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FOREIGN TRIP REPORT

ORNL/FTR-3509

DATE: 21 December 1989

SUBJECT: Report of Foreign Travel of M. G. Jenkins, Development Staff Member, Metals and Ceramics Division

TO: Alvin W. Trivelpiece

FROM: M. G. Jenkins

PURPOSE: The traveler visited Japan to attend and to present a paper at the "First Japan International SAMPE Symposium and Exhibition" during which key aspects of the processing, fabrication, and applications of advanced materials, including ceramics, were emphasized. The Institute of Industrial Science, University of Tokyo, was visited in Tokyo to review fabrication and mechanical properties testing of filament-reinforced/carbon matrix composites. At the Toyohashi University of Technology in Toyohashi, high-temperature fracture testing methods and various aspects of the micro-mechanical analysis of fracture in ceramics were discussed. Methods of tensile testing ceramics were examined at three laboratories: the Government Industrial Research Institute and the Japan Fine Ceramics Center, both in Nagoya, and the Kyocera Central Research Laboratory in Kokubu. The JFCC user program, similar to the user center at the High Temperature Materials Laboratory, was also investigated.

SITES VISITED:

11/25-27	Institute of Industrial Science Tokyo, Japan	A. Okura K. Ahlborn Y. Kagawa
11/28-30	First Japan International SAMPE Symposium and Exhibition, Chiba, Japan	
12/01-02	Toyohashi University of Technology Toyohashi, Japan	M. Sakai
	Government Industrial Research Institute Nagoya, Japan	T. Ohji H. Tabata
	Japan Fine Ceramics Center Nagoya, Japan	H. Awaji

12/03-05 Kyocera Central Research Laboratory M. Kaji
Kokubu, Japan H. Miyata

ABSTRACT: The traveler attended the "First Japan International SAMPE Symposium and Exhibition" to present the paper "Empirical and Analytical Determination of the Fracture Resistance of a TiB_2 Particle/SiC Matrix Composite." Key aspects of the conference were on the processing, fabrication, and applications of advanced materials, including high-temperature structural ceramics.

Visits with researchers at the Institute of Industrial Science, University of Tokyo were quite profitable since valuable information and insights were gained in the fabrication of carbon filament composites, mechanical testing methods for brittle materials, and the modeling of fracture mechanisms.

Valuable tours were taken at several important Japanese government-affiliated research centers, which included Toyohashi University of Technology in Toyohashi, the Government Industrial Research Institute in Nagoya, and the Japan Fine Ceramics Center also in Nagoya. These visits revealed the advanced state of tensile testing structural ceramics at elevated temperatures as it now stands in Japan. The committed industrial approach to testing, evaluation, and applications of structural ceramics was revealed at the Kyocera Central Research Laboratory in Kokubu during a very open two-day visit.

TRIP REPORT

This foreign travel involved three major activities:

1. Participation in the "First Japan International SAMPE Symposium and Exhibition."

2. Visits to government-affiliated research centers to observe testing, evaluation, and applications of structural ceramics at elevated temperatures.

3. Visit to an industrial research laboratory to observe testing, evaluation, and applications of structural ceramics at elevated temperatures.

1. "First Japan International SAMPE Symposium and Exhibition," 11/28-30

This was an international conference attended by participants from Japan, the United States, the United Kingdom, the Federal Republic of Germany, France, Belgium, Taiwan, the People's Republic of China, India, and Israel. As befitted the mission of the Society for the Advancement of Materials Processing and Engineering (SAMPE), the sessions covered a number of topics ranging from basic materials research to materials processing to materials applications. Sessions included such themes as polymers and polymeric composites, adhesives, intermetallics, glasses, construction materials, and superconductors.

Of particular interest were those sessions dealing with engineering ceramics, ceramic matrix composites, filaments, carbon/carbon composites, and applications in aerospace and transportation. The traveler presented the paper "Empirical and Analytical Determination of the Fracture Resistance of a TiB_2 Particle/SiC Matrix Composite" at a session on ceramic matrix composites. A copy of the proceedings of this conference which contains all the papers presented in their entirety is retained by the traveler.

2. Visits to Government-Affiliated Research Centers, 11/25-27, 12/02-03

At the Institute of Industrial Science (IIS), University of Tokyo in Tokyo, Professor Akimitsu Okura and Dr. Klaus Ahlborn hosted the visit. Professor Okura is the head of the Okura laboratory where a novel hot-pressing method has been developed for fabricating filament-reinforced/carbon matrix composites. Dr. Ahlborn, an invited researcher from FRG, is developing testing methods for determining the mechanical properties of the composites as functions of temperature. Professor Okura was quite open and enthusiastic about his fabrication process which has been licensed to a Japanese start-up venture, Across Company, Limited. Of particular interest to Professor Okura are methods for making high-temperature brake linings which are being investigated by a representative of Akebono Brake Company who is working full-time at IIS.

