OCCASION

This publication has been made available to the public on the occasion of the 50th anniversary of the United Nations Industrial Development Organisation.

DISCLAIMER

FAIR USE POLICY

CONTACT

Please contact publications@unido.org for further information concerning UNIDO publications.

For more information about UNIDO, please visit us at www.unido.org

UNITED NATIONS INDUSTRIAL DEVELOPMENT ORGANIZATION
Vienna International Centre, P.O. Box 300, 1400 Vienna, Austria
Tel: (+43-1) 26026-0 · www.unido.org · unido@unido.org

22296

<UNIDO Project TF/GLO/95/005>

Workshop on Testing and Evaluation of Mechanical Properties of Ceramics

March 16 ~ 21, 1998

Submitted by Dr. Seong-Jai Cho
Principal Researcher
Materials Performance Group
Materials Evaluation Center
Korea Research Institute of Standards and Science (KRISS)
P.O. Box 102, Yusong, Taejon 305-600, Republic of Korea
Tel. (+82) 42 868 5388, Fax (+82) 42 868 5032, E-mail : sjcho@kriss.re.kr

Korea Research Institute of Standards and Science (KRISS)

1. Introduction

Advanced materials technology is one of the key technologies for a wide range of industrial sectors having a major influence on the economic and industrial competitiveness. In addition, worldwide demand for advanced materials production has been increasing rapidly for the last decades. For example, advanced ceramics in world market is expected to increase 8.5% a year between 1990 and 2000 to reach US$25 billion market in 2000, up from US$11 billion in 1990.

Reliable methods of testing and evaluation of advanced ceramics are crucial for their successful development and efficient incorporation into competitive industrial products. However, the development of widely recognized evaluation methods are very slow and dispersed in developing countries. As part of efforts to draw a concerted activity from developing countries in recognized testing and evaluation methods of advanced ceramics, Korea Research Institute of Standards and Science (KRISS) proposed a project of "establishment of testing techniques for flexural strength and fracture toughness of fine ceramics" under the umbrella of the UNIDO program to establish an International Center for Materials Evaluation Technology (ICMET) which was initiated by UNIDO and KRISS. This project aims at establishing testing methods for the flexural strength and fracture toughness of fine ceramics among the participating countries in the ICMET program, including P.R. China, India, Malaysia, Singapore, and Thailand. In the agenda of the project, a workshop and Round Robin Tests (RRTs) among the participating countries was included and in accordance with the agenda, the workshop on testing and evaluation of mechanical properties of ceramics was organized at Materials Evaluation Center, KRISS from March 16 ~ 21, 1998.

2. Participants

Dr. Sutiporn Chewasatn
Director, Production Process Development Laboratory
Thailand Institute of Scientific and Technological Research (TISTR)
196 Phahonyothin Rd, Chatuchak, Bangkok 10900, Thailand
Tel. (+66) 2 579-1121/30 ext. 2014, Fax (+66) 2 579-6538

Ms. Wasana Khonawona
Production Process Development Laboratory
Thailand Institute of Scientific and Technological Research (TISTR)
196 Phahonyothin Rd, Chatuchak, Bangkok 10900, Thailand
Tel. (+66) 2 579-1121/30, Fax (+66) 2 579-6538

Mr. Ling Xiao
Senior Engineer
Shanghai Research Institute of Materials (SRIM)
99 Handan Road, Shanghai, P.R. China
Tel. (+86) 21 65420775, Fax (+86) 21 65420554

3. Trainers

Dr. Seong-Jai Cho, Principal Researcher, Materials Evaluation Center, KRISS
Dr. Kyung-Jin Yoon, Senior Researcher, Materials Evaluation Center, KRISS
Mr. Dong-Jin Kim, Senior Technician, Materials Evaluation Center, KRISS
Mr. Sang-Jin Park, Senior Technician, Machine Shop, KRISS

4. Training Schedule *(* Some photographs taken during the workshop are attached Annex A.)*

March 16 (Mon.)

- Introduction to KRISS
- Introduction to ICMET Project
- Lab Tour (Materials Evaluation Center, KRISS)
  - Materials Performance Lab.
  - Microstructure Science Lab.
  - Crystal Evaluation Lab.
  - Nano Characterization Lab.
  - Surface Analysis Lab.
  - Epitaxial Semiconductor Lab.
- Welcome Party (hosted by Dr. Seong-Jai Cho)

March 17 (Tue.)

- Specimen Machining *(6 hours)*
  - Lecture by Dr. Seong-Jai Cho *(3 hours)*
  - Practical Session at KRISS Machine Shop by Mr. Sang-Jin Park *(3 hours)*

March 18 (Wed.)

