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ASSESSMENT OF JAPANESE TECHNOLOGY IN ADVANCED GLASS AND CERAMIC FIBERS

DONALD R. MESSIER
CERAMICS RESEARCH BRANCH

June 1992

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US ARMY LABORATORY COMMAND
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ABSTRACT

Summarized herein are the findings from a two month trip to Japan from mid-September to mid-November 1991 to evaluate Japanese technology in oxynitride glasses and fibers and in carbide and nitride fibers and whiskers. The information discussed was obtained through visits to universities, companies, Government institutes, and through attendance at three conferences. It was learned that the development of a process for the production of oxynitride glass fibers is still being actively pursued and that, while high temperature instability problems are well-recognized, the production of carbide or nitride fibers with good high temperature stability is still several years away. Also discussed are new developments in several research areas including ceramic matrix composites, sol-gel technology, ceramic powder preparation, and high strength ceramics.

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INTRODUCTION

Background Information

This report summarizes the results of a two month trip to Japan to assess recent developments in emerging technologies of major significance to the U.S. Army and, to a lesser extent, to the materials community in general. The trip was intended to provide up-to-the-minute information on topics critical to the attainment of improved resin matrix and ceramic composites for armor and heat engine applications.

While the importance of the matrix cannot be ignored, the fiber or whisker phases used in making composites critically affect their properties. On the one hand, high strength, high modulus, oxynitride glass fibers promise to reduce weight in glass fiber-reinforced composites for structural applications such as armored vehicles. On the other hand, the development of ceramic matrix composites has been hampered by the lack of suitable fibers and whiskers. The potential of oxynitride glass fibers and the need for better ceramic fibers have both received a lot of attention in Japan, and Japanese efforts in these technological areas equal or surpass those in any other country. In addition to providing a perspective for evaluating and guiding U.S. Army work in the subject areas considered, the trip was expected to yield new information about advanced materials, as well as techniques for making glass and ceramic fibers and whiskers.

Objectives

The Japanese are on the verge of producing in quantity oxynitride glass fibers that surpass existing glass fibers in stiffness and have the potential to exceed them in tensile strength as well. While the U.S. Army Materials Technology Laboratory (MTL) has an active research effort and strong knowledge base in oxynitride glass and fiber technology, commercialization of that technology has not yet begun in the United States. Information available prior to this trip was insufficient to assess how close the Japanese were to their stated objective of manufacturing oxynitride glass fibers in commercial quantities and to clear up confusion between claimed and actual properties of the fibers in question. The information desired is important to MTL and the U.S. Army for planning future research on oxynitride glass fibers and guidance in letting contracts for manufacturing technology efforts.

A critical problem in the development of fiber-reinforced ceramic matrix composites (CMCs) has been the lack of fibers suitable for the fabrication of such composites. The commercially available fiber used in much of the early work in the fabrication of CMCs is *Nicalon* fiber supplied by Nippon Carbon Company. While nominally silicon carbide, that fiber is well known to be noncrystalline and to contain considerable amounts of oxygen and nitrogen. The oxygen impurities, as well as the presence of free carbon in this fiber and in many other polymer-derived fibers, make them thermally unstable at temperatures several hundred ^{o}C below the high temperatures required for densifying ceramic matrices. While a good number of Japanese companies are in various stages of developing and marketing ceramic fibers for CMCs, information was desired on Japanese progress in resolving this critical issue of thermal stability.

Scope of the Study

As indicated in the Objectives Section above, the technology assessment was limited mainly to oxynitride glasses and fibers and nitride and carbide ceramic fibers. Inevitably, however, both of the subjects are related to composites, and ceramic fiber and whisker making technology are related to ceramic powder technology. The itinerary, therefore, included visits to some installations concerned with those subject areas.

Approach

The main approach to information gathering included visits to companies, research institutes, and universities, as well as attendance at several conferences held in conjunction with the 100th anniversary of The Ceramic Society of Japan. The visits usually included seminars or briefings by the author on his research.

Itinerary

With the exception of a trip to Dow Corning Corporation in early September, all of the activity reported herein occurred between 19 September 1991 and 15 November 1991. Table 1 lists, chronologically, all of the activities that took place in conjunction with the trip, and Table 2 lists, alphabetically, installations visited along with subjects discussed with individuals at those locations. Table 3 is an alphabetized compilation of copies of business cards of people met during the trip. It is hoped that readers will contact the individuals concerned for information beyond what could be provided in this report.

Organization of this Report

In any trip such as this the expectations of what will be gained from a particular visit are not always realized. In addition, topics are often discussed that, while interesting, do not relate to the purpose of the visit. Aside from these considerations, it is evident from Tables 1 and 2 that a detailed description of each visit and conference attended could fill a large volume and probably serve no useful purpose.