Dr. Ahlborn completed his doctorate in materials science at the Kernsforschungszentrum Karlsruhe, Institut fur Material- und Festkorpperforschung IV, in Karlsruhe, FRG, partially under the direction of Professor D. Munz. Dr. Ahlborn is developing specialized grips for testing the filament-reinforced composites in uniaxial stress fields. Unfortunately, he is meeting with limited success, since non-gage section failures are still a problem. In a related area, Dr. Ahlborn is video-recording internal crack growth mechanisms (e.g., crack branching, micro-cracking, grain-bridging) in a transparent zirconia. The real-time, visual observation of propagating cracks is providing both new and corroboratory evidence for fracture models.

Also at IIS is Professor Yutaka Kagawa, who is conducting research on the fracture of filament-reinforced/brittle matrix composites. Dr. Kagawa has developed a number of hybrid numerical/analytical models for describing the fracture process in these materials. However, unlike some analysts, he is also conducting empirical tests on model material systems to verify the modeling. He is finding good agreement with his fracture models and is now working to apply the concepts to design-life predictions.

At the Toyohashi University of Technology in Toyohashi, Professor Mototsugu Sakai provided an excellent overview of the work being conducted on monolithic and composite structural ceramics in the Department of Materials Science. Of particular interest were techniques which involved laser/optical methods with reported displacement resolutions of 5-10 μm at temperatures of 1000°C-1400°C. Professor Sakai is actively pursuing methods of characterizing the fracture resistance in these materials, most notably in regard to the R-curve behaviour. He has been developing a tensile grip system in order to conduct fracture tests in a uniform stress field. However, limited success has been forthcoming in this regard, although the use of a "crack-stabilizer" on ceramic compact tension (CT) specimens has allowed the study of stable crack-growth behaviour using simple straight-through cracks as opposed to chevron-notch cracks. He has also shown good progress in applying essential work-of-fracture concepts to filament-reinforced materials using reduced-gage-section bend bars.

At the Government Industrial Research Institute (GIRI) in Nagoya, Dr. Hideyo Tabata and Mr. Tasuki Ohji demonstrated the tensile testing facilities which have been developed to conduct research on tensile strength, slow crack growth, and fracture characteristics of ceramics at elevated temperatures. GIRI has contracted to do basic research for Japan's Ceramic Gas Turbine (CGT) project, and the work of Mr. Ohji is essential to the fundamental understanding of the behaviour of ceramics under long-term exposure to the operating conditions of the CGT. Mr. Ohji's fast-fracture tensile specimen has been copied by some other researchers (notably Professor R. Tressler at the Pennsylvania State University) and has been shown to be suitable for evaluating the strain-rate (stress-rate) dependence of the stress-strain curves as well as the ultimate

tensile strengths at elevated temperatures. In this specimen, a flat plate is first machined with a formed grinding wheel to produce a rough "dog-bone" shape. The radii of the ends of the "dog-bone" are 6.0 mm and contact either 5.5 mm or 6.0 mm radius pins in SiC clevis load fixtures. The 5.5 mm radius pin is used to provide point contact in the specimen radius to minimize bending components due to friction during fast fracture tests. The 6.0 mm radius pin is used to provide uniform load distribution during the long-term creep tests. Percent bending values of about 2.0 were reported by Mr. Ohji, which he noted were sufficiently low for his satisfaction.

An interesting variation of this fast-fracture specimen is the creep (or stress rupture) specimen with integral gage-section flags. The specimen is being used in conjunction with Zimmer optical extensometers, NEMS dead-weight creep frames, and NEMS resistance-heated furnaces to measure the time-dependent deformation of potential CGT materials at 1400°C for 10,000 h. Excellent, overall, long-term stabilities with displacement resolutions of less than 10 μm are being reported.

At the Japan Fine Ceramic Center (JFCC), Dr. H. Awaji served as host. The JFCC is a user and research center similar to the HTML at ORNL. Approximately 45 people funded by industry, local government, and academe [11 billion yen (operating) and 7 billion yen (capital) for FY 1989] conduct basic and applied ceramics research, test development and standardization, data base generation, and basic education and training on ceramics.