- Historic Site Tour (Kyongju)

March 18 (Thu.)

- Flexural Strength *(3 hours)*
  - Lecture by Dr. Seong-Jai Cho *(1.5 hours)*
  - Practical Session by Mr. Dong-Jin Kim *(1.5 hours)*
- Weibull Analysis of Strength Data *(3 hours)*
  - Lecture by Dr. Kyung-Jin Yoon *(1.5 hours)*
  - Practical Session by Dr. Kyung-Jin Yoon *(1.5 hours)*

March 20 (Fri.)

- Fracture Toughness *(6 hours)*
  - Lecture by Dr. Seong-Jai Cho *(3 hours)*
  - Practical Session by Mr. Dong-Jin Kim *(3 hours)*
- Farewell Party (hosted by Dr. Yang-Koo Cho, Director, Materials Evaluation Center, KRISS)

March 21 (Sat.)

- Fracture Toughness *(3 hours)*
  - Practical Session by Dr. Seong-Jai Cho *(3 hours)*
- Distribution of Fixtures and Alumina Specimens for RRTs (2 flexure fixtures, a bridge loading fixture and 50 bend bar specimens)

5. Summary of Lectures

Specimen Machining

A lecture was given on the ceramic grinding mechanisms and their implications in specimen preparation. Special emphasis was put on how to avoid cracking formation during the grinding. Grinding conditions to prevent the cracking were also discussed in the lecture.

Flexural Strength Testing Technique

Comparative analysis between tensile strength and flexural strength was made in the lecture, and factors that produce errors in strength testing was discussed. In particular, friction error caused by the fixtures with non-rotating rollers was emphasized. Effects of specimen size, spans, loading rate were also explained in the lecture.

Weibull Analysis of Strength Data

Concepts of the weakest link theory and Weibull statistics were discussed. In addition, maximum likelihood estimation technique was explained for the calculation of Weibull parameters from strength data, and effect of specimen volume on the strength data was interpreted.

Fracture Toughness Testing Technique

A brief introduction was made on various methods of fracture toughness testing technique such as Indentation Fracture (IF) method, Indentation Strength (IS) method, Chevron Notched Beam (CNB) method, Single Edge V-notched Beam (SEVNB) method, and Surface Crack in Flexure (SCF) method. Details of Single Edge Precracked Beam (SEPB) method was also explained.

6. Summary of Practical Sessions

Specimen Machining Process

Demonstration was given to the participants about control of diamond tools such as truing and dressing, machining of flexural specimens, rough grinding, final grinding and chamfering processes.

Flexural Strength Testing *(* Strength data are attached in Annex B)*

Flexural strength of alumina (Coors AD 995) was measured in the testing. Two 4-point-bend fixtures with different spans of 40/20mm and 30/10mm were used in the flexural strength testing.

Weibull Analysis of the Strength Data *(* Results are illustrated in Annex C)*

The strength data was analyzed by maximum likelihood estimation. Weibull modulus of the alumina material shows high according to the data.

Fracture Toughness Testing

Fracture toughness of alumina samples were measured by using Single Edge Precracked Beam (SEPB) method. Five validate data obtained from 20 specimens.

7. Evaluation of the Workshop

For evaluation of the workshop, a questionnaire was distributed to each participant. (* *The results are attached in Annex D).*

8. Conclusion

Lectures were first given in the workshop to the participants on the testing techniques of flexural strength and fracture toughness of ceramics in order to provide them with theoretical backgrounds prior to the practical sessions. In the practical sessions, they practiced themselves testing on the flexural strength and fracture toughness of reference alumina material with the theoretical backgrounds. The workshop was successfully completed. But less people participated in the workshop than we expected mainly because of the economic difficulty most participating countries in the ICMET project are now facing.