In view of the above, the approach taken herein is to summarize topically only the information obtained that directly relates to the objectives of this technology assessment. While some additional information has been included, no attempt was made to make the report all-inclusive. It is hoped that anyone interested in specific subjects included in Table 2, but not further discussed in the report, will contact the author for further information. Similarly, the reports on the conferences contain only general information with specific technical details included under the appropriate topic in the report.

CONFERENCES ATTENDED

Background

Being in Japan during this particular time period provided opportunities to attend several excellent conferences. The Ceramic Society of Japan celebrated its 100th anniversary in 1991 and part of the celebration included hosting several international conferences, all of which were more auspicious than they may have been on another occasion. A problem, however, for an individual with interests in both glass and ceramics was that the glass meeting and one of the ceramic meetings took place concurrently. The result was that the author's time was divided spending one day at the International Conference on New Glasses and the following two days at the Fall Meeting of The Ceramic Society of Japan.

Because of the international participation, the language of all of the conferences was English. This was adhered to pretty well except for a number of poster papers which ranged from all Japanese to somewhere in between. A volume of the conference proceedings was furnished for everything except the poster papers at the ceramic society meeting; only abstracts were included for those papers. As already noted in the Introduction, specific information from individual conferences is included under discussions of specific topics further on in this report.

International Conference on New Glasses (ICNG), Tokyo, 16 October 1991

This conference, while featuring a substantial number of outstanding foreign speakers, was attended mostly by Japanese as were the other two meetings. More than 200 researchers were present which exceeded the expectations of the conference organizer, Professor Sumio Sakka of the Kyoto University Institute for Chemical Research. The lectures included several interesting presentations about the optical properties of glass. In addition to lectures, the program included poster papers presented on each day of the conference making it possible to attend all of the latter in a single day. All in all, it was an excellent conference and well worth attending.

Fall Meeting of The Ceramic Society of Japan and International Symposium on Ceramics, Yokohama, 16 Through 18 October 1991

This was a four day meeting that included a Manufacturer's Exhibition in addition to the Ceramic Society Meeting and International Symposium. The meeting culminated on 18 October 1991 with an awards ceremony and banquet commemorating the 100th Anniversary of The Ceramic Society of Japan; the total attendance was around 1,000. The symposium included 40 lectures, mostly by speakers from abroad, and the Ceramic Society Meeting featured 376 poster papers, all by Japanese. The author focused almost exclusively on the poster sessions which represented a good cross section of contemporary activities in ceramics in Japan.

International Symposium on the Science of Engineering Ceramics, Mikawa-Haitsu, Koda, 21 Through 23 October 1991

This was a two and a half-day meeting in a resort-type setting intended to encourage interaction among the participants. A unique feature of the meeting was the sleeping arrangements, which included up to six participants sharing a room. While the cast of speakers was again international, the majority of the 150 to 175 attendees were Japanese. The meeting had an excellent program consisting of 73 presentations in two concurrent sessions and a poster session comprising 23 papers. As hoped by the organizers, the meeting did provide ample opportunities for personal contacts and informal discussions with other attendees.

OXYNITRIDE GLASSES AND GLASS FIBERS

Background Information

One principal objective of this trip was to assess the state-of-the-art of oxynitride glass and fiber technology in Japan. As seen in the Shimadzu Oxynitride Glass Fiber Section below, this objective was met successfully by visits to a number of key people, as well as by attendance at the International Conference on New Glasses.

Shimadzu Oxynitride Glass Fiber

As noted in the Introduction, a goal of this technology assessment was to find out as precisely as possible how much progress the Japanese, more specifically the Shimadzu Corporation, have made in oxynitride glass fiber development, what the properties of the fiber are (versus what they appeared to be from conflicting available information), and what their plans are, if any, to manufacture the fiber.

Visits with Professor Sakka of the Kyoto University Institute for Chemical Research and Professor Soga of Kyoto University helped explain their connections with the Shimadzu oxynitride glass fiber work. While Sakka and Soga are on different campuses of the university (about a one hour train ride apart) they have been close collaborators for many years and have similar, although separate, research interests. Shimadzu (primarily an instrument manufacturer) got started on the oxynitride glass fiber program by contacting Professor Sakka and asking what his suggestions would be for a new venture in the materials area. Sakka suggested two possibilities: oxynitride glass and sol-gel. Shimadzu chose oxynitride glass. In the early stages of Shimadzu's work (estimated to have begun six or seven years ago) Soga served Shimadzu as a consultant to help them get started. Although neither Sakka nor Soga currently consult with the company, both showed the author spools of oxynitride glass fiber that Shimadzu had given them very recently. The fiber looked light-colored and clear, and it was noted by Sakka that two years ago the Shimadzu fiber was black in color (suggesting the presence of a lot of metallic impurities).

