

Spain

ECerS Refractories Symposium 2015

D. A. Jarvis

As an integral part of the European Ceramic Society (ECerS) event a Refractories Symposium was organised to rank as one of their eight main industry and technical segments under the umbrella of the main annual meeting held in Toledo, Spain, from 21–25 June 2015.

The main meeting took place in the Hotel Beatriz and was largely integrated into the overall programme but a concurrent meeting of the industrial and technical sections of the Asociación Nacional de Fabricantes de Productos Refractarios, Materiales y Servicios Afines (ANFRE) were also held in the Hotel Domenico and well attended by the Spanish based member companies some of which are also installers. There were two separate meetings one on administration and legislation and one on testing standards for materials. Information published by ANFRE indicates that in recent years, installation of refractories has been variable but there was an increase in production of refractories in Spain due to rising exports.

Introduction

As in most other developed markets the production of refractories was split between about 45 % bricks and 55 % monolithics with half of the bricks being aluminosilicate but only about one third of the monolithics being aluminosilicate.

Since its formation in 1976 ANFRE has promoted all aspects of refractories in the Iberian Peninsula. It collates and circulates industry statistics for all of the companies each year. ANFRE has also just been involved in translating a German book on refractories engineering into Catalan for sale to Spanish speaking members in the industry.

The Refractories Symposium was chaired by Sandra Abdelhouhab of BCRC Belgium assisted by Victor Mendoza of Insertec Spain, Dirk Holland of Calderys Germany and David Jarvis, refractories engineer from Great Britain. There were 30 presentations over four sessions on the Monday and Wednesday of the week long conference. The speakers were drawn from a number of countries around the world and all sessions were well attended with between forty and fifty people present at each. In addition to the presenters well known academics such as Professor Golastanifard from Iran and Professor Victor Pandolfelli from Brasil were involved and in attendance.

The call for papers requested presentations on refractories that improve the energy sav-



Fig. 1 A General view of the delegates at the Refractories Symposium

(With acknowledgement to the Spanish Ceramics and Glass Society (Sociedad Española de Cerámica y Vidrio) and the European Ceramic Society)

ings in the industry, refractories based on recycling processes to avoid environmental impacts and refractories based on local raw materials to reduce the dependency of the EU on imports. These and other interesting topics were presented to and discussed by the audience present.

Keynotes

The keynote speech in the first session on Monday 21 June was **"Process Control in the Recycling of Refractory Waste from a Steel Induction Furnace"** by P. Silva Abilio and Tessaleno C. Devezas of C-MAST, Department of Electromechan-

ical Engineering University of Beira Interior, Covilhã; Ana M Segadães, CICECO, Department of Materials and Ceramics Engineering, University of Aveiro/PT; and Rogério A. Lopes of DURITCAST SA, Travassô/PT.

Raw materials have a direct impact on all refractory products. Refractories production is expensive and requires a lot of energy and the international recommendations

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Fig. 2 Roberto Caballero of Insertec/ES presents a keynote speech which covered a review of refractories in the aluminium industry in the session chaired by Sandra Abdelouhab, EMRA
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emphasize the necessity of the reduction of the demand for primary raw materials, to alleviate the supply risks and reduces climate change impact. Consequently the use of recycled raw material is an alternative challenge to reduce the cost of refractories, import market dependency and to limit the environmental impact especially with regard to CO₂ emissions.



Fig. 4 Vanessa Halapa of Calderys Research/FR discussing anticorrosion coatings developed for refractory anchors and steel vessels
(With acknowledgement to the Spanish Ceramics and Glass Society (Sociedad Española de Cerámica y Vidrio) and the European Ceramic Society)



Fig. 3 Edward Sabolsky of West Virginia University/US presents his paper on conductive ceramics for temperature measurement in the session co-chaired by David Jarvis, consultant/GB, and Dirk Holland of Calderys/DE
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Spent refractory is often beneficiated and recycled however, the recycling of refractory heavily depends on the economic viability rather than on the recyclability. This dependency is related with the added value of the recycled raw material as a substitute for primary raw material and also depends on the parameters of the recycling process, such as milling and separation efficiency, mixture design optimization, water and additives added, sintering temperature, amongst others.

The paper described how material recovered from the dismantled refractory lining of a steel induction furnace was sorted out into

different contamination levels and characterized. After milling, different separation techniques were conducted and the particle size distributions were optimized to reproduce the material used in the original lining and others composition of self-flow castables.

The results for the mechanical, physical and thermodynamic properties of test pieces sintered at 1200, 1350, and 1500 °C respectively, were discussed. The process control of different mixture compositions was optimized to provide the best added value using such recycled materials.

In the second session, the keynote speech was **"Characterization of the Com-**



Fig. 5 ANFRE meeting delegates on the terrace of the Hotel Domenico at their Annual Meeting 2015
(With acknowledgement to ANFRE)

pressive Creep Behaviour and High Temperature Dimension Stability of Pure Silica Brick”

by Pascal Pilate, Véronique Lardot and Francis Cambier of BCRC, Belgian Ceramic Research Centre with Erwan Brochen, FGF, Forschungsgemeinschaft Feuerfest – Höhr-Grenzhausen/DE.

The main applications of “silica bricks” are in the construction of coke ovens and glass furnace crowns. These materials have a high well-defined and narrow melting range giving excellent resistance to high temperatures. Moreover, in glass furnaces, the degradation of the crown does not lead to major production disturbances because silica is fully compatible with the glass composition and does not modify it. Today, end users such as flat and container glass producers require materials with improved features such as better corrosion resistance and higher refractoriness. This is necessary to withstand the more extreme service conditions such as high temperature and



Fig. 6 The meeting of the ANFRE Technical Committee Toledo 2015
(With courtesy of ANFRE)

especially the very high alkaline vapours in the oxygen assisted combustion in modern furnaces. One solution is to use pure silica material without any lime present and having a low level of impurities normally below 0,1 %.

The purpose of the work presented was to determine the creep behaviour and the dimensional stability of pure silica at temperatures in excess of 1500 °C.

A batch of pure silica bricks was selected and characterised by their chemical and



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crystallographic compositions, density and porosity. The high temperature behaviour was determined through the refractoriness under load test and measurement of the bending strength versus temperature. The microstructure was