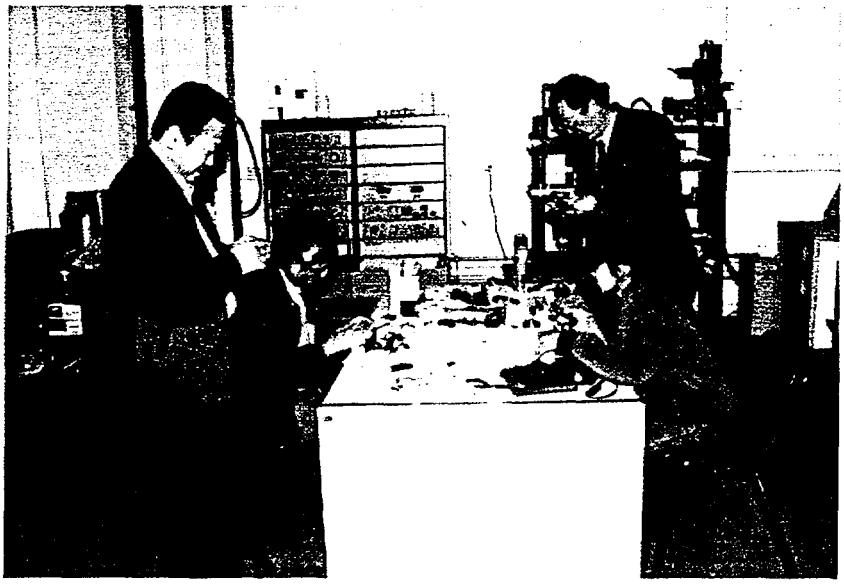
Two flexural fixtures with different span, a bridge loading fixture for SEPB fracture toughness testing, and fifty alumina specimens were distributed to each participant for the purpose of the Round Robin Tests (RRTs). The fixtures and specimens will be distributed to the other ICMET participating countries for the RRTs as well. Silicon carbide specimens are now being machined for distribution to all the ICMET participating countries.

Annex A. Some photographs taken during the Workshop/Training

Participants and trainers, Dr. Stiporn, Ms. Wassala and Mr. Ling from left of the front, Dr. Cho and Dr. Yoon, from left of the rear.

At the Hankook Condominium of Kyungju city.

Participants are taking a lecture given by Dr. Cho.



Participants are taking a practical session on fracture toughness testing with the assistance of Mr. Kim.

Annex B. Flexural strength data.

Top support N Boja

***** Flexure testing data sheet *****

Date : 19/3/98 4 point(✓) or 3 point ( ) 40/20

Temperature : ℃ Material : Al_2O_3

No	width, w (mm)		thickness, t (mm)		Fracture load (kN or kgf)	Strength(MPa)	Remarks
1.	4.014 4.013 4.009	4.012	3.014 3.012 3.010	3.012	0.4331	357	just break ~~0.4331 kN.~~
2	4.004 4.013 4.010	4.010	3.004 3.001 2.999	3.001	0.3996	332	
3	4.007 4.006 4.013	4.009	3.013 3.009 3.009	3.010	0.4158	343	
4	4.013 4.014 4.009	4.012	3.004 3.089 3.008	3.034	0.3661	297	
5	4.008 4.006 4.007	4.007	3.006 3.003 2.999	3.003	0.4142	304	
6	4.010 4.011 4.010	4.010	3.000 3.006 3.005	3.004	0.4188	347	
7	4.021 4.021 4.024	4.022	3.005 3.006 3.011	3.007	0.4263	352	
8	4.009 4.009 4.007	4.008	2.998 2.997 3.000	2.998	0.4197	350	
9	4.028 4.034 4.023	4.028	3.009 3.010 3.011	3.010	0.3999	329 ~~328.7383~~	
10	4.004 3.997 4.002	4.001	3.011 2.998 2.998	3.002	0.4168	347 ~~346.7899~~	
11	4.015 4.010 4.010	4.012	3.003 3.009 3.012	3.006	0.3828	316 ~~316.3564~~	
12	4.005 4.005 4.006	4.005	3.007 3.004 3.006	3.006	0.3999	332 ~~331.5066~~	
13	4.005 4.005 4.003	4.004	2.998 2.997 3.001	2.999	0.4096	341 ~~341.2147~~	
14	4.017 4.026 4.025	4.023	3.010 3.011 3.010	3.010	0.4193	345 ~~345.1145~~	
15.	4.029 4.023 4.022	4.025	3.008 3.006 3.008	3.007	0.4193	346 ~~345.6316~~	