The visit to Shimadzu combined with attendance at a poster paper at the International Conference On New Glasses (ICNG) given by Minakuchi (the leader of the fiber development group) cleared up much of the confusion concerning the type of oxynitride glass fiber that Shimadzu is actually producing. Some of their earlier claims (in the news media and in their U.S. Patent) indicated properties far superior to what they are now actually making on a development scale. They showed the author a spool of fiber that was estimated to contain a kg or so similar to what Sakka and Soga had shown earlier. The fiber is being made in quantity, with tows being drawn from a molybdenum container with 50 to 100 bushings. No process details were provided or equipment shown; the meeting was held in a conference room at Shimadzu headquarters.

The Shimadzu fiber is made from a Ca-Mg-Si-Al-O-N glass containing 12 wt% N and having a density of around 3 g/cc. When asked, they stated that the Young's modulus of the fiber that they are making in quantity is 115 GPa, although they claim values up to 140 GPa for other compositions. These figures compare to earlier claims in their patent and in a press release several years ago of elastic moduli up to 200 GPa or more which is incredibly high for a glass. What they are now claiming is not unreasonable and is similar to MTL results for Mg-Si-Al-O-N glass fibers containing considerably less nitrogen. They report tensile strength values in the range of 3 GPa to 4 GPa for their fiber which is respectable but far less than their original claims. Although they were asked about fiber testing, language difficulties made it unclear whether or not the values reported were for pristine fibers. When asked for samples of fibers for testing, they refused stating they were concerned that the properties might not be as good as reported.

The meeting at Shimadzu was attended by six staff engineers, all of whom seemed to be involved in the project. It is likely that there are additional technicians, etc., who were not present. From this meeting, it is obvious that Shimadzu has had (and continues to have) a substantial effort on oxynitride glass fibers. For example. they showed (at a distance) a copy of a phase diagram of their oxynitride glass system that was the result of over two years of daily experiments. They concluded from this study that close control of the melting temperature was very critical to making clear glass and, consequently, high strength fiber. They also hinted (surprisingly, in view of the proprietary nature of what they are doing) that the oxynitride phase diagram closely resembles the known phase diagram for the Ca-Mg-Si-Al oxide system.

When shown a small sample of Mg-Si-Al-O-N glass fiber from MTL, they were impressed by its clarity and said that they were unable to make glasses in that system because of large losses of MgO during melting. Judging from our experience, it is likely that their problem owed to the particular MgO that they used and not a basic problem with that particular oxide.

This meeting clearly answered, affirmatively, the question about their plans to manufacture and market their oxynitride glass fiber. Despite that, however, they seemed a little uncertain as to what that market is. An immediate application that they see for the fiber is in metal matrix composites and they showed an Al alloy matrix composite specimen reinforced with their oxynitride glass fibers.

NIRIM/Colloid Research Institute Sol-Gel Derived Oxynitride Glass and Ceramic Fibers

Immediately prior to this trip, the author learned of a new type of silicon oxynitride glass fiber derived via the sol-gel route and said to contain far greater amounts of nitrogen than fibers prepared by other means. While on this trip, a new announcement described a ceramic (*sialon*) fiber prepared by the same technique. The process was devised jointly by the Colloid Research Institute and the National Institute for Research in Inorganic Materials (NIRIM). Before the author's trip to Japan, a co-inventor of the process, Mr. M. Sekine, had already returned to his company, the chemical division of Nippon Steel, from a stint at Colloid Research Institute where the processing research was done. While Sekine was thus unavailable for a visit, a talk with the other co-inventor, Dr. M. Mitomo of NIRIM, provided additional useful information on this development.

According to Mitomo the announcement that came out was derived from a newspaper article and was not necessarily scientifically accurate. While the article claimed that the fiber was transparent, it is at best translucent because the requirement for nitriding the material in ammonia requires that it contain fine porosity which remains in the fiber after processing. Such porosity will also limit fiber tensile strength, estimated by Mitomo to be around 1 GPa for the sol-gel oxynitride glass fiber. This value is far lower than the fiber tensile strengths in the 3 GPa to 5 GPa range obtained for MTL and Shimadzu oxynitride glass fibers.

Also not stated in the newspaper article is that the reported results are for a few fibers made by a batch process consisting of heating short lengths of fiber in ammonia for times of four to eight hours. At this stage it is obvious that a lot of study would be required to develop a process for making a continuous fiber. Furthermore, such fibers would be low in strength and probably large in diameter (25 μm to 30 μm) compared to the Shimadzu and MTL fibers (10 μm to 15 μm). Although the sol-gel process, therefore, does not seem to be competitive with other processes for making oxynitride glass fibers, it is an impressive technological feat that may certainly have applications to the fabrication of high nitrogen content oxynitride glasses.

