

IIC

BULLETIN



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CONTENTS

Editorial Desk	1
INSTITUTE ACTIVITIES	2
STUDENT SECTION	6
TECHNICAL SECTION		
1. Ultra High Temperature Ceramics : Use and its Prospect	8
<i>by Shirshendu Chakraborty</i>		

Editorial Desk

Dear Members,

It is of immense pleasure to bring out the third IIC –e bulletin. The main intention of this bulletin is to make our members keep abreast of the continual development taking place in different field of glass and ceramics. It also covers the various activities of Indian Institute of Ceramics. The delay in publishing this issue is due to unavailability of the articles. We are hopeful that our distinguished members will contribute by sharing their knowledge and expertise in the forthcoming issues which will be beneficial for all. Student members are also encouraged to contribute with their papers which will help them to develop their skill of scientific paper writing.

Wishing you all the best,

Thanking you,

Somnath Sinhamahapatra

Editor

INSTITUTE ACTIVITIES

Highlights of the 41st Annual Session, December 2015

41st Annual Session of Indian Institute of Ceramics, held on 15th December, 2015.

Joint Inaugural Session

The joint Inaugural Ceremony of the 78th Annual Session of The Indian Ceramic Society, 66th Annual Session of All India Pottery Manufacturers' Association and 41st Annual Session of Indian Institute of Ceramics was held on December 15, 2015 at Christ University, Bangalore.

Welcome Song

In the inaugural ceremony, Smt. Monika Shah, an eminent classical vocalist of Gujarat performed a solo classical vocal recital. This was of very high standard. In the history of Annual Sessions, for the first time the inaugural programme was preceded by a cultural programme and everybody enjoyed and appreciated the same.

About 200 delegates from all over India and abroad comprising scientists, technologists, students, equipment manufacturers, raw material suppliers and end-users in the field of glass and ceramics, refractories and allied disciplines, academic institutions, R&D organizations as well as guests and invited speakers actively participated in the Session.

Inaugural Programme

After the inaugural song, Dr. S.N. Misra, Scientist-in-Charge, CSIR-CG&CRI, Naroda Centre and Convener, GTGC-2013 was requested to escort the Chief Guest, Shri Maheshwar Sahu, IAS, Principal Secretary, Govt. of Gujarat and the Guest of Honour, Shri Kamal Dayani, IAS, Industries Commissioner, Govt. of Gujarat and other dignitaries on the dias.

Mr. G.G. Trivedi, Executive Director, Somany Ceramics Ltd., Gujarat and Chairman, Organizing Committee especially rendered his hearty welcome to Dr. S.N. Misra Scientist-in-Charge, CSIR-CG&CRI, Naroda Centre and Convener, GTGC-2013, Dr. A.K. Chattopadhyay, Vice President,

InCerS, Prof. Arun Kr. Varshneya, Professor of Glass Science, The New York State College of Ceramics, Alfred University, USA, Shri Rupesh C. Shah, Chairman, Gujarat Chapter, InCerS, Shri S.K. Kudaisya, Chief Patron & Managing Director, Sabarmati Gas Ltd., Shri Subhash Dave, Chief Patron & Managing Director, Sabarmati Gas Ltd., Chief Guest, Shri Maheshwar Sahu, IAS, Principal Secretary, Govt. of Gujarat and the Guest of Honour, Shri Kamal Dayani, IAS, Industries Commissioner, Govt. of Gujarat for their gracious presence on the occasion. Shri Maheshwar Sahu, IAS, Principal Secretary, Govt. of Gujarat and Chief Guest, 76th Annual Session of the Indian Ceramic Society officially inaugurated the occasion by lighting the ceremonial lamp.

Annual General Meeting :

The 41st Annual General Meeting of Indian Institute of Ceramics was held at Christ University, Bangalore. Dr. Arup Kumar Chattopadhyay, President of the Institute was in the Chair. The meeting was attended by about 150 members.

Annual Report on the activities of the Institute during the year 2015

Dr. Arup Ghosh, Hony Secretary presented a brief summary of the activities of the Institute during the year 2015. The report contained informations about the activities of the Institute during the year which were as follows :

(A) Committee Meetings :

During the period from January to December, 2011 a series of meetings were held as stated below :

Council – One (1 No.), Executive Committee – Two (2 Nos.), Examination Committee – Two (2 Nos.), Publication Committee – One (1 No.)