***** Flexure testing data sheet *****

Date : 19/3/98 4 point(✓) or 3 point ( ) 40/20

Temperature : ℃ Material : Al_2O_3

No	width, w (mm)		thickness, t (mm)		Fracture load (kN or kgf)	Strength(MPa)	Remarks
16.	4.009 4.005 4.004	4.007	3.002 2.993 2.992	2.996	0.4193	350 ~~349.7389~~	
17	4.003 4.005 4.005	4.004	3.008 3.002 3.002	3.003	0.4193	348 ~~348.3705~~	
18	4.001 4.000 4.003	4.001	2.996 2.999 2.998	2.998	0.4085	341 ~~340.7858~~	
19	4.002 4.003 4.002	4.002	2.988 3.000 3.003	2.997	0.4082	341 ~~340.5777~~	
20	4.003 4.006 4.010	4.006	3.001 2.999 3.001	3.000	0.4196	342 ~~349.1430~~	
21	4.011 4.012 4.014	4.012	3.002 3.002 3.001	3.002	0.4149	344	
22	4.012 4.020 4.017	4.016	3.002 3.005 2.999	3.002	0.3570	296	
23	4.002 4.004 4.002	4.004	2.999 2.999 3.001	3.000	0.4319	~~347~~ 360	
24	4.005 4.008 4.010	4.008	2.994 2.996 3.002	2.997	0.4161	~~340~~ 347	
25	4.006 4.009 4.009	4.009	3.002 3.002 3.002	3.002	0.4090	~~331~~ 340	
26	4.020 4.020 4.029	4.021	3.011 3.014 3.011	3.012	0.3899	321	
27	4.002 4.007 4.011	4.007	2.997 3.000 3.004	3.000	0.3727	~~362~~ 310	
28	4.004 4.005 4.009	4.004	3.000 3.012 3.005	3.006	0.4361	~~385~~ 362	
29	4.019 4.013 4.017	4.016	3.016 3.012 3.013	3.013	0.4069	335	
30.	4.009 4.006 4.005	4.007	3.004 3.031 3.002	3.012	0.4196	346	

***** Flexure testing data sheet *****

Date : 1998. 3. 19 4 point( ) or 3 point ( ) 30/10

Temperature : ℃ Material : Al_2O_3

No	width, w (mm)		thickness, t (mm)		Fracture load (kN or kgf)	Strength(MPa)	Remarks
1	4.018 4.015 4.016	4.016	3.009 3.011 3.010	3.010	0.4121	340	
2	3.918 3.917 3.917	3.918	3.008 3.014 3.010	3.011	0.3897	329	
3	4.016 4.017 4.021	4.018	3.021 3.015 3.014	3.017	0.3079	252	
4	4.008 4.003 3.999	4.003	3.006 3.006 3.011	3.008	0.397	329	
5	3.917 3.915 3.913	3.915	3.003 3.004 3.006	3.004	0.3745	318	
6	4.006 4.009 4.011	4.009	3.011 3.015 3.014	3.013	0.3953	326	
7	3.919 3.918 3.918	3.918	3.005 3.002 3.000	3.002	0.3973	338	
8	4.015 4.014 4.015	4.015	3.015 3.009 3.010	3.011	0.3873	319	
9	4.019 4.019 4.026	4.021	3.012 3.007 3.006	3.008	0.3735	308	
10	4.024 4.024 4.021	4.023	3.003 3.006 3.010	3.006	0.4204	347	
11	4.015 4.021 4.017	4.018	3.007 3.009 3.010	3.009	0.4100	338	
12	4.010 4.011 4.007	4.009	3.006 3.006 3.009	3.007	0.3845	318	
13	4.015 4.014 4.008	4.012	3.008 3.009 3.010	3.009	0.4017	332	
14	4.025 4.029 4.019	4.024	3.010 3.010 3.012	3.011	0.4051	333	
15	4.028 4.024 4.020	4.024	3.011 3.018 3.019	3.016	0.4012	323	

***** Flexure testing data sheet *****

Date : 1998.3.19 4 point(✓) or 3 point ( ) 30/10

Temperature : ℃ Material : Al_2O_3

No	width, w (mm)		thickness, t (mm)		Fracture load (kN or kgf)	Strength(MPa)	Remarks
16	4.010 4.010 4.005	4.008	3.007 3.006 3.005	3.006	0.4176	346	
17	4.012 4.009 4.009	4.010	3.010 3.011 3.008	3.010	0.4179	345	
18	4.016 4.017 4.018	4.017	3.018 3.014 3.016	3.016	0.3881	318	
19	4.018 4.019 4.020	4.019	3.005 3.006 3.008	3.006	0.4017	332	
20	4.022 4.021 4.023	4.022	3.014 3.014 3.016	3.015	0.4037	338	
21	4.008 4.005 4.001	4.005	3.003 3.007 3.007	3.008	0.4082	338	
22	4.019 4.017 4.021	4.019	3.005 3.005 3.012	3.007	0.3506	289	
23	4.002 4.006 4.011	4.006	3.014 3.013 3.012	3.013	0.3948	326	
24	3.919 3.919 3.917	3.918	2.999 3.003 3.010	3.003	0.4017	341	
25	4.020 4.020 4.023	4.021	3.016 3.007 3.007	3.010	0.3847	317	
26	4.015 4.015 4.016	4.015	3.009 3.006 3.013	3.009	0.4105	339	
27	4.024 4.023 4.021	4.023	3.010 3.006 3.008	3.008	0.4003	330	
28	4.022 4.023 4.021	4.022	3.013 3.005 3.006	3.007	0.3974	328	
29	4.019 4.021 4.026	4.022	3.011 3.003 3.007	3.007	0.4022	332	
30	3.917 3.920 3.917	3.918	3.007 3.002 3.007	3.005	0.3773	320	

Annex C. Weibull analysis results of flexural strength data.