The facilities are state-of-the-art with a large portion of the equipment of Japanese origin, including custom hot presses, hot isostatic presses (HIP), sintering furnaces, chemical analyses, Auger electron spectroscopy, 400 keV transmission electron microscopy (TEM), secondary ion mass spectrometry (SIMS), ultrasonic machining, diamond grinders, slicers, etc. Of particular interest was the emphasis on nondestructive evaluation (NDE) techniques such as submerged acoustic microscopy and scanning acoustic microscopy. In conjunction with a private company, NDE data bases are being developed on various components via micro-focus X-ray and computed tomography. The belief is that, by using a multiple-system approach, proof-testing of ceramic components can be eliminated. Interestingly, the facilities seemed under-utilized with large amounts of empty space, many machines standing idle, and few researchers in evidence.

The high-temperature tensile testing facilities are still being developed, although Dr. Awaji was quite enthusiastic and open about a JFCC-developed, room-temperature arrangement. In this system, a button-head specimen with 20.0-mm-diameter heads and a 6.0-mm-diameter gage section is used. A two-piece, conical collet interfaces with the specimen at a flat surface on the underside of the button-head. The conical collet is loaded through a series of well-lubricated fixtures mounted on a series of precision ball bearings. During a test, the strain-gaged specimen is preloaded to determine the initial percent

bending. A system of set screws on the grip is then adjusted to produce the minimum achievable bending strain in the specimen. During a series of room-temperature tests on an NGK silicon nitride with an ultimate tensile strength of about 1000 MPa, percent bending ranged from 0.8 to 4.0 with a mean of about 2.0. In two separate batches of test specimens, there were two button-head failures in five tests and one button-head failure in nine tests. The button-head failures were attributed to poor machining of the specimens. Costs (material and fabrication) of the specimens were quite reasonable at 80,000 to 100,000 yen ($515 to $715) each. Drawings of both the gripping system and the specimen type are retained by the traveler.

The high-temperature tensile testing facilities are being developed around the Instron Super Grip hydraulic couplers, the Instron short furnace, and the Instron water-cooled, straight-collet, button-head specimen grip, all similar to those presently installed at the HTML. At the time of the visit, the Instron components had not been installed but had been arranged on a table for display. Dr. Awaji seemed somewhat skeptical of the Instron advertising claims for this system but was willing to try the system with an open mind.

3. Visit to Kyocera Central Research Laboratory, 12/03-05

At the Kyocera Central Research Laboratory in Kokubu, presented an impressive picture of Kyocera's commitment to ceramic technology and applications. Presentations were given by members of Kyocera's Advanced Ceramic Department, the Materials Development Division, and the Non-oxide Division. Topics included: mechanical properties (stress-temperature ranges, Weibull strengths, fracture toughness, slow crack growth behaviour, stress rupture, creep resistance, and oxidation characteristics) of SN220M, SN235, and SN251 monolithic silicon nitrides. Of particular interest were measurement techniques such as those dealing with elastic constants, crack growth resistance, tensile strength, and creep testing.

The tensile testing facilities were clean, well organized, and well maintained with little effort or expense wasted on "unnecessary" sophistication. Kyocera uses a variation of a powder grip system and a cylindrical, button-head specimen similar to that used at ORNL. Few grip-related failures have been reported and the percent bending is over a range of 0.5 to 6.0, which is also similar to that found at ORNL for solid collet systems. Some of the data presented showed the relationship between tensile strength and percent bending at failure for 31 tests of SN220M at room temperature. Interestingly there was no clear correlation between high strength and low bending. Theoretical Weibull analyses conducted for the effects of both volume and bending provided two important results:

(1) There is a predictable relation between strength and test volume such that strengths for various volumes can be extrapolated from test data.
(2) Percent bending of up to 5.0 can be tolerated in a tensile test with little decrease (about 2.5%) in the determined characteristic strength; however, at

values greater than 5.0 the amount of decrease is dramatically increased (i.e., 5% bending is an acceptable upper limit for tensile strength testing).

These conclusions are similar to those drawn at HTML in an investigation of grip designs for tensile specimens. It should be emphasized, however, that no conclusions have yet been drawn regarding the effects of bending on the measurement of displacement (or strain) in tests such as creep testing at elevated temperatures. Obviously, both the material behaviour as well as the displacement measurement will be affected if the strain distribution over the specimen cross-section is not uniform due to undesirable bending components.