It also appears that Nippon Steel sees no market for a new fiber of this particular type at present and will, therefore, not develop it further in the near future.

Bulk Oxynitride Glasses

Interesting and relevant new information on bulk oxynitride glasses was obtained from Mr. H. Unuma of the Government Industrial Development Laboratory (GIDL) in Hokkaido; both from his paper presented at ICNG, and from a visit to his laboratory. Unuma reported on some studies in conjunction with coworkers at Hokkaido University on the molecular dynamics of the structure of a sodium silicate-type oxynitride glass. Contrary to what might be expected, his results showed the average coordination number of nitrogen in the glass to be closer to two than the expected value of three; i.e., each nitrogen atom coordinated to three silicon atoms. Also discussed in the paper were results of the dependence of glass properties on nitrogen content, information relevant to optimizing glass composition. Studies are also in process at GIDL on the synthesis of silicon nitride and silicon carbide powders by plasma-assisted and laser-assisted CVD. Interestingly, Unuma reported that he had made a perfectly clear piece of sodium silicon oxynitride glass with some white silicon nitride powder that had been made in-house at GIDL. Moreover, he stated that the glass remained clear after remelting.

Despite having been very active in the past in oxynitride glass work, Professor A. Makishima of the University of Tokyo has little current activity in the area. His two colleagues who are performing the oxynitride glass research that is presently going on were both absent at the time of my visit and little was learned about their current oxynitride glass activities. Makishima did report, however, that they had obtained some disappointing alkali corrosion resistance results for some sodium silicate oxynitride glasses. That result may have had more to do with the particular glass composition than with oxynitride glasses in general; other oxynitride glasses show excellent corrosion resistance.

Professor Tsukihashi and colleagues, also at the University of Tokyo, have conducted some very useful and relevant work on the high temperature chemistry of oxynitride glasses but their main interests seem to be in other systems. Despite a recent publication on the topic, they do not seem to be planning further research on oxynitride glass systems.

In the recent past, much of the Japanese research in oxynitride glasses was conducted by Professors Sakka and Soga of Kyoto University. While both retain interest in the subject, neither seems to have much current research on oxynitride glass systems.

CERAMIC FIBERS AND COMPOSITES

Background Information

The objective of this section of the technology assessment was to obtain information on new developments in silicon carbide and silicon nitride fibers that are suitable for the fabrication of ceramic matrix composites (CMCs). It turned out that the itinerary planned for this part of the trip was far too ambitious and the number of visits had to be reduced from what was originally planned. The visits that were made, however, together with the conferences attended gave at least a representative look at what is happening in this technological area.

Ceramic Fibers

It was found from this survey that a key problem in the fabrication of continuous fiber CMCs; i.e., the requirement for fibers with good high temperature stability, is well recognized and the subject of considerable current research. It should be added that the Japanese work is in process despite the absence of a Japanese commercial market for such fibers. While the lower grade silicon carbide fiber; e.g., *Nicalon*, has found a market niche the author was often asked what was known about the market for high quality ceramic fibers and where ceramic composites were being used.

Several interchanges with Professor Kiyohoto Okamura of Osaka Prefecture University indicated that he remains very active in research on polymer-derived ceramic fibers. A paper presented by Okamura and coworkers at the Ceramic Society of Japan Meeting discussed in detail work on the pyrolysis and high temperature stability of Ube's *Tyranno* Si-Ti-C-O ceramic fiber. Another paper presented by Okamura at the Engineering Ceramics Meeting dealt with the effect of oxygen on the pyrolysis of polymer-derived Si-C-O (*Nicalon*) fibers. Conversations with Okamura indicated that he is very interested in the high temperature thermal stability of these types of fibers and is also involved in efforts to find new methods of curing polymer-derived fibers to improve their high temperature stability. In addition to his collaboration with at least two companies (Nippon Carbon and Ube) making fibers, he is conducting in-house research on the spinning of fibers from commercially obtained polymer precursors.

A trip to Dow Corning provided an interesting and useful status report on their work on improved HPZ and PCS polymer-derived nitride and carbide fibers. While Dow Corning markets Nippon Carbon's *Nicalon* silicon carbide fiber in the U.S., they are also developing their own ceramic fibers. Dow Corning's HPZ fiber is a Si-N-C-O composition similar to *Nicalon* but with better high temperature properties. The HPZ fiber, however, is rather high in oxygen, as are all similar fibers, and subject to the same high temperature thermal decomposition problems as the other fibers of this type. Fibers derived from the PCS precursor, on the other hand, can be made as low oxygen content (<0.1 w/o), stoichiometric, crystalline SiC, retaining the excellent room temperature and high temperature mechanical properties of silicon carbide. While the production of short lengths (100 mm) of small diameter (8 μm to 10 μm) fiber with excellent properties has been demonstrated, further process development is needed to produce continuous fiber.