40/20

Number of data = 30
Weibull modulus = 27.331
Characteristic strength = 344.948
Unbiased Weibull modulus = 26.137

90% confidence bounds

m	(q_0.05	q_0.95)	m_upper	m_lower
	.8227	1.3283	31.7683	19.6775
S_o	(t_0.05	t_0.95)	S_upper	S_lower
	-.3353	.3306	349.4021	340.6127

	S_o	m	S_av	variance
MLE	344.9480	26.1371	337.8127	16.1424
Linear model	346.0866	20.4738	337.1112	20.4243
Best LSE	345.6790	22.6983	337.5264	18.5022
Median rank	345.8523	21.6784	337.3421	19.3362
? model	346.0248	22.5236	337.8056	18.6572

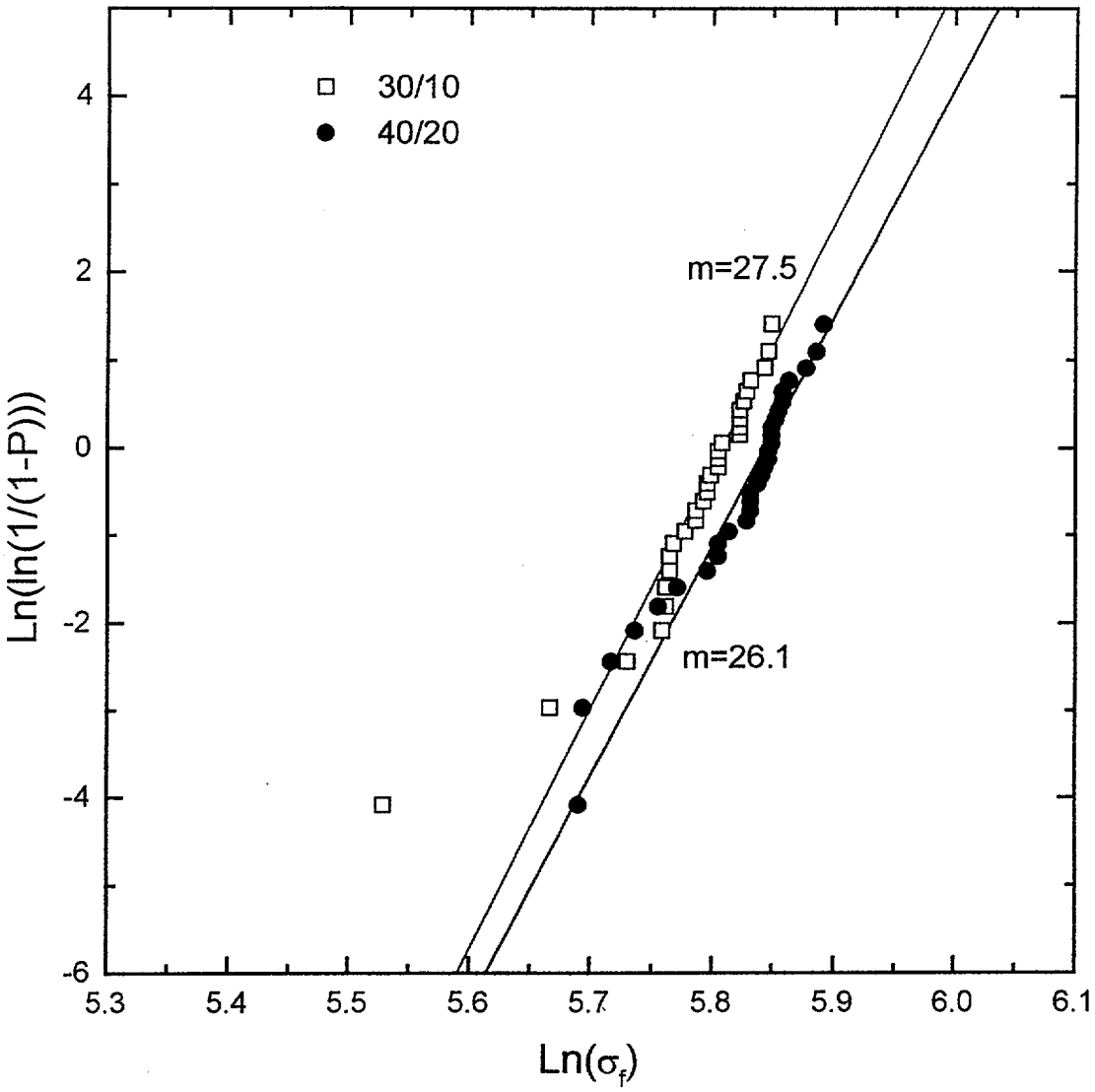
30/10

Number of data = 30
Weibull modulus = 28.753
Characteristic strength = 333.204
Unbiased Weibull modulus = 27.497

