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FOREIGN TRIP REPORT

ORNL/FTR-3097

DATE: October 20, 1988

SUBJECT: Report of Foreign Travel of V. J. Tennery, High Temperature Materials Section, Metals and Ceramics Division

TO: Alexander Zucker

FROM: V. J. Tennery

PURPOSE: To attend working group meeting for Subtask 2, Ceramic Powder Characterization, IEA Annex II, and 2nd International Conference on Ceramic Powder Processing Science. Both meetings were held in the Kur- and Kongresszentrum in Berchtesgaden, Federal Republic of Germany.

SITES VISITED: 10/7-14/88 Conference Berchtesgaden,
The Federal Republic of Germany

ABSTRACT: The traveler attended the conference, "2nd International Conference on Ceramic Powder Processing Science," and participated in the Subtask 2 working group meeting on ceramic powder characterization being conducted by the United States, the Federal Republic of Germany (FRG), and Sweden under terms of the Annex II agreement. In addition, the traveler participated in an unscheduled meeting with Dr. R. Neumann, KFA, Jülich, FRG, and Dr. A. Brückner-Foit, KFK, University of Karlsruhe, FRG, to review major problems encountered in receiving mechanical property data from the FRG under Subtask 4 of the Annex.

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COMPREHENSIVE TRIP REPORT

Subtask 2 Working Group Meeting
IEA Annex II on Structural Ceramics

Kur- and Kongresszentrum
Berchtesgaden, Federal Republic of Germany

A preliminary meeting was held in the morning of October 10, attended only by the senior representatives of the three countries to review the agenda and ground rules to be followed for the primary meeting, which started at 2 p.m. The major purpose of this meeting was to determine that the primary meeting to start in the afternoon would occur in a planned and orderly manner. The preliminary meeting started at 9 a.m. and lasted approximately two hours. The two people who primarily led the meeting were R. Neumann, KFA (FRG), and S. Hsu, National Institute of Standards and Technology (NIST) (formerly NBS) (USA). Hsu has the lead responsibility in Subtask 2 of the Annex for coordinating data submission, compilation, and analysis. Neumann has the responsibility of funding and coordinating all research activities under the Annex in the FRG. At this meeting, A. Dragoo and Hsu of NIST distributed a draft copy of the data compilation from Subtask 2. This document includes all data received to date resulting from work in the three countries on ceramic powder characterization, and includes data in both tabular form and in graphical form. This compilation includes all data for the four powders studied, and is the largest assemblage of ceramic powder property results from a large number of laboratories ever completed.

During this preliminary meeting, it was agreed that the following schedule will be met for completing the work in Subtask 2 on ceramic powder characterization.

12/15/88	All corrections in current data set due to Hsu, NIST.
4/30/89	Part 1 of Final Report (Data Only) to printer (responsibility of Hsu).
6/1/89	Hsu submits final revised Part 2 report to IEA Executive Committee for comment.
8/31/89	Draft of final report distributed by NIST to participants.
12/15/89	Part 2 of Final Report (Analysis and Recommendations) is published and distributed to participants.
12/15/89	Statistical analysis of data started. (Optional as to completion; responsibility undetermined, but possibly NIST.)

Publication of Part 2 of the final report will officially conclude Subtask 2, and since this subtask will be the last finished under the Annex, its completion will also officially conclude the work under Annex II. Neumann, senior person present representing the FRG, was quite emphatic that all research associated with Annex II, including publication and distribution of the final reports, should be finished by the end of CY 1989.

The three national coordinators for Subtask 2 agreed to meet at the time of the American Ceramic Society meeting in Cocoa Beach, Florida, during the period January 15-18, 1989. The major objective of this meeting is to review conclusions and recommendations for each of the three subject areas in the subtask, namely, (1) chemical characterization, (2) particle size distribution, and (3) other physical properties. These three areas are the responsibility of A. Dragoo, NIST (USA); H. Hausner, Technical University of Berlin (FRG); and R. Pompe, Swedish Silicate Institute (Sweden). At the 91st Annual Meeting of the American Ceramic Society, April 23-27, 1989, these three national coordinators will meet again to review their national positions on the state of ceramic powder characterization and the conclusions from this meeting will form the basis of the report due to the Executive Committee from Hsu by June 1, 1989.