Kyocera also showed the importance of surface healing as opposed to surface finish on the measured strength. Using modulus-of-rupture (MOR) tests of SN220M at room-temperature, it was demonstrated that, for postmachining surfaces with $R_a > 20$ μm, the MOR values decreased up to 30% for the untreated specimens while the heat-treated specimens showed no decrease in strength. The heat treatment consisted of an anneal in ambient air for a total of 4 h, in which there was a ramp of 1 h from 20°C to 1000°C followed by a hold at 1000°C for 3 h. While this study was conducted on only one material, which incidentally does show a moderate degree of oxidation behaviour, it does demonstrate the importance of alleviating surface damage due to the fabrication process.

SUMMARY

The "First Japan International SAMPE Symposium and Exhibition" provided an excellent forum at which to exchange ideas and to gain insight into the direction of research programs on an international scale. Some of the work, especially in the development and application of advanced monolithic ceramics, is encouraging for the heat engine programs especially in the United States. The limited usefulness of the ceramic composites is becoming more apparent as problems concerning processing, fabrication, and predictable behaviour continue to raise questions.

The tours of various laboratories and research centers were highly profitable. Of particular interest for the traveler were the various methods of tensile testing ceramics at elevated temperatures. In this regard, Kyocera and JFCC have systems similar to those at ORNL, although Mr. Ohji's systems at GIRI bear further investigation because of his diversity of successful tests. Failure of the specimen in the grip area seems to be a common problem for all researchers and is not limited to the systems currently in use at ORNL. The whole question regarding minimum bending strain is still open and has only been partially solved and/or addressed by the Japanese researchers.

The overall impression of the Japanese commitment to the development and application of structural ceramics is quite good. There is a strong, concerted effort by industry, backed by the government, to become proficient in the

development, fabrication, and use of advanced ceramics. Obviously, this is not a short-term interest, apparent from the size of the facilities, number of people, and amount of money devoted to this effort.

APPENDIX A

Itinerary

11/24-25	En route from Oak Ridge to Tokyo.
11/25	Meeting with K. Ahlborn, Institute of Industrial Science, Tokyo.
11/26	Weekend.
11/27	Meeting with A. Okura, K. Ahlborn, Y. Kagawa, Institute of Industrial Science, Tokyo.
11/28	Attended sessions of "First Japan International SAMPE Symposium and Exhibition," Chiba.
11/29	Presented paper "Empirical and Analytical Determination of the Fracture Resistance of a TiB_2 Particle/SiC Matrix Composite," at the "First Japan International SAMPE Symposium and Exhibition," Chiba.
11/30	Attended sessions of "First Japan International SAMPE Symposium and Exhibition," Chiba.
12/01	Meeting with M. Sakai, Toyohashi University of Technology, Toyohashi. Meeting with T. Ohji, H. Tabata, Government Industrial Research Institute, Nagoya. Meeting with H. Awaji, Japan Fine Ceramics Center, Nagoya.
12/02	Meeting with M. Sakai, Toyohashi University of Technology, Toyohashi.
12/03	En route from Nagoya to Kagoshima.
12/04	Meeting with M. Kaji, H. Miyata, Kyocera Central Research Laboratory, Kokubu.
12/05	Meeting with M. Kaji, H. Miyata, Kyocera Central Research Laboratory, Kokubu. En route from Kagoshima to Tokyo.
12/06	En route from Tokyo to Oak Ridge.

APPENDIX B

List of Persons Contacted to a Significant Extent

Organization	Name	Title
Institute of Industrial Science, University of Tokyo	K.F.H. Ahlborn	Invited researcher
	A. Okura	Professor, Head of Okura laboratory
	Y. Kagawa	Professor, Head of Kagawa laboratory
Toyohashi University of Technology	M. Sakai	Professor, Department of Materials Science
Government Industrial Research Institute, Nagoya	T. Ohji	Research Engineer, Ceramic Engineering
	H. Tabata	Director, Research Planning Office
Japan Fine Ceramics Center	H. Awaji	Acting Section Manager, Engineering and Mechanics of Ceramics Division
Kyocera Central Research Laboratory	M. Kaji	Research Engineer, Computer Aided Technology Division
	H. Miyata	Manager, Computer Aided Technology Division

APPENDIX C

Literature Acquired

1. *Proceedings of First Japan International SAMPE Symposium and Exhibition,* Chiba, Japan (November 28-30, 1989).

2. K.F.H. Ahlborn et al., "Crack Growth Resistance of Unidirectional Carbon Fiber/Carbon Matrix Composites," preprint of paper submitted to the American Ceramic Society (1989).

3. K.F.H. Ahlborn et al., "Observation of the Influence of Microcracks on the Crack Propagation Inside of Transparent ZrO_2," preprint of paper submitted to the American Ceramic Society (1989).