90% confidence bounds

m	(q_0.05	q_0.95)	m_upper	m_lower
	.8227	1.3283	33.4215	20.7016
S_o	(t_0.05	t_0.95)	S_upper	S_lower
	-.3353	.3306	337.2925	329.2224

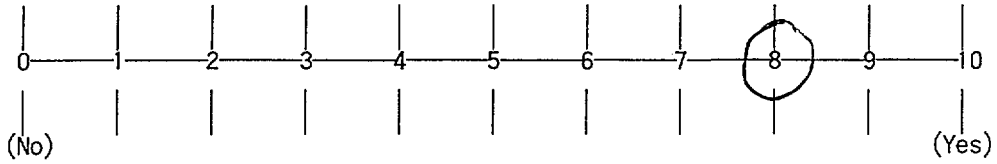
	S_o	m	S_av	variance
MLE	333.2042	27.4973	326.6315	14.8545
Linear model	336.5234	16.5020	325.9108	24.3180
Best LSE	335.7722	18.7621	326.3419	21.5149
Median rank	336.1062	17.6968	326.1531	22.7509
? model	336.1677	18.6369	326.6688	21.6762


30/10
40/20
m=27.5
m=26.1
Ln(ln(1/(1-P)))
Ln(σf)
4
2
0
-2
-4
-6
5.3
5.4
5.5
5.6
5.7
5.8
5.9
6.0
6.1

Annex D. Evaluation sheets answered by participants.

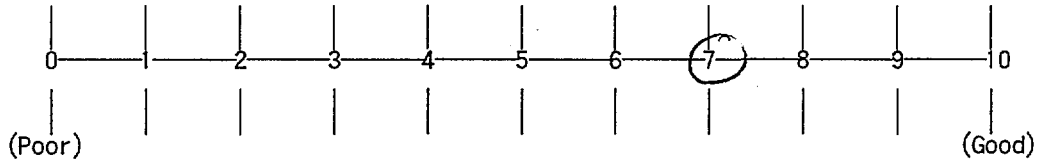
Evaluation sheet for ICMET Workshop/Training on Fine Ceramics

1. Do you think this workshop/training program helpful to you? Please mark below.



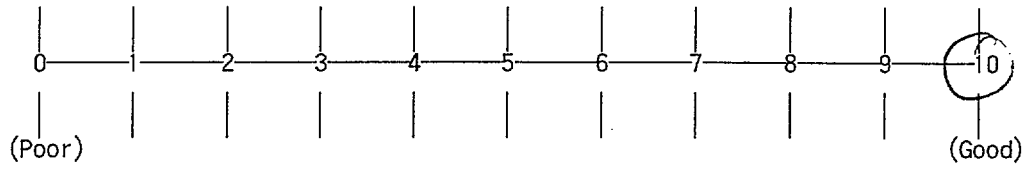
Comments : Advantage of this workshop/ Training is to generalize and standardize Technical skills for conducting material testing among member countries.

2. What do you think of the lectures given in the workshop?



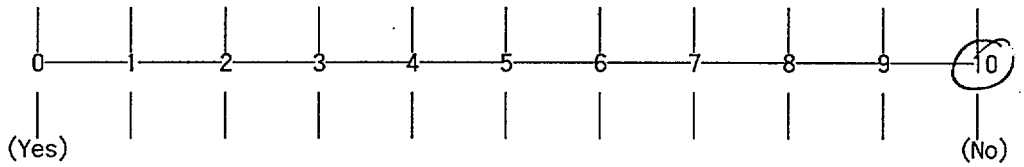
Comments : If theorical aspects was more concentrated in the lecture, it would provide more transparent view of testing principle

3. What do you think of the practical sessions?



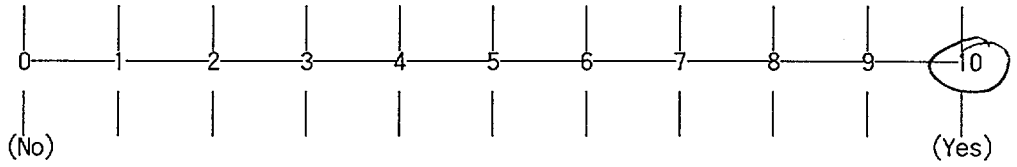
Comments : excellent facilities and preparation

4. Did you have any difficulties in transportations, eatings, etc, during the workshop/training?



Comments :

5. Do you want the ICMET Project on "Fine Ceramics" to be continued?



If you want, what subjects do you want to be addressed in the project?