Some very impressive work on the development of high strength BN fibers from borazine is being done by Professor Yoshiharu Kimura of the Kyoto Institute of Technology. He has made fibers with tensile strengths of 1 GPa and belie'ves they have the potential to go as high as 2.5 GPa. The fibers also have excellent high temperature strength and high temperature stability. In fact, Kimura claims that the fibers are usable to 2000^{0}C; he has also made BN/BN composites by techniques similar to what are used to make the fiber and indicates that Mitsubishi-Kashen and Rhone-Poulec are both interested in commercializing the development. Because of the need for a weak interface in CMCs and because BN is compatible with so many ceramic materials, BN fibers appear very promising as candidate reinforcement materials for the fabrication of such composites, and Kimura's work deserves close watching by ceramic composite researchers.

An interesting alternative approach to the forming of silicon carbide fibers was discussed by R.S. Storm of Carborundum Corporation at the Engineering Ceramics Meeting in Koda. They report making strong (1.25 GPa), 99% dense fibers with good high temperature properties by sintering of particulate fiber preforms. They noted they can make fiber as small as 25 μm in diameter using their present process and believe they could go as low as 15 μm.

A visit at Nippon Carbon Corporation (NCK) Research Laboratory with Dr. Yoshikazu Imai and colleagues yielded little new information. NCK is developing an improved silicon carbide fiber (*Super Nicalon*) that is low in oxygen (they stated they can make fiber with less than one wt% oxygen) by means of a radiation cure of the polymer. Although they stated they hope to begin production next year at a rate of one tonne per month, that estimate is probably overly optimistic. NCK is being assisted in the development by Professor Okamura and by Mr. Teguchi of the Japanese Atomic Energy Establishment.

A visit to the Tonen Corporation Research and Development Laboratory with Mr. Takeshi Isoda and his group was interesting and informative. At present, they have a silicon nitride fiber in limited production and are researching improved fibers; however, they see no domestic market for their fiber and are interested in selling it to the U.S. Defense/Aerospace community. While Tonen makes an excellent silicon nitride fiber, they state that its high temperature behavior makes it unsuitable for ceramic composites. The fiber is unstable above 1200°C, oxidizing in air, and crystallizing when heated in nitrogen above that temperature. They are working on a new polyborosilizane-derived Si-B-N fiber that promises much improved high temperature stability. Remarkably, the new fiber is said to remain amorphous after heating for an hour at 1700°C in nitrogen. The fiber is in the early stages of development, and Tonen hopes to have material available for testing in two years.

Ceramic Matrix Composites (CMCs)

While this assessment was not directly concerned with CMCs, the fibers discussed above have applications in CMCs. Also, the discussions about fibers led to some fallout information on composites. It, therefore, seems appropriate to discuss briefly some composites information that came out of this trip.

In general, there seems to be less activity in composites research in Japan than in the U.S., perhaps because of the lack of a defense/aerospace market in Japan. Judging from the two ceramics meetings that were attended, there is much more Japanese activity in whisker-reinforced CMCs than in continuous fiber-reinforced CMCs. Furthermore, research on the latter was mostly on CMCs consisting of oxide, nitride, or carbide ceramic matrices reinforced with carbon/graphite fibers. When asked why, in view of the obvious high temperature oxidation problem with C-containing systems, there was still interest in such systems, one researcher replied that it was probably because they served as model systems to look at toughening increases gained from the use of fiber reinforcements rather than as prototype functional materials.

One composites research activity that stands out, although not directly related to the topics in this report, is the work on nanocomposites by Niihara and coworkers at the Osaka University Institute for Industrial Research. Niihara's group presented several papers at each of the two ceramics meetings attended and, in the author's judgment, this work is the most creative and exciting development in ceramic materials research in Japan, or perhaps anywhere for that matter.

Also noteworthy in the composites field is the excellent research on the properties of, and toughening mechanisms in, CMCs and carbon/carbon composites by Professor Eiichi Yasuda and his group at the Nagatsuta Branch of the Tokyo Institute of Technology. They are very carefully studying the role of the fiber matrix interface in toughening, and at the effect of the fiber surface morphology (i.e., smooth or rough?) in the process. Yasuda's work has involved a wide variety of ceramic materials and should hold great interest for anyone in the field of brittle matrix composites.