4. Agency of Industrial Science and Technology, MITI, "Government Industrial Research Institute, Nagoya," overview literature (1989).

5. New Energy and Industrial Technology Development Organization, "Ceramic Gas Turbine, Moonlight Project," program literature (1989).

6. T. Ohji et al., "Tensile Rupture Strength and Fracture Defects of Sintered Silicon Carbide," reprint from *J. Am. Ceram. Soc.,* 72 (4) (April 1989).

7. T. Ohji et al., "Yielding Phenomena of Hot-pressed Si_3N_4," reprint from *High Temp. Technol.*, 5 (3) (August 1987).

8. T. Ohji, "Towards Routine Tensile Testing," reprint from *Int. J. High Technol. Ceram.*, 4 (1988).

9. T. Ohji et al., "Dynamic and Static Fatigue Strength and Crack Propagation of Engineering Ceramics," preprint submitted to *J. Jpn. Ceram. Soc.* (1989).

10. T. Ohji et al.,"Tensile Strength and Bridging Stress of Whisker Reinforced Silicon Nitride," preprint submitted to *J. Am. Ceram. Soc.* (1989).

11. T. Ohji et al.,"Fracture Energy and Tensile Strength of Silicon Nitride at High Temperature," preprint submitted to *J. Jpn. Ceram. Soc. (*1989).

12. T. Ohji et al., "Dependence of High Temperature Tensile Strength for Hot-pressed Silicon Nitride on Displacement Rate," preprint submitted to *J. Mater. Sci.* (1989).

13. S. Sakaguchi et al., "Slow Crack Growth of Engineering Ceramics Measured by Load Relaxation Tests on Chevron Notched Beam," reprint from *Proceedings of the Thirtieth Japan Congress on Materials Research* (1987).

14. "Japan Fine Ceramics Center," overview literature (1989).

15. "JFCC-Japan Fine Ceramics Center," promotional literature (1989).

16. T. Watanabe et al.,"Evaluation of Fracture Toughness by a Single Edge V-Notched Beam Method," preprint from *Proceedings of the Japan-Korea Ceramics Seminar* (December 1989).

17. H. Awaji, "Tensile Specimen and Grip," engineering drawing (1987).

18. M. Sakai, "Crack Growth Resistance of Ceramics with Crack Stabilizer," reprint from *J. Soc. Mater. Sci. Jpn.,* 38 (424) (1989).

19. M. Sakai et al., "Dimensionless Load-Displacement Relation and Its Application to Crack Propagation Problems," reprint from *J. Am. Ceram. Soc.,* 72 (3) (1989).

20. M. Kaji et al., "Silicon Nitride as Structural Materials," reprint of presentation given at Kyocera Central Research Laboratory, Kokubu, Japan (July 19, 1989).

21. M. Kaji et al., "Evaluation of Techniques of Mechanical Properties," reprint of presentation given at Kyocera Central Research Laboratory, Kokubu, Japan (July 19, 1989).

22. Japanese Standards Association, "Testing Method for Flexural Strength (Modulus of Rupture) of High Performance Ceramics," JIS R 1601-1981, reprint of English translation (1981).

23. Kyocera Corporation, "Human Technology Kyocera," sales literature (1989).

APPENDIX D

Distribution List

1. D. B. Waller, Assistant Secretary for International Affairs and Energy Emergencies, DOE, Washington
2. M. H. Chiogioji, Director, Office of Transportation Systems, Conservation and Renewable Energy, DOE, Washington
3. J. M. Davis, Assistant Secretary, Conservation and Renewable Energy, DOE, Washington
4. A. A. Chesnes, Director, Heat Engine Propulsion Division, Office of Transportation Systems, Conservation and Renewable Energy, DOE, Washington
5. R. L. Egli, Assistant Manager, Energy Research and Development, DOE/ORO
6. D. J. Cook, Director, Safeguards and Security Division, DOE/ORO

7-8. Office of Scientific and Technical Information, P.O. Box 62, Oak Ridge, TN 37831

9. B. R. Appleton
10. R. A. Bradley
11. C. R. Brinkman
12. R. S. Carlsmith
13. M. K. Ferber
14. W. Fulkerson

15-19. M. G. Jenkins

20. D. R. Johnson
21. K. C. Liu
22. J. O. Stiegler
23. V. J. Tennery
24. A. W. Trivelpiece

25-26. Laboratory Records Department

27. Laboratory Records Department - RC
28. Laboratory Protection Division
29. ORNL Patent Section
30. ORNL Public Relations Office
31. M&C Records Office