All aspects of mechanical testing should be addressed including internal standard eg. IEC/ISO guide 25, ISO 9000 series

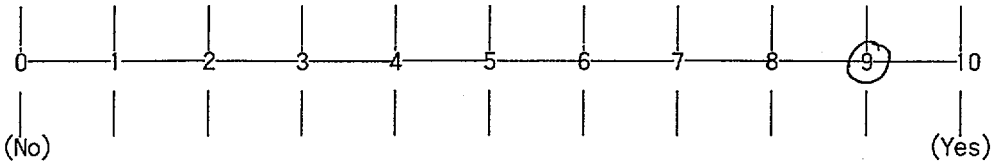
6. Other comments on this Workshop/Training or on the other ICMET projects.

-

Name: SUTIPORN CHEWASATN Signature: S. Chewasatn

Evaluation sheet for ICMET Workshop/Training on Fine Ceramics

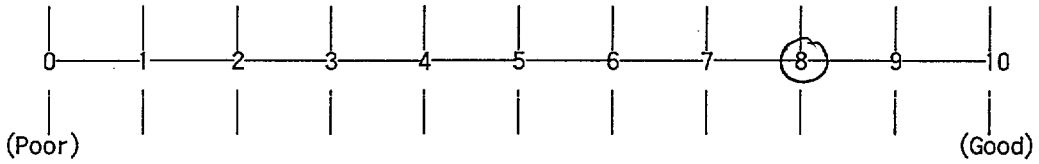
1. Do you think this workshop/training program helpful to you? Please mark below.



Comments :

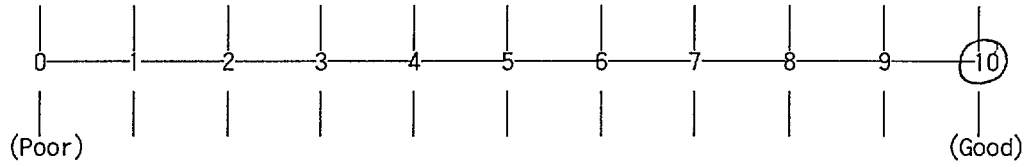
I think this Workshop / training program makes to understand about Bending test / hardness / fracture toughness / etc.

2. What do you think of the lectures given in the workshop?



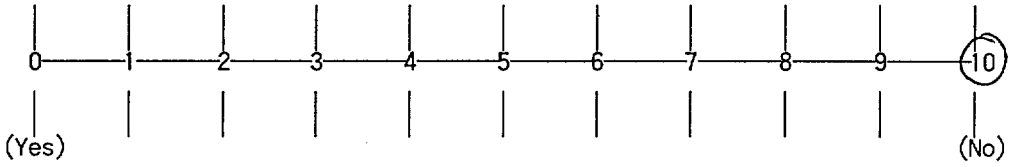
Comments :

3. What do you think of the practical sessions?



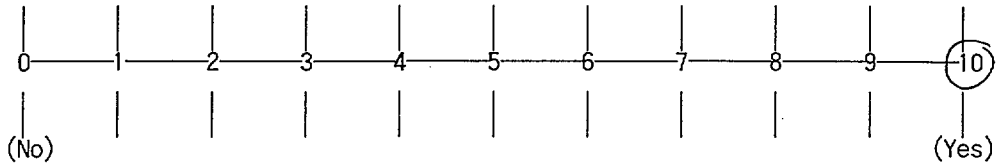
Comments : Excellent facilities and preparation

4. Did you have any difficulties in transportations, eatings, etc, during the workshop/training?



Comments :

5. Do you want the ICMET Project on "Fine Ceramics" to be continued?



If you want, what subjects do you want to be addressed in the project?

ISO 9000 series , IEC/ISO guide 25

6. Other comments on this Workshop/Training or on the other ICMET projects.

This Workshop/Training should have many participants because we can exchange knowledge and experience.

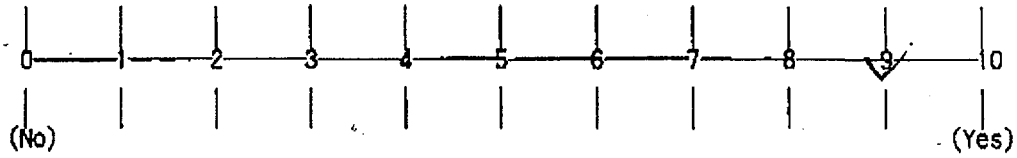
Name : WASANA KHONGWONG Signiture : Wasana Khongwong.