Other noteworthy Japanese composites activities include research on particle or platelet reinforced CMCs, and on microstructures with elongated grains or duplex natures. Among topics that stand out in the latter category is work reported in several places on alpha/beta sialon composites. It could well be that such approaches could ultimately represent the most viable route to the formation of ceramics that combine both high strength and high fracture toughness.

GLASS AND CERAMIC TECHNOLOGY

Background Information

While the principal subjects of the technology assessment have been discussed in the preceding sections, it was felt useful to add a section that briefly deals with other significant information learned on the trip that does not fit into any of the other categories. As in the preceding sections, they have been divided, perhaps somewhat artificially, into glass-related and ceramic-related categories.

Glass Technology

The International Conference on New Glasses provided a glimpse into some exciting new research in glass technology. There seems to be a lot of interest in the nonlinear optical properties of glasses with an eye to photonics applications. Also, there were some interesting papers on techniques such as sputtering to form glasses for such applications.

A leading researcher in the preparation of glasses by sputtering, as well as by sol-gel techniques, is Professor Soga of Kyoto University. Other interesting work on sol-gel glasses is being done by Sakka and his group at the Kyoto University Institute for Chemical Research and by Makishima and colleagues at The University of Tokyo. Professor Sakka's group have made a wide variety of fibers (both glass and crystalline) via the sol-gel technique. Fiber compositions have ranged from pure silica to crystalline ceramic superconductors with many other examples in between. Makishima has worked on sol-gel methods for the preparation of silica optical fibers, as well as for making porous glasses for use in removing mercury contamination from water.

Ceramic Materials Technology

Ceramic powder synthesis is an active research area at several of the universities that were visited. Professor Mizutani and his group at the O-Okayama branch of T.I.T. are investigating powder preparation by several techniques including precipitation and spray pyrolysis. They have used the latter technique to prepare very fine (1 μm or less) silicon nitride powder. Professor Yoshimura and his colleagues at the Nagatsuta Branch of T.I.T. have had a long standing program on the preparation of fine powders and crystals by hydrothermal techniques, and they have had their process for the preparation of zirconia powder commercialized by the Chichibu Cement Company. Professor Kijima at the Kyoto Institute of

Technology has produced ultrafine (5 nm), high purity, very sinterable silicon carbide powder by plasma chemical vapor deposition. Kijima also reported plasma sintering compacts of that silicon carbide powder to full density without additives.

Some excellent research on sintering is being conducted by Dr. Mamoru Mitomo of The National Institute for Research in Inorganic Materials. Mitomo has had a long-term interest in silicon nitride and has done some outstanding work on that material including his present detailed study of the development of the microstructure of sintered silicon nitride. This work is providing new insight into the understanding of sintering, as well as toughening mechanisms in that important ceramic material.

Another recent development well worth mentioning is the ultrahigh strength alumina-zirconia reported by Professor Tomozo Nishikawa of K.I.T. The material consists of 70% to 80% zirconia (stabilized with 3% yttria), and 20% to 30% alumina. Professor Nishikawa reports mean bend strengths of 3000 MPa, and states he has seen values as high as 3400 MPa. In all probability, this is the strongest ceramic material ever fabricated.

SUMMARY

Conferences

The three conferences that were attended provided a good overview of current activities in glass and ceramics in Japan. The extensive use of poster sessions greatly assisted communication; the posters were mostly in English, and the poster format allowed for questioning the authors (most of whom knew at least some English) about points that needed clarification. Much of the research reported is applied and the quality is generally very good.

Oxynitride Glasses and Glass Fibers

It was confirmed that the Shimadzu Corporation is still very actively developing their process for manufacturing oxynitride glass fibers and it was found that the fiber is not yet in production (even on a pilot plant basis). The new oxynitride fiber reported by Colloid Research Institute, while highly publicized, is far from being a commercial product. Excellent information was obtained as to the breadth and depth of oxynitride glass research and development in Japan, and as to possible approaches to improving the quality of our oxynitride glasses and fibers.

Ceramic Fibers and Composites

The assessment yielded useful information on the development of silicon carbide and silicon nitride fibers with improved high temperature stability. The markets for such fibers appear limited at present, perhaps slowing their development, and it will be several years or more before fibers are available in quantity. The new BN fiber being investigated by Kimura at the Kyoto Institute of Technology looks very promising and its development should be followed. Aside from limited activity on composites made with continuous carbon fibers, most of the ceramic composites research in Japan seems to focus on whisker-reinforced materials of the type used for tool bits.

Glass and Ceramic Technology

There appears to be substantial activity at Kyoto University and Tokyo University, as well as at other installations not visited, on the preparation of glasses and compounds in various forms via sol-gel techniques. Another active research area at universities and Government laboratories is the synthesis of high purity, sinterable ceramic powders prepared by a variety of methods. Also noteworthy is the study on the preparation of ultrahigh strength zirconia-alumina at K.I.T.