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9. Dr. Barundeb Mukherjee	Member
10. Dr. Shankar Ghatak	Member
11. Mr. Somnath Sinhamahapatra	Convener

A.I.I.Ceram. Examination report — 2014

A total of ninety-nine (99) students in new syllabus and twenty-nine students (29) in old syllabus were enrolled in 2015 for A.I.I.Ceram. Examination, out of which ninety-three (93) students appeared for new syllabus and Twenty-three (23) students appeared for old syllabus during October, 2015 at

Kolkata, Chennai, Bangalore & Khurja Centres. The theoretical examination was held during the period from 27th October to 3rd November, 2015 in the above four centres and the practical & viva-voce examination was held from 6th November to 8th November, 2015 at Kolkata centre only..

Biometric System

A biometric system has been introduced in the office for recording the attendance of the office staff. By adopting this system the attendance have become more transparent.

STUDENTS' SECTION

A.I.I.CERAM. EXAMINATION RESULT — 2014

On the recommendations / approval of the Examination Committee the results have been published on 12th February, 2015. An overall analyses of the result is tabulated as below followed by the detailed list of pass-out candidates (students).

No. of candidates enrolled	– 50 (Old Syllabus)
No. of candidates enrolled	– 89 (New Syllabus)
No. of candidates appeared in the examination	– 39 (Old Syllabus)
No. of candidates appeared in the examination	– 85 (New Syllabus)
No. of candidates absent	– 11 (Old Syllabus)
No. of candidates absent	– 04 (New Syllabus)
No. of candidates appeared for the Final part of the examination	– 32 (Old Syllabus)
No. of candidates appeared for the Final part of the examination	– 18 (New Syllabus)
No. of successful candidates	– 13 (Old Syllabus)
No. of successful candidates	– 05 (New Syllabus)
Percentage of passed candidates in Final examination	– 40.6% (Old Syllabus)
Percentage of passed candidates in Final examination	– 27.78% (New Syllabus)

Grade (Old Syllabus)

1 st Class	2 nd Class	Pass (P)
02	11	Nil

Grade (New Syllabus)

1 st Class	2 nd Class	Pass (P)
01	04	Nil

List of successful candidates for the year 2015

Sl. No.	Name	Registration No.	Roll No.
1.	Ms. G. Shilpa	IC-111967	N144008
2.	Mr. Atul Kumar	IC-111993	N147015
3.	Mr. Satendra Singh	IC-111998	N147019
4.	Mr. Alpesh Jayasukhlal	IC-122023	N144028
5.	Mr. Aditya Mishra	IC-122055	N147044
6.	Mr. Nishant Sharma	IC-081647	147010
7.	Mr. Dalveer Singh	IC-081664	141011
8.	Mr. Satendra Singh	IC-081667	141012
9.	Mr. Rajesh Kumar	IC-081674	141013
10.	Mr. B. Nagaraju	IC-081683	146015
11.	Mr. Jalaramaiah	IC-081685	146016
12.	Mr. S. Vijay Kumar	IC-081713	146017

AWARDS

For the year 2014 Examinations, the Examination Committee has decided to bestow “**SAHAJ MEMORIAL AWARD**” on Ms. G. Shilpa (Registration No. IC111967 & Roll No. N144008) for the best performance securing the highest total marks (576) in the part-II examination and “**AMIC PRIZE**” on Mr. Hari Babu Challari (Registration No. IC091793 & Roll No. 146027) securing the highest total marks (121) in Ceramic Science – I & II (Section – A).

Ultra High Temperature Ceramics: Use and its Prospect

Shirshendu Chakraborty

CSIR-Central Glass and Ceramic Research Institute, Kolkata 700032

Abstract : Ultra high temperature ceramics (UHTC) categorically used as leading edge and nose cone of rocket and supersonic vehicle, jet engines and other conventional sectors like fuel components of high temperature nuclear reactors, electrodes, cutting tools, etc. Transition metal based borides and carbides are mainly classified into these category. Because of strong kinetic restrictions of sintering like high covalent bonding, low diffusion rate and oxygen impurities on raw powder particle surface, both high temperature and external pressure are essential for consolidation of UHTCs. Many groups attempted various compositions and techniques for consolidation of UHTC based systems without deteriorating the high temperature desired structural properties. The uses and the future prospect of UHTCs and its composites are reviewed.

Keywords: UHTC; Aerospace; Structural Ceramics.

1. Introduction

Hypersonic programme will propose the next generation higher speed and altitude flight vehicles which technically operate at speeds beyond Mach 5 [1]. Many hypersonic programmes like National Aero Space Plane Programme (NASP), Hyper-X, FALCON, HyShot, HyCAUSE etc. have already taken part in different advanced countries. However, still many unsolved problems exist due to aerodynamic friction and thermal stresses. In material aspect, the classification is mainly based on the temperature range in which they can operate incessantly on an airframe. Titanium alloy and metal matrix composites are generally use in the temperature range of 300-900°C and above that ultra high temperature ceramics and carbon-carbon composites are mainly used. We will start by discussing only non-oxide based UHTCs.