Fax to : Dr. Seong-Jai Cho

From : Lin Xiao
SRIM, China

Evaluation sheet for ICMET Workshop/Training on Fine Ceramics

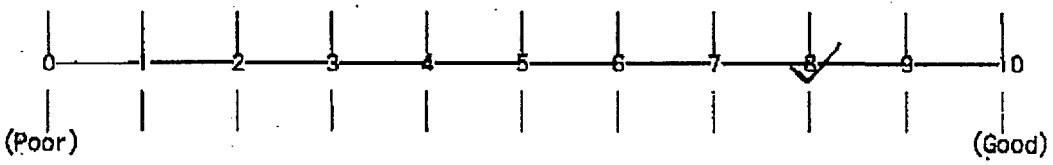
1. Do you think this workshop/training program helpful to you? Please mark below.



Comments :

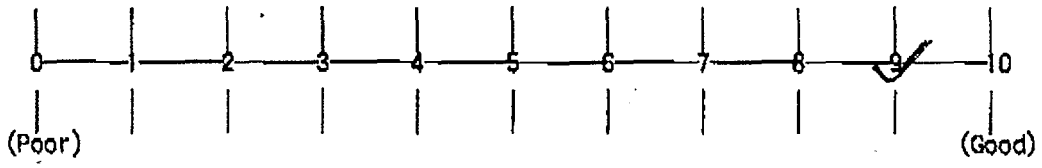
Through this workshop I exchange my study works with other and learn a lots of information about fine ceramics.

2. What do you think of the lectures given in the workshop?



Comments :

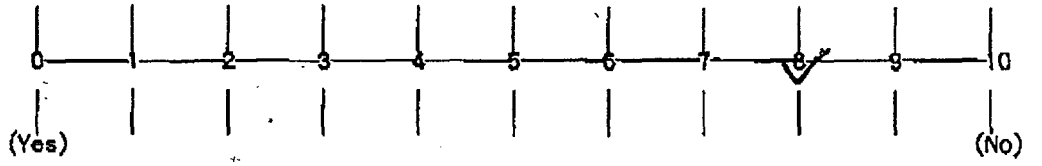
3. What do you think of the practical sessions?



Comments :

Before this workshops I always do the test work of ceramics myself but don't know much the specimen, this time I know the abts inclose the machined specimen and precracked specimen.

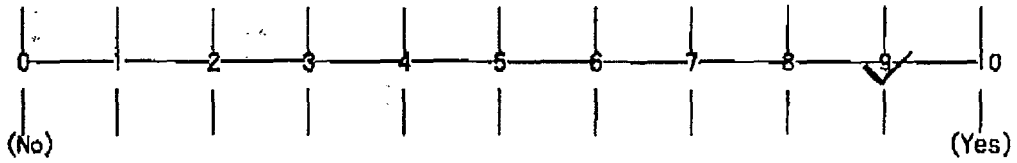
4. Did you have any difficulties in transportations, eatings, etc, during the workshop/training?



Comments :

I amit accustomed a little to the Korea food.

5. Do you want the ICMET Project on "Fine Ceramics" to be continued?



If you want, what subjects do you want to be addressed in the project?

As the ceramics are used mainly in high temperature circumstance, so I think the next subject of project would be the high temperature property of ceramics.

6. Other comments on this Workshop/Training or on the other ICMET projects.

Name: Ling Xiao Signature

Financial Statement of Expenses

(Unit : US dollar)

Items	Expenses
Hotel Accommodation Charges	$60/night x 7 nights x 3 persons = *$1,260*
Boarding	$45/day x 7 days x 3 persons = *$945*
Remuneration for Lecturers	- $40/hour x 10.5 hour = *$420* - $35/hour x 10.5 hour = *$367.5*
Reception/Farewell Party	- $30/person x 10 persons = *$300*(Reception) - $30/person x 10 persons = *$300*(Farewell Party)
Miscellaneous (including costs for reproduction, communications, etc.)	$359.25(10% of total costs)
Fixtures and Specimens for Round Robin Test(RRT)	RRT for flexural strength: *$7,700* - 4 point bend fixture with rotating rollers($3,000) - Specimens($4,700) RRT for fracture toughness: *$7,300* - Bridge indentation fixture with acoustic sensor($6,000) - Specimens($1,300)
Total	**$18,951.75**