CONCLUSIONS

Attendance at the glass and ceramics conferences provided an excellent overview of current research in Japan, as well as opportunities for making personal contacts with Japanese researchers.

The objectives of the oxynitride glass and fiber part of the technology assessment were fully met, if not exceeded.

Although useful information was obtained on ceramic fibers and composites, this technological area is much broader than oxynitride glasses and fibers and, thus, was covered in less detail.

The trip provided additional interesting information on topics not specifically related to the goals of the technology assessment.

The personal contacts that were initiated led to insights not obtainable in any other way and those contacts should be maintained.

The information gained on the trip will be invaluable in planning future U.S. Army research and development on ceramic fibers and on oxynitride glasses and fibers.

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Table 1. CHRONOLOGICAL ITINERARY: ASSESSMENT OF JAPANESE TECHNOLOGY IN ADVANCED GLASS AND CERAMIC FIBERS

SEPTEMBER 1991

DATE	EVENT/LOCATION
9/3:	Boston-Midland, MI
9/4:	Dow Corning Corporation, Midland, MI (Lipowitz), Midland-Boston
9/19:	Boston-Tokyo
9/20:	Arrived in Tokyo
9/23:	ARO-FE
9/24:	STCFE, ARO-FE
9/25:	ARO-FE
9/26:	T.I.T., Tokyo (Mizutani)
9/27:	Tokyo University (Makishima)
9/30:	T.I.T., Yokohama (Yoshimura)

OCTOBER 1991

DATE	EVENT/LOCATION
10/1:	Tokyo-Kyoto
10/2:	Kyoto University Institute for Chemical Research (Sakka): Seminar
10/3 (a.m.):	Kyoto Institute of Technology (Kijima)
10/3 (p.m.)	Kyoto University (Soga)
10/4:	Shimadzu Corporation (Minakuchi): Seminar
10/5-10/14:	Annual Leave
10/15:	ARO-FE
10/16 (Day):	Tokyo, International Conference on Science and Technology of New Glasses
10/16 (Evening)	Yokohama, 100th Anniversary Meeting of Ceramic Society of Japan
10/17-10/18:	Yokohama, 100th Anniversary Meeting of Ceramic Society of Japan plus Banquet and Ceramic Exhibition
10/21:	Tokyo-Koda
10/21-10/23:	Engineering Ceramics Meeting, Koda (Niihara)
10/23:	Koda-Nagoya

OCTOBER 1991
(continued)

DATE	EVENT/LOCATION
10/24:	Nagoya-Tokyo, ARO-FE
10/25:	ARO-FE
10/28:	Asahi Glass Research Laboratory, Yokohama (Kimura): Seminar
10/29:	Tonen Corporation, Saitami (Takeshi Isoda): Briefing
10/30:	ARO-FE
10/31:	NCK Research & Development, Yokohama (Imai): Seminar

NOVEMBER 1991

DATE	EVENT/LOCATION
11/1:	Tokai Carbon, Shizuoka (Okuda): Seminar
11/4:	ARO-FE
11/5:	Tokyo-Sapporo, Hokkaido GIDL (Unuma), Hokkaido University (Kodaira): Seminar
11/6:	Sapporo-Tokyo, ARO-FE
11/7:	NIRIM, Ibaraki (Mitomo): Seminar
11/8:	ARO-FE
11/12:	ARO-FE
11/13:	ARO-FE
11/14:	ARO-FE
11/15:	Tokyo-Boston
11/15:	Arrived in Boston