In the closing of this preliminary meeting, Neumann made a very strong statement that the final recommendations resulting from this work should include a set of specific technical recommendations independent of any considerations of a future Annex III activity. He felt that what was needed was the broadest scientific recommendation for consideration by the Executive Committee, and that the Committee does not want to receive direct suggestions that certain additional work be done within a future Annex III. This material will be included in the Part 2 report noted earlier. The representatives of the United States and Sweden agreed to this position.

The formal Subtask 2 meeting started at 2 p.m. on Monday and continued through Tuesday, October 11. The agenda is Appendix C of this trip report. Complete tape recordings of this meeting were made by L. Boesch of Engineering & Economics Research, Inc. (EER), and will form the basis of a much more complete rendition of the proceedings than will be given here by the traveler. The meeting was started with a brief introduction and welcome by Neumann on behalf of the host country, followed by a response by Schulz of the USDOE. This meeting was attended by about 20 people most of the time, with about 8 from the United States, 9 from the FRG, and 3 from Sweden. Attendance lists were prepared and will be included in the detailed meeting report being prepared by Boesch for the DOE. The U.S. and Swedish representatives included two industrial representatives most of the time, while West German industrial institutions represented varied from two to four at various times during this meeting.

Hsu discussed the fact that the first goal of the meeting was to review for the attendees those powder property measurements from laboratories in the three countries, which at least "appear" to give reasonable agreement. The data compilation prepared by NIST was distributed. This compilation includes tabular and graphical versions of all data received by NIST from the 25 participating laboratories. Most laboratories provided only selected data for each powder. In some cases, only one or two powders were studied by a given laboratory. Dragoo then reviewed results for the chemical analysis data for three of the four powders being studied in this subtask. The three discussed were the so-called reference nitride powder (Starck LC-10 from the FRG); the "test" powder, a silicon nitride from UBE in Japan; and the silicon powder from KemaNord in Sweden. Results for the Toyasoda zirconia powder from Japan were not discussed, since these were covered in detail last November at the Orlando working group meeting. The chemical analysis results for both major and minor elements were not particularly encouraging. Analysis results for the major elements typically varied by as much as 2 to 3%, while the trace element analysis results in several cases varied by factors of 10 or more. Pompe of Sweden then presented an overview of results from surface area, morphology, and other similar physical property measurements on these powders. The tap density was one property found to be reasonably reproducible among the 25 laboratories participating in this subtask. Several laboratories determined powder surface area, and it was a surprise to the attendees that values obtained using single-point BET were typically different from values obtained for the same powder using multi-point BET techniques.

Hausner of the Technical University of Berlin then reviewed results for particle size and particle density measurements. The data reported had been reduced to tabular form typically including the d(10), d(50), and d(90) sizes, i.e., the particle size at which 10, 50, and 90 % of the population is finer than the stated value. It was apparent from many of these data that some laboratories had used quite different powder dispersion methods for preparing the specimens for measurement. In addition, several different instrument types were used, including X-ray sedimentation and laser light scattering. Hausner then provided data from his laboratory which showed the results obtained for Starck silicon carbide powder with the same X-ray sedimentation instrument when using All liquid from the Micromeritics Corporation (USA) and water with 0.5% butlyamine. The water plus 0.5% butlyamine combination resulted in particle sizes at the 10, 50, and 90% population levels which were from six to over ten times smaller than obtained using the All fluid, which is a conventionally used dispersant liquid for these powders. These results were used to illustrate the critical importance of powder dispersion for accurate particle size measurements. Other results provided later by Pompe also illustrated the importance of using direct observational methods, such as scanning and/or transmission electron microscopy wherever possible to confirm results obtained from indirect measurements, such as X-ray sedimentation or laser scattering methods. It was pointed out that, generally, water cannot be used as the dispersion medium for silicon nitride powders due to detrimental chemical reactions. However, it was also confirmed that many laboratories routinely use water as the dispersion medium for silicon nitride powder measurements. It was considered to be a very unfortunate circumstance that only 2 laboratories of the 25 participants used scanning electron or transmission electron microscopy to directly determine the size of the powder particles for the four powders that were studied. New image analysis software and instrumentation should be able to provide direct measurement of particle size distributions from micrographs obtained with these instruments for any ceramic powder once an unbiased dispersion technique is available. Density measurements for powders were discussed at some length, since it was clear from the data compilation that some laboratories using techniques requiring particle density as a given input value were using densities for the material which were even above the X-ray density. The relatively new laser scattering instruments were discussed at length by a representative from Lonza GmbH, including the fact that for this type of measurement the optical absorption coefficient for the particle, including all phases within the particles, is required as an input parameter. For the case of both silicon carbide polytypes and the phases for silicon nitride, accurate values for these coefficients are lacking. Researchers using these instruments typically use "nominal" absorption coefficients, the value of which has a direct effect on the particle size distribution values.