The ultra high temperature ceramics (UHTCs) are a very advanced class of ceramic materials that can sustain their both physical and structural properties and integrity at extremely high temperature ($\geq 1800^\circ\text{C}$) and also possess good chemical resistance and oxidation resistance even in adverse and corrosive environments [2-4]. The UHTC materials were first extensively studied between 1960s and 1970s by

different Russian and U.S Laboratories for the testing of hypersonic flight with sharp leading edges connected with planetary atmosphere re-entry and most systematic effort was initially executed by the Man Labs Inc. (Cambridge) company, under a research program funded by the Air Force Materials Laboratory (AFML), US [3, 5-7]. However, the growth of UHTC components has been initially slightly discontinuous and again rekindled as NASA Ames required for two high performance aerospace experiments, SHARP-B1 (1997) and SHARP-B2 (2000) (Sharp Hypersonic Aero-thermodynamic Research Probes) [6]. These two successful trials in collaboration with the US Air Force and Sandia National Labs briefly tested a nose tip and sharp leading edges composed of UHTC materials (HfB_2 and $\text{ZrB}_2\text{-SiC}$) to tangible re-entry atmospheres. After these testings, many Labs were progressing to improve the process technology and also the materials properties of other Zr, Hf metal based carbides and borides. There have been significant efforts to develop UHTCs components in Europe and china as well. Recently, NASA Glen has started one major programme for continuous improvement of UHTC based fibre composites. It is obvious that the future prospective for ultra high temperature ceramics extent

a wide number of needs arising from futuristic space, defence, nuclear and thermal plants sectors.

1.1 Properties of UHTCs

Transition metal of groups IV and V (Zr, Ti, Hf, Ta, Nb etc.) based borides, carbides and nitrides were recognized as the most promising candidates for UHTC applications. These materials show very high melting point (>2500°C) due to strong covalent bonding, excellent chemical and thermal stability even at extreme environmental conditions due to high negative free energy for formation and good mechanical properties like hardness, fracture toughness, flexural strength and Young's moduli [8-10]. These ceramics stand firm against erosion and oxidation under adverse and corrosive conditions and also reveal low co-efficient of thermal expansion (CTE) which reduces thermal stress between contact surfaces of metal and ceramic and high thermal diffusivity upto elevated temperature which enhances thermal radiation into the

atmosphere over contact surfaces. The UHTCs should be light weight in itself either in monolithic or in coating form [11,12]. Some typical physical and mechanical properties of different UHTCs are tabulated in Table 1.

1.2 Application of UHTCs

The salient applications are [8, 15]:

- i. Leading edge, nose caps, vanes and engine cowl inlets of both hypersonic space and re entry vehicles
- ii. High velocity air breathing engines, e.g. scramjets
- iii. Stabilizers of reverse thrust petals and thrust diverters
- iv. Thermal protection units in defence applications
- v. Electrical heaters igniters and refractory crucible
- vi. Fibre interface coatings and electrodes

Material	Crystal structure	Density (gm/cc)	Melting temperature (°C)	Hardness (GPa)	Thermal Conductivity (W/m.K)
ZrB ₂	Hexagonal	6.09	3246	20 – 24	60 – 80
ZrC	FCC	6.59	3532	23 – 26	20 – 25
ZrN	FCC	7.09	2980	20 – 22	18 – 28
TiB ₂	Hexagonal	4.52	3225	30 – 33	22 – 28
TiC	Cubic	4.91	3067	26 – 30	50 – 60
TiN	FCC	5.39	2950	22 – 24	15 – 22
HfB ₂	Hexagonal	10.5	3250	21 – 28	60 – 72
HfC	FCC	12.67	~3900	24 – 26	19 – 24
HfN	FCC	13.9	3385	>30	17 – 14
TaB ₂	Hexagonal	12.54	3040	28	32 – 38
TaC	Cubic	14.50	3800	16.7	20 – 24
TaN	Cubic	14.30	3090	16 – 20	10 – 18
NbB ₂	Hexagonal	6.97	3050	20 – 22	20 – 28
NbC	Cubic	7.6	3500	19	28 – 34