Table 2. TOPICAL ITINERARY: ASSESSMENT OF JAPANESE TECHNOLOGY IN ADVANCED GLASS AND CERAMIC FIBERS

INSTALLATION	CONTACT/DISCUSSION TOPIC(S)
Asahi Glass Research Laboratory, Yokohama	Dr. Hiromichi Nishimura, Dr. Hiroshi Abe, Dr. Setsuro Ito. Seminar. Discussion included nothing about Asahi's current research activities.
Dow Corning Corporation Midland, MI	Dr. J. Lipowitz, R. Jones, T. Barnard, E. Pudnos, J. Rabe, G. Vogel. Seminar. HPZ Si_3N_4 Fibers and PCS SiC Fibers.
Hokkaido University, Sapporo	Professors K. Kodaira, T. Yokokawa, J. Takahashi, T. Furusaki, M. Higuchi. Seminar. Research on Melts, Hydrothermal Crystal Growth.
Hokkaido GIDL, Sapporo	Mr. H. Unuma, Dr. R. Yoshida, Mr. J. Nagao. Oxynitride Glasses and Glass-Ceramics, Powder and Thin Film Preparation.
Kyoto University Institute for Chemical Research	Professor Sumio Sakka, Dr. Toshinobu Yoko. Seminar. Oxynitride Glass Research, Sol-Gel Methods for Glass Preparation, Sol-Gel Preparation of Glassy and Crystalline Oxide Fibers, Shimadzu Corporation Oxynitride Glass Fibers.
Kyoto University	Professor Naohiro Soga. Meeting of the Ceramic Society of Japan, Shimadzu Corporation Oxynitride Glass Fibers, Bonding in Glasses, Sol-Gel Glasses, Glasses by Sputtering.
Kyoto Institute of Technology	Professor Kazunori Kijima. Synthesis of Ultrafine SiC Powder to Make Fully Dense Sintered SiC, Plasma Sintering. Professor Tomozo Nishikawa. Ultrahigh Strength Zirconia-Alumina. Dr. Nobuyuki Takeuchi. Glass Crystallization Mechanisms. Professor Masahiko Nakamura. Powder Science and Technology. Professor Yoshiharu Kimura. Synthesis of High Strength BN Fibers from Polymers, BN/BN Composites, Superconducting Oxide Fibers.
NIRIM, Ibaraki	Drs. Mamoru Mitomo, Y. Hasegawa, S. Inoue, A. Nukui, S. V. Lee. Seminar. Oxynitride Glass Research, Oxynitride Fibers, Sintering of Si_3N_4.
Nippon Carbon Corporation (NCK), Yokohama	Dr. Yoshikazu Imai, Michio Takeda, Hirofumi Harada. Seminar. SiC (*Super Nicalon*) Fibers.

Table 2. TOPICAL ITINERARY: ASSESSMENT OF JAPANESE TECHNOLOGY IN ADVANCED GLASS AND CERAMIC FIBERS (continued)

INSTALLATION	CONTACT/DISCUSSION TOPIC(S)
Shimadzu Corporation, Kyoto	Hiroyoshi Minakuchi, Haruo Osafune, Hiroyuki Fujii, Katsuhiko Kada, Kuniaki Kanamaru, Toshifumi Fukui. Seminar. Advanced Development of Oxynitride Glass Fibers and Production Process for Same.
Tokai Carbon Company, Fuji Research Laboratory	Mr. Motohiro Yamamoto, Hiroshi Okuda, Tohru Kida, Itsuro Matsu, Akira Yamakawa, Minoru Fukazawa. Seminar. Si_3N_4 Matrix-SiC Particle Composites, C-Fiber Reinforced SiC and B_4C Composites, Carbon Black.
Tokyo University	Professor Akio Makishima. Oxynitride Glass Research, Sol-Gel Glasses, Rare Earth Aluminate Glasses, Expert Systems for Designing Glasses. Professor Taketo Sakuma. Superplastic Zirconia. Professor Fumitaka Tsukihashi. Thermodynamic Studies on Oxynitride Glass Systems.
Tokyo Institute of Tech., Ooyama (Tokyo)	Professor Nobuyasu Mizutani. Ceramic Powder Synthesis and Characterization, Thin Film Preparation, Zro_2 Single Crystal Growth.
Tokyo Institute of Tech., Nagatsuta (Yokohama)	Professor Masahiro Yoshimura. Whisker-Glass Composites, Hydrothermal Zirconia Powders, Hydrothermal Machining, Super-Conducting Thin Films. Professor Eiichi Yasuda. Toughening Mechanisms in Ceramic Matrix/Whisker-Reinforced Composites, CMCs with SiC Whiskers Formed **In Situ**, Magnesia Bicrystals, Carbon-Carbon Composites. Professor Ken-ichi Kondo, Dr. Hisako Hirai. Diamond Synthesis by Shock Compaction. Profession Zenbe-E Nakagawa, Mr. Naoya Enomoto. Oxide Glass and Ceramic Materials, Ultrasonically Assisted Powder Preparation.
Tonen Corporation, Saitama (near Tokyo)	Dr. Takeshi Isoda, Osamu Funayama, Y. Shimizu, H. Aoki, and K. Sato. Briefing. PSZ Preceramic Polymer, Si_3N_4 Fibers, Si-N Matrix Composites from Polyborosilizanes for Improved Thermal Stability.
U.S. Army STCFE	Mr. David W. Baker, Mr. Arihito Tabata, Major Rick MacDougall. STCFE Activities, Possible Travel Assistance.

Table 3. ALPHABETIZED COMPILATION OF BUSINESS CARDS OF INDIVIDUALS CONTACTED WHILE DOING ASSESSMENT OF JAPANESE TECHNOLOGY IN ADVANCED GLASS AND CERAMIC FIBERS

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