The meetings of the Tuesday morning and afternoon sessions included chemical analysis, surface chemistry measurements, and particle size distribution measurements of silicon nitride powders. The afternoon session was devoted to summarization of technical conclusions and recommendations and discussion of recommendations for the final report. The chemical analysis discussion was particularly interesting, since it again illustrated the great difficulty encountered in analyzing ceramic materials (either powders or sintered material) for either major or minor elements. W. Genthe of the Technical University of Berlin presented results obtained in determining the blank values for nine elements typically determined for silicon carbide and silicon nitride due to use of different materials for containing the sample and the acid or other chemicals used to dissolve the compound prior to analysis. Results for dissolution of samples of silicon nitride and silicon carbide were compared with acid dissolution and lithium borate dissolution at 1100°C. The results showed that lower blank values were usually obtained using acid dissolution. Additionally, results for inductively coupled plasma analysis techniques, which are often used for determination of Si, Fe, Cr, Cu, Al, Ca, Mg, and Na, were discussed including problems in preparing a "representative solution" of the sample. R. Pugh of the Institute for Surface Chemistry, Stockholm, presented recent results on electrophoretic measurements of silica, silicon oxy-nitride, and silicon nitride, and showed that the isoelectric point for these phases occurs over a wide range of pH, depending upon the preparation history of the suspension. This result makes preparation of a stable suspension of these phases for use in fabrication of silicon nitride ceramics very difficult unless the pH is controlled over a relatively narrow range. Measurements on Starck silicon nitride powder (reference in Subtask 2), showed the isoelectric point varying from a pH of 3 to 9, depending upon how the suspension was prepared and aged prior to the measurement. Leaching of a number of silicon nitride powders in water for up to 30 days resulted in the isoelectric point changing to a common value near pH = 7. The meaning of this result was unclear, i.e., was the result due to hydrolysis of the nitride, or was the "true surface" being exposed due to long time suspension in water? ESCA and Auger spectroscopy results on ceramic powders were discussed as a means for characterizing the surface contaminants of powder particles, but no definitive conclusions were provided for reliably preparing the specimens without contaminating the surfaces. There was also considerable discussion of use of DRIFT (diffused reflectance infra-red Fourier transform spectroscopy) techniques for characterizing the chemical state of ceramic powder surfaces. Results obtained for silicon carbide powder heat treated at temperatures up to 1100°C for 3 h were used to illustrate the application of this technique. Results for silicon carbide were used to determine the reaction kinetic parameters for powder oxidation.

Measurement of particle size distributions was reviewed in considerable detail by Pompe of Sweden. Agglomerates, including their detection and removal, were the major impediments to "good" measurements. The question was raised and discussed regarding the usefulness of removing agglomerates from a powder specimen prior to particle size measurement since it is widely accepted that agglomerates in powders are major sources of critical fracture flaws in sintered ceramics. No clear conclusion was made by the group on this point.

The "Technical Issues" session included, for the chemical characterization results, consideration of how to identify and eliminate "outlier" results from the data set prior to statistical analysis at NIST. There was no general agreement as to how such outliers could be identified. In addition, the general consensus of the group was that reliable methods for trace element analysis was of considerable importance for silicon nitride powders, since, for instance, it is generally thought that Ca and Fe at typical concentrations act as sintering aids but reduce high-temperature strength. Lack of specific details used in each laboratory for each type of elemental analysis prevents identification of causes for different results obtained by different laboratories. Neumann said that this specific analysis information should be required from all participants as soon as possible.

