system

Johns Manville CRADA:

A meeting was held on February 10, 1998 with Dr. Walter Johnson from Johns Manville to discuss the change of the Joint Work Statement as a result of a 40% budget cut in the Silicide program at LANL. Funding to support the CRADA for FY98 was reduced from \$500K to \$250K. The meeting focused on identifying critical activities/tasks which would be most important in meeting the objectives of the CRADA. Three areas of the CRADA were identified for investigations to be performed during FY98:

- Coatings to improved refractory performance
- Material/glass compatibility and performance testing
- Component fabrication

The following are actions which will completed in FY98 to support the change in the Joint Work Statement:

- Receive from Walt Johnson new silica bricks which will be subsequently plasma sprayed with a $\text{MoSi}_2/\text{Al}_2\text{O}_3$ composite coating and field tested at Johns Manville.
- Johns Manville will supply all details associated with the testing of the coated refractory bricks including; atmosphere conditions, control samples and placement of test bricks.

- Johns Manville will supply LANL with Stellite rods which have been coated with silicides. Glass compatibility studies will be investigated at LANL.
- Johns Manville will supply LANL with a drawing of a component to be fabricated from a MoSi_2 -based composite. LANL will investigate industry capabilities to fabricate components 8.00" - 9.00" diameter. This size is beyond the current capability at LANL.

Glass Corrosion Studies:

Glass corrosion studies this quarter focused primarily on the long term durability of Mo_5Si_3 , machinable Al_2O_3 (Cotronics, Brooklyn, NY), Fe-Cr-Si alloy, and MoSi_2 plasma sprayed coatings. In addition, we completed TEM investigations of the MoSi_2 /glass and Mo_5Si_3 /glass interfaces above, at, and below the glass line.

Static corrosion studies were done at 1300°C for 5, 12, 24, 48, 96, 168, and 340 hours. Dimensional analyses of many of the corrosion coupons are in progress and will be reported next quarter. In general, MoSi_2 appears to perform better than all of these materials, with the exception of Mo_5Si_3 , which performs relatively similarly to MoSi_2 below the glass line. However, above the glass line Mo_5Si_3 oxidizes catastrophically.

TEM results completed on MoSi_2 and Mo_5Si_3 at the various locations in the melt have assisted us in fully elucidating the mechanisms of corrosion that we hypothesized in previous quarterly reports. Two major publications are nearly complete^{1,2} the later detailing these microstructural studies. The TEM studies illustrate that the mechanisms of corrosion vary with location in or above the glass melt due to changes in the activity of oxygen and the propensity for dissolution of Si. It has been demonstrated that above the glass line MoSi_2 forms a relatively protective layer of silica. Below the glass line, Si depletion through dissolution, leads to the development of a protective Mo-rich layer comprised of Mo_5Si_3 , and discontinuous Mo oxides, Mo particles, and crystalline SiO_2 . At the glass line neither a protective silica layer nor Mo-rich layer forms due to the combined effect of rapid dissolution of SiO_2 and simultaneous vaporization of Mo products caused by the locally high oxygen activity. Based on these observations, a phenomenological model has been developed that describes the overall processes of corrosion at each of the three locations. Some of the more interesting features observed by TEM are shown in Figs. 1-4.

References:

1. Y. S. Park, R. G. Castro, K. J. Hollis, J. J. Petrovic, and D. P. Butt, "Corrosion of Molybdenum Disilicide Materials in Molten Alkali Borosilicate Glass, I: Kinetics and Comparison to Other Refractories," to be submitted to *J. Am. Ceram. Soc.*, 1998.
2. Y. S. Park, R. D. Field, and D. P. Butt, "Corrosion of Molybdenum Disilicide Materials in Molten Alkali Borosilicate Glass, II: Microscopy and Mechanisms," to be submitted to *J. Am. Ceram. Soc.*, 1998.

Figure 1. TEM image showing a complex layered structure composed of Mo_5Si_3 , Mo_3Si , α -Mo, and SiO_2 after exposure of MoSi_2 at 1300°C , 48 hours below the glass line.

Figure 2. TEM image of large faceted MoO_2 particles observed near the MoSi_2 /glass interface after 1300°C , 48 hours below the glass line.

Figure 3. TEM image of a region near the MoSi_2 /glass interface showing cristobolite growth on Mo_5Si_3 . The sample was exposed below the glass line for 48 hours at 1300°C .

Figure 4. TEM micrograph of ternary and quaternary Mo-rich phases identified in the molten glass just away from the MoSi_2 /glass interface.

Joining MoSi_2 to Stainless Steel Using Amorphous Metal Interlayers:

Joining of MoSi_2 still continues at a limited level. Studies completed in the past period illustrated that both the temperature and time were critical for successful fabrication of MoSi_2 /amorphous metal/stainless steel joints. This was attributed to adequate melting and flow characteristics of the metallic-glass braze. At higher temperatures (1050°C), shorter joining times were sufficient, while at lower temperatures ($900\text{--}1000^\circ\text{C}$) joining times in excess of 60 minutes were necessary. In an attempt to duplicate real life joining scenarios where a MoSi_2 ceramic joining tube might be brazed to a stainless steel tube, concentric ring shaped samples, 3 mm in thickness were made. The gap between the outer stainless ring and inner MoSi_2 tube was either 25 or 50 micrometers,

corresponding to one or two foil thicknesses (brazing foil thickness is approximately 25 microns). These joints were completed by heating the assemblies to 1050°C for 60 minutes. The completed joints can be seen in Figure 5.

These joints are going to be used for push out testing to evaluate the joint strength. Simultaneously, compression tests will also be performed on C-ring or O-ring samples to evaluate joint strength. FEM modeling is underway to determine the residual stresses in these assemblies. These calculations will be validated using neutron and x-ray diffraction.

Figure 5. MoSi₂ joined to stainless steel using amorphous metal interlayers.