1.3 Transition metal based borides, carbides and nitrides

Selection and design of a most appropriate aerospace component require an integrated knowledge between the aerothermal, aerodynamic, component service life and also safety requirement under adverse environment condition. In the late 1960s, the first high temperature structural ceramics has primarily set on alumina, silicon carbide and silicon nitride and the entire research and development were concerted on the development of different structural components like armors (both vehicle and body), ball bearings, cutting tools, turbine blades, crucibles etc. [16]. The recent increasing interest in high speed hypersonic vehicle indicates the requirement for advanced ultra high temperature ceramics components. There are only around 100 materials/compounds with melting/fusion points above 2500°C, including refractory metals (i.e. Mo, W, Re, Ta, Os, Hf etc), oxides (i.e. ThO₂, HfO₂, UO₂, ZrO₂ etc.) and non-oxides (i.e. ZrB₂, ZrC, ZrN, NbC, HfC, TaB₂, TiB₂ etc.) compounds [17-18]. Among them, transition metal (Gr. IV and V) based borides, carbides and nitrides have offer unusual combination of structural properties including high melting temperature and strong chemical bonds that give them both high temperature stability and outstanding mechanical properties [19-20]. We will commence by discussing these three categories.

1.3.1 Borides

Transition metal based borides normally have hexagonal crystal structure of the AlB₂ prototype where metal atom (M= Zr, Hf, Ti etc.) planes are arranged in hexagonally close-packed array and B planes are formed in sp² hybridization by stacking in an MBMBMB sequence (2D graphite-like rings). The boride structures forms very strong covalent bonding between boron-boron rings and metal-boron bonds that gives very high hardness and temperature stability. While the close packed metal layers illustrate characteristics consistent with metallic bonding that give them outstanding intrinsic thermal conductivities like Copper. This property makes boride very

attractive for rocket science where thermal stress is an important concern. The unique characteristic of the electronic nature and also the presence of free valence electron in M-M metallic bond result high electrical conductivities than other transition metal based carbides and nitrides [19-21]. The oxidation resistance of the borides are very high due to in-situ formation of B₂O₃ forming species at adverse atmosphere upto 1200°C which obstruct further oxygen diffusion to the bulk material. Furthermore, the oxidation resistance is increase after addition of SiO₂ upto 1600°C due to formation of borosilicate glass. Many researchers were reported the optimized composition of ZrB₂ system after addition of SiC (10-30 vol%).

1.3.2 Carbides

Transition metal based carbides are also used as UHTCs due to its high melting point in respect to aforementioned refractory borides. Maximum researches were concentrated on Hf and Ta based carbides due to its very high melting points (~3800°C) w.r.t other carbides. They normally have cubic close packed NaCl-type structure with a face-centred cubic B1 symmetry and have a large number of vacancies (non-stoichiometry especially in carbon site) in the lattice that tailor the both structural and electrical properties of the carbide. Unfortunately, carbides are not use normally in aerospace as the borides due to their high oxidation at relatively lower temperature, high brittle-ductile transition temperature and large spread in strength features [22]. However, for purpose where there is a sudden increase in temperature especially above 2000°C, these carbides are very much useful.

1.3.3 Nitrides

Transition metal based nitrides have almost comparable properties as carbides but for continuous high and adverse temperature applications, the loss of nitrogen and oxidation from the lattice deteriorates their both physical and structural properties [19]. Hence, the usages of transition metal based nitrides are limited in aerospace field. Moreover, transition

metal based nitrides mainly used in microelectronics and magnetic devices.

Another most recent concept demonstrated by NASA for incorporation of C-fibre in the different UHTCs materials and corresponding modelling and computational approach also reduced the parameters like processing, microstructure, properties and performance of the UHTCs. If the sizes of the components are in meter range like leading edge or nose cone, the whole components must be segmented. In that case interfaces/joints between two different segments are very significant design concern. It is also important that modelling and experimental parts are coupled together for development of a real life component.

1.4 Concluding Remarks

Ultra-high temperature ceramics are a group of materials that offer a novel set of properties like extremely high melting point, high hardness, good chemical and structural stability upto elevated temperature. UHTCs materials have been developed since the 1960s especially for R&D for aerospace applications. UHTCs are generally categorised as transition metal based (Gr. IV and V) borides, carbides and nitrides and these materials have a enormous potential in structural applications including hypersonic flight, re-entry vehicles, electrodes, cutting tools, furnace elements, crucibles and high temperature shielding. For making real life components, appropriate sintering and defect free compaction either in bulk or coating form and finally design aspect is essential. This review has covered the properties of different non-oxide based UHTCs compounds and their prospect for development of futuristic hypersonic vehicles.

Acknowledgments

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