Pompe led a discussion of the fact that multi-point BET results were generally higher than values obtained for single-point BET results for the same powder. Pugh stated that only krypton should be used for a powder having an area greater than about 2 m^2/g, while other attendees were not certain. The attendee from Daimler-Benz (FRG) stated that they typically got about the same results regardless of which method was used. P. Matje of ESK (FRG) stated that they now use only multi-point measurements for silicon carbide powders since they obtain more reproducible results, and these are typically 5% lower than single point values. Following all of this discussion, the group decided that BET type measurements were in a pretty good condition, and would probably not merit attention in any future work. Similarly, tap density measurements were determined to give acceptable results.

Hausner led a discussion on particle size measurement needs and results. It was concluded that better dispersion methods for each type of ceramic powder are required, including results obtained for these various dispersions in the main types of particle-measuring instruments. There is a critical need for techniques for specimen preparation and analysis using both SEM and TEM, including automated image analysis to obtain particle size distribution parameters. One of the critical issues here is to obtain a "representative" specimen for analysis in these high-magnification instruments. Neumann suggested that the goal should be to identify the simplest method and determine if in future work, reproducible results are possible. The attendees all agreed with this suggestion. This particular point will be reviewed specifically at the Cocoa Beach meeting in January, 1989.

Hsu then started a discussion relative to recommendations and possible future IEA research in this area. Major points considered included, sampling, atmosphere/environment effects on powder surfaces, contamination from grinding media, and sample dispersion and general preparation procedures. Hsu asked that all participants carefully check their data values and inform him of any necessary corrections by December 15, 1988. A description of the methods of specimen preparation and property measurement noted earlier is also due to NIST by December 15, 1988. S. Natasohn (USA) commented that he understood from the Orlando meeting of 1987 that a statistical analysis of the data was to be included in the final report for Subtask 2. Hsu replied that such an analysis was not going to be done as part of the present IEA activity, and that such analyses could or would be done in a later activity, now considered as optional. This meeting closed with some discussion of possible future activities, possibly as part of an Annex III activity on ceramic powders. This discussion, which did not result in any final decisions being made, included alternate ideas of either all participants doing the same experimental activities on perhaps one selected powder or dividing up into relatively small groups and carrying out something like a 4 × 8 matrix of duplicate runs on a number of powders which would allow such techniques as ANOVA to be used for analyzing inter- vs intra-group variations in the results.

Special Meeting Regarding Subtask 4
Mechanical Properties of Structural Ceramics

This meeting was held after completion of the Monday afternoon session related to Subtask 2 at the request of Neumann, KFA (FRG). He had requested A. Brückner-Foit of the KFK, University of Karlsruhe, to travel to Berchtesgaden specifically to meet with me and determine if we could resolve some of the data-sharing problems which have plagued Subtask 4. These problems pertain particularly to the unavailability of proper data from the German laboratories, and thus is specifically the responsibility of the group to which Brückner-Foit belongs at the KFK. Many letters and discussions have been held on this matter in the past year. Brückner-Foit and Professor D. Munz, her supervisor at Karlsruhe, were given responsibility for the FRG coordination of Subtask 4 early in Annex II by Neumann. For nearly one year, we and

Sweden have been totally unable to obtain from Karlsruhe complete mechanical property data sets, and until recently, we thought this was due to lack of knowledge by the FRG participants of the software to be used (Lotus 1-2-3). Presently, the United States and Sweden are completely lacking the mechanical strength data for the silicon nitride specimens provided by the United States and Sweden accompanied by the specimen designator numbers which allow tracing of the history of the specimens regarding original billet production time and the location of the specimen in the billet.

At the Amsterdam meeting in June, Brückner-Foit specifically asked me for these designators, even though they had been sent to the FRG almost a year earlier. These were sent within three weeks of my return from the Amsterdam meeting. In three letters since that time, I have requested the required data, and all to no avail. This was made clear during the meeting on October 10. Brückner-Foit then made it clear that she felt her computer program, which she wrote, would do the required calculations and that she would not purchase the required Lotus software to prepare the necessary files for transmittal to us and Sweden. She said that she could perhaps prepare the required data in ASCII format. I agreed reluctantly to this and stated that we needed these data immediately. She then said that she had an engineering student who was "analyzing these data" as part of a thesis. She thus revealed that they at Karlsruhe are analyzing the very data we have been seeking from them. The work is being done as part of this student's thesis in mechanical engineering. I was told that he could prepare the necessary files, but that in two days he was returning to his co-op job with a West German company. If he didn't have time to do it now, it would be at least three months before such files could be prepared for us. I again stated that we needed the data immediately for our own analysis. Neumann said that he understood and wished that he could help the process.

