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### 22296

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# Workshop on Testing and Evaluation of Mechanical Properties of Ceramics

March 16 ~ 21, 1998

Submitted by Dr. Seong-Jai Cho
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#### 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

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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

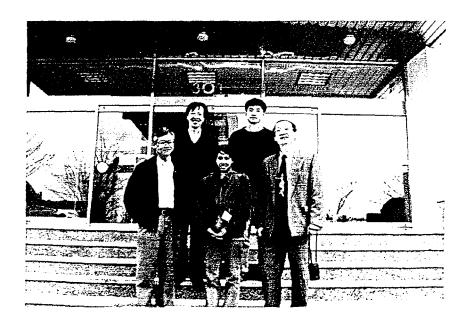
For evaluation of the workshop, a questionnaire was distributed to each participant. (\* The results are attached in Annex D).

#### 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



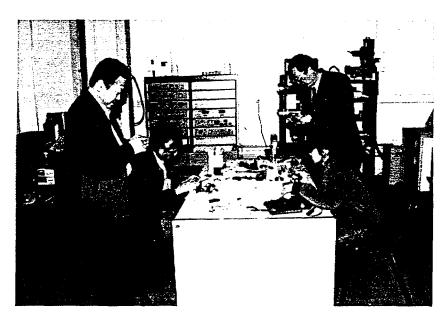
Participants and trainers, Dr. Stiporn, Ms. Wassala and Mr. Ling from left of the front, Dr. Cho and Dr. Yoon, from left of the rear.



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.

4 point( $\checkmark$ ) or 3 point ( ) Material :  $\iint_{\mathbb{R}^n} f(x) dx$ 

4-0/20

Temperature:

`emperat	ture :	$^{\circ}$	IV	Iaterial: #	t1203		
No	width,	, w (mm)	thicknes	s, 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	367	just break
2	A 004 A 013 A 010	4.010	3.004 3.001 2.999	3.001	0. 3996	332	
3	4.004 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	4.00% 4.00b 4.007	4.007	3.00b 3.003 2.999	3.003	0.4142	304	
6	1.010 1.011 1.010	4.010	3.000 3.00b 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, 00%	2.998 2.997 3.000	2.998	0.4197	350	
9	4.028 1.034 4.023	4.028	3.009 3.010 3.011	3.010	0.3999	329 328.7363	
10	4.004 3.994 4.002	4.001	9,011 2,998 2,998	3.002	0.4168	3 49 346.7839	-
11	4.015 4.010 4.010	4.012	3.003 3.009 3.042	3 006	0.3828	316 316.3564	
12	4.005 4.005 4.006	4.005	3.007 3.004 3.006	3 00b	0.3999	332 331-5066	
13	4.005 4.005 4.003	4.004	2.998 2.997 3.001	2.999	0.4096	34/ 341, 2197	· ·
14	4.014 4.026 4.025	4.023	3.010 3.011 3.010	3.010	0.4193	345,1145	
15.	4.029 4.023 4.022	4.025	3.008 3.00b 3.008	3.007	0.4193	346 345.6316	

Date:

19/3/98

4 point(✓) or 3 point ( )

4%20

Temperature:

 ${\mathbb C}$ 

Material: Aloga

1 CHIPCIA	temperature: C iviateriai · H <sub>2</sub> U <sub>3</sub>						
No	width	, w (mm)	thicknes	ss, t (mm)	Fracture load (kN or kgf)	Strength(MPa)	Remarks
	4,009		3.002			350	
16.	4.00%	4.007	2,993	2.996	0000	349.7383	
	4.008	4.007	2.992	2.496	0.4193	97 (1.100)	
	4.003		3.00%			348	
17	4.005	4.004	3.002	3.003	0 11120	348,3709	••
17	4.005	1	3.002	1 0	0.4193	240,9104	
	4.001		2096			341	
18	4.000	4.001	2.999	2.998	0.4085	340.7858	
	4.003		2.998	1 2-110	0,7003	210, 4076	
	4.002		2.988			341	
19	4.003	4.002	3 000	2.997	0.4082	340,677	
(1)	4.002	4.002	3.003	1 2-11		उपरा, ७१न र	
	4.003		3, 001		,	34P	=4,6
20	4.006	4.00b	2.999	3.000	o and	. , , ,	
1 20	4.010		3. CO1	7.000	0.4196	349.1430	
	4.011		3.002		<del> </del>		
21	4.012	4.012	3 002	3.002	0.4149	3/1/1	
71	4.014	4.0.2	3.001		0.4149	344	
	4.012		3.002				
22	4,020	4.01b	3.005	3.002	V 3632	901	
``	4.017	(	2.999	7,007	0.3570	296	
	4,002	4.004	2.999	3.000	0.4319	347360	
23	4,004		2.999				•
	4.002	T.VU (	3,001				
	4.005		2.994				
24	4,008	4.00%	2.496	2.999	0.4161	340341	
	4.010		3.002	- (17		1 240 24-/	
	4.00%	4.009	3.002	3.002	0.4090	391340	
25	4.009		3,002				
<i>L</i> )	4.009	¥	3.002				
	4.020		3.011				
26	4.020	4.021	3.014	3.012	0.3899	321	
	4.020		3.011				
27	4.002	4.007	2.997	3.000	0.8727	<del>962</del> 310	
	4.007		3,000				
	4.011		2.004			-5000.0	
	4.004		3.000				
28	4.005	4.004	3,012	3.006	n (12/1	385 362	
	4.009		3.005	0. 5 -	0.4361	50,70	
29	4.019	4.01b	3,016	3.013	0.4069		
	4.013		3,012			335	
	4.017		3.013		U-700"	7/1	
	4.009		3.004				
<i>3</i> 0.	4.006	4.007	3.031	3.012	0-4196	346	
	4.005	7.001	3.002	1	0-7191	1240	
	•						

 Date: 1998.3.19
 4 point( ) or 3 point ( )

 Temperature:
 C
 Material: AL>D;

No width, w (mm) thickness, t (mm) Fracture load (kN or kgf)  4 0 18	remperau	.u.c •	· · · · · · · · · · · · · · · · · · ·	т.	iaueriai ·	79-203	, , , ,	
1	No	width,	, w (mm)	thicknes	s, t (mm)	1	Strength(MPa)	Remarks
1		1.018		3008				
2 3.517 3.718 3.014 3.017 0.3897 329  3 40.6 3.017 4.018 3.017 4.018 3.017 4.018 3.017 4.018 3.017 4.018 3.017 4.018 3.017 4.018 3.017 4.018 3.017 4.018 3.017 3.019 3.008 0.397 329  5 3.517 3.515 3.008 3.008 0.397 329  5 3.517 3.515 3.008 3.018 3.018 6.019 3.019 3.019 0.3973 326  6 4.009 4.009 3.017 3.013 0.3973 326  7 3.518 3.018 3.018 3.017 3.013 0.3973 328  8 4.011 4.011 3.011 3.011 0.3873 319  9 4.011 4.015 3.001 3.011 0.3873 308  10 4.024 4.023 3.006 3.006 3.006 0.4204 347  11 4.021 4.028 3.006 3.006 4.024 3.001 4.021 4.021 3.006 3.006 3.006 4.021 4.021 3.006 3.006 3.006 3.006 4.021 3.021 4.021 3.021 3.021 0.4051 3.021 0.			16.016		7 5 18	0.00.1.0.1	2110	
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Date: 1938.3.18

 $^{\circ}$ 

Temperature:

4 point() or 3 point ()

30/10 Material: A1203

width, w (mm) thickness, t (mm) Fracture load Strength(MPa) No Remarks (kN or kgf) 3.007 4.010 346 4.010 4008 3,006 0.4176 16 3,006 4.005 7.005 4.012 7,010 0.4179 345 3.010 4.010 4,009 3.011 1) 4.003 3.008 4016 3.018 0.3881 319 4.017 4.017 3016 3.014 18 4.018 3.016 4.018 3.005 332 0.4017 4.019 3,006 3.006 4019 19 3.008 4022 3.014 338 0.4037 4.022 210.5 4.021 3.014 20 4.023 2.016 4,008 338 -4.005 3.008 4.005 3,007 0.4082  $\nu$ I 4.001 3.007 4.019 3.005 0.3506 289 3,007 4.019 4017 3.005 22 4021 3.012 4.002 3.014 32b 4.006 4.006 3.013 3.013 0.3948 23 4011 7.012 3.919 2.999 341 7.919 3.918 3.003 3.003 0.4617 24 3.917 4.020 3,016 317 4.020 0.3847 4.021 3.04.0 25 4.023 3.007 4015 339 0.4105 4.015 3009 4015 26 4.016 3.013 4.024 3,010 330 27 4023 3008 0.4603 4.023 3,006 4.021 3.008 4.022 3.013 4023 4.022 0.3974 328 3.005 3.007 28 4.021 3.006 4.019 3.011 4.022 0.4022 332 4.021 3.003 29 3.007 4026 3.917 3.007 320 3.918 0-3773 3.920 3002 3005 30

Annex C. Weibull analysis results of flexural strength data.

Number of data = 30 Weibull modulus = 27.331 Characteristic strength = 344.948 Unbiased Weibull modulus = 26.131

Chibiasca Weibah modulus 20.15:

90% confidence bounds (q\_0.05 q\_0.95) m\_lower m\_upper .8227 1.3283 19.6775 31.7683 S\_o (t\_0.05 t\_0.95) S\_lower S\_upper -.3353 .3306 349.4021 340.6127  $S_o$ m S\_av variance MLE 344.9480 26.1371 337.8127 16.1424 20.4738 337.1112 20.4243 Linear model 346.0866 Best LSE 18.5022 345.6790 22.6983 337.5264 Median rank 345.8523 21.6784 337.3421 19.3362

22.5236 337.8056

18.6572

30/10

? model

Number of data = 30

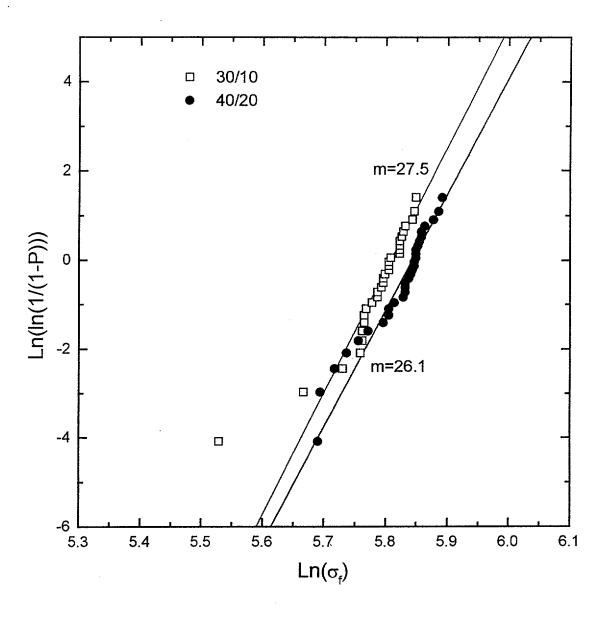
Weibull modulus = 28.753

Characteristic strength = 333.204

Unbiased Weibull modulus = 27.497

346.0248

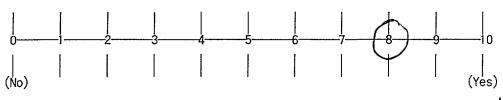
90% confidence bounds ( q\_0.05 q\_0.95) m\_lower m\_upper .8227 1.3283 33.4215 20.7016 S\_o (t\_0.05 t\_0.95) S\_upper S\_lower 329,2224 -.3353.3306 337.2925 S\_o m S\_av variance MLE 27.4973 326.6315 14.8545 333.2042 Linear model 336.5234 16.5020 325.9108 24.3180 Best LSE 21.5149 335.7722 18.7621 326.3419 Median rank 17.6968 326.1531 22.7509 336.1062 ? model 336.1677 18.6369 326.6688 21.6762



Annex D. Evaluation sheets answered by participants.

### Evaluation sheet for ICMET Workshop/Training on Fine Ceramics

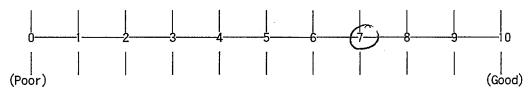
1. Do you think this workshop/training program helpful to you? Please mark below.



Comments: Advantage of this workshop

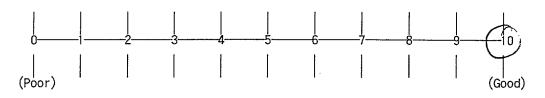
Training is to peneralize and standardize Technical skills for conducting material techniq amoung member countries.

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



Comments: If theorical aspects was more consentrated in the lecture, it would provide more fransparent view of festing principle

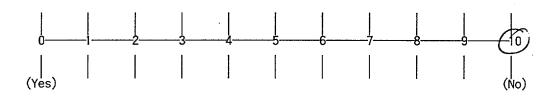
3. What do you think of the practical sessions?



Comments:

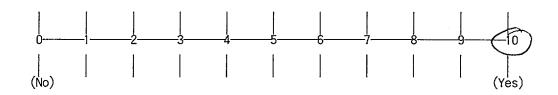
excellent facilities and preparation

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: eq. 150/150 gurde 25, 150 9000 series

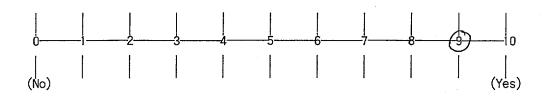
6. Other comments on this Workshop/Training or on the other ICMET projects.

Name: SUTIPORN CHEWASATN

Signiture: S. Chewasahi

## Evaluation sheet for ICMET Workshop/Training on Fine Ceramics

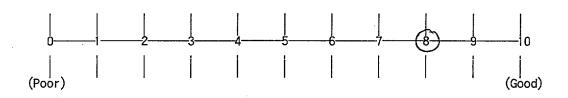
1. Do you think this workshop/training program helpful to you? Please mark below.



#### Comments:

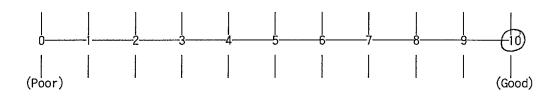
I think this Workshop/training program makes to understand about Burding test/hardness/tracture toughness/etc.

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



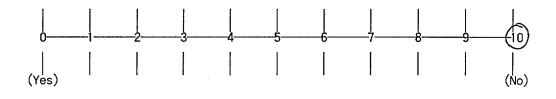
Comments:

3. What do you think of the practical sessions?



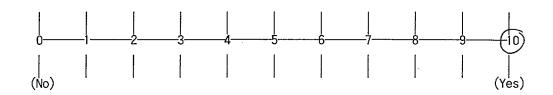
Comments: Excellent facilities and preparation

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?

130 9000 series, IEC/Iso quide 25

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

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

Name: WASANA KHONŒWONŒ Signiture: Marana Khongungo.

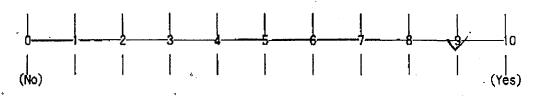
Fax to: Dr. Seong-Jai Cho

From: Lin Xiao

SRIM, China

### Evaluation sheet for ICMET Workshop/Training on Fine Ceramics

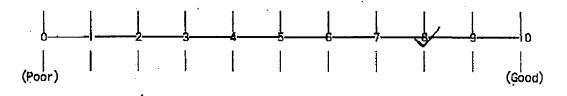
1. Do you think this workshop/training program helpful to you? Please mark below.



#### Comments:

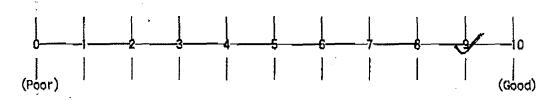
Through this workshop I exchange my sludy works with other and learn a lots of information about fine ceramics.

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



Comments :

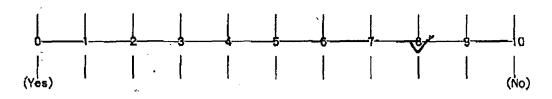
3. What do you think of the practical sessions?



#### Comments:

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

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

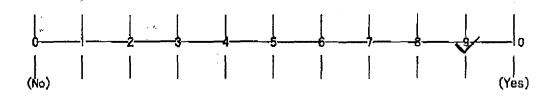


Comments:

a withe

I amn't accustomed to the Konea 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 subject of propert would be the high temperature property of ceramics.

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

Name: Ling Xiao

Signiture Big

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#### **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. <u>75</u>