I also reviewed corrections I felt were necessary in Brückner-Foit's report on the statistical analysis portion of Subtask 4. My letter of September 11 had not yet been received in the FRG. The meeting ended, and Neumann told me privately that he hopes that the matter is now resolved. I concluded that this entire situation is now out of his control, and that we may never receive the required data, in spite of the costs and effort we expended in obtaining specimens with processing time and billet location traceability.

2nd International Conference on Ceramic Powder Processing Science

Kur- and Kongresszentrum
Berchtesgaden, Federal Republic of Germany

This was the second of a new series of meetings on ceramic powder science, with the first being held in Orlando, Florida, in November 1987. The meeting was held under the auspices of the Deutsche Keramische Gesellschafft e.V., and had an attendance of approximately 300 people with well over half being from outside the FRG. The meeting was organized with 30 presented papers and 83 posters. A total of 2 of the papers and 12 of the posters were no-shows. In the traveler's opinion, there was not particularly much new information presented at this meeting. One conclusion accepted as a general theorem was that agglomerates must be avoided in ceramic powders, but there is no general agreement as to how one easily and in a straight forward manner detects their presence, prevents their formation, or removes them from a synthesized powder. This is the central issue in ceramic powder science at the present, and no really new ideas seem to be at hand for dealing with the problem. The favorite approach for systems amenable to aqueous chemistry relies on colloidal techniques to form the particles by controlled nucleation and crystallization and never allowing the suspension to dry prior to green body formation. Many feel this is an impractical approach. Others feel that more conventional powder synthesis techniques can be used, followed by removal of agglomerates by some means such as centrifugal or gravitational separation. Demonstration of this approach on a reasonable production scale was not discussed at this meeting. One of the more interesting papers on the influence of particle size, the distribution and surface area on sintering and microstructural development by authors from Ceramic Process Systems in the USA was withdrawn.

Some of the more interesting results included powder/structure/property relationships for AlN ceramics. A paper by M. Yonezawa of the NEC corporation in Japan described research on plasma synthesis of AlN powder from Al powder and ammonia in a reactor capable of synthesizing greater than 100 g of powder per hour with a mean particle size of about 0.1 μm based upon transmission microscopy results. The surface area varies from 20 to 90 m^2/g with an Al:N ratio of 0.91 to 0.93. The major contaminant is oxygen in the 2–5 wt % range, while other impurities total 100 ppm or less. The powders must be stored in high purity nitrogen, and FTIR and NMR techniques are used for characterizing the state of oxygen and hydrogen impurities in the powders. Ceramics fabricated from these powders still require temperatures of nearly 1400°C to achieve high densities. Poor physical properties are obtained unless special sintering additives, such as yttrium oxide or yttrium fluoride are used. When this is accomplished, thermal conductivities as high as about 240 W/mK are achieved for these ceramics. However, such high values can be achieved only by processing at high temperatures, for example 1900°C for 100 h. Achieving high thermal conductivity required oxygen impurities being trapped at the grain boundaries in a yttrium garnet oxide phase. Without this trapping process, the oxygen diffuses into the AlN structure and decreases the conductivity due to impurity scattering of the phonons.

An interesting paper by M. Sacks of the University of Florida reviewed techniques for achieving theoretical density in alumina ceramics at temperatures as low as 1150°C. Sacks used commercially available small-particle-size powders and developed techniques for removing existing agglomerates and manipulating the particle size distribution using liquid sedimentation. The same success was then achieved for silica particles. The "suspension processed alumina" achieved a higher density at a lower temperature than possible using Messing's "seeded boehmite" approach, which was described in another paper at this meeting. Finally, F. Lange of the University of California, Santa Barbara, presented a paper in which he basically refuted many of the recent "theoretical" papers focussed on predicting the behavior of powders and instead based his work on an early concept of Furnas (1928) to explain simple particle packing in multi-sized systems. The same ideas were then applied to whisker packing to show the physical limitations on forming dense arrays of ceramic whiskers suitable for fabrication of ceramic whisker reinforced composites. An interesting result from this paper was the use of a deflocculant active both on alumina powder particles used to form the matrix of the composite and SAFFIL alumina fibers. Such a system was used to demonstrate the ability to use pressure infiltration to form a high green density composite body, "if" the deflocculant was active on both phases. The more general case, where the powder and fibers have different dispersion chemistry, is much more difficult, and little has been reported on approaches to solve this very pressing practical problem.

APPENDIX A

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1988

10/7–8	Travel from Oak Ridge, TN, to Munich, FRG, via plane
10/9	Travel by auto from Munich to Conference site at the Kur- and Kongresszentrum Berchtesgaden, FRG
10/10–13	"2nd International Conference on Ceramic Powder Processing Science," and IEA Annex II, Subtask 2 Working Group Meeting
10/13	Travel from Berchtesgaden to Munich, FRG, by auto
10/14	Travel from Munich, FRG, to Oak Ridge, TN, via plane

APPENDIX B

LITERATURE ACQUIRED

1. IEA Annex II, Subtask 2, Powder Characterization IEA Workshop, Berchtesgaden, FRG, October 11 & 12, 1988, provided by NIST personnel as part of their Annex II, Subtask 2 activity under the Ceramic Technology for Advanced Heat Engines Project supported by ORNL under an interagency agreement.

2. X-Ray Diffraction of Ceramic Powders, by Gary M. Crosbie and Ernest D. Stiles, Research Staff, Ford Motor Company.

3. Bestimmung von Teilchengrösse und Gitterverzerrungen an Vershieden Keramikpulvern, by R. Prümmer and E. Reisacher, Frauenhofer-Institut for Werkstoffmechanik, Freiburg, FRG

4. Abstracts of the 2nd International Conference on Ceramic Powder Processing Science, October 12-14, 1988, Berchtesgaden, Federal Republic of Germany.

APPENDIX C

Final Draft

IEA/ANNEX II, Subtask 2

POWDER CHARACTERIZATION

- AGENDA -

IEA Working Group Meeting

October 10 and 11, 1988

Kur- und Kongresshaus, Kongress Room I

Berchtesgaden, Bavaria, FRG

Dr. S. M. Hsu, Chairman

Monday, Afternoon Session

2:00 p.m.	Welcome Response	Dr. R. Neumann, KFA Julich, FRG Mr. R. Schultz, DoE/HQ, USA
2:15	Goals of Meeting	Dr. S. M. Hsu, NIST, USA
2:30	Overview of Data Discussion Leader: Dr. S. M. Hsu	
	(1) Chemical Composition, Dr. A. Dragoo, NIST (35 min. plus 10 min. discussion)	
3:15	Break	
3:30	(2) Physical Properties I - Surface Area, FSSS, Morphology etc. Dr. R. Pompe, SSFI, Sweden	
	(3) Physical Properties II - Particle Size and Density Prof. Dr. H. Hausner, TU Berlin, FRG	

Tuesday, Morning Session

Problems in Characterization of Powders
Technical Presentations (presentation 15 min., followed by 5 min. discussion)

8:30 Issues in Chemical Analysis of Powders
Discussion Leader: Dr. A. Dragoo
(1) W. Genthe, TU Berlin: Influence of sample preparation on results of chemical analysis
(2) P. Matje and K. A. Schwetz, ESK: Determination methods for carbon and free carbon in SiC

9:10 Issues in Physical Property Characterization (Surface Area etc.)
Discussion Leader: Dr. R. Pompe
(1) Dr. R. Pugh, Institute for Surface Chemistry, Sweden: Surface chemical characterization of Si_3N_4 and SiC powders

9:30 Issues in Particle Size and Density Measurements:
Discussion Leader: Prof. Dr. H. Hausner
(1) Dr. R. Pompe, On the characterization of particle size of Si_3N_4 powders

9:50 Break

10:10 Discussion of Technical Issues
Chemical Properties Dr. A. Dragoo
Physical Properties I Dr. R. Pompe
Physical Properties II Prof. Dr. Hausner

12:00 Luncheon

Tuesday, Afternoon Session

1:30 Technical Conclusions and Recommendations
Discussion Leader: Dr. S. M. Hsu

2:45 Break

3:00 General Meeting Dr. S. M. Hsu

1) Recommendations for final report

2) Future IEA programs

3) Other Business

4:45 Closing Remarks Dr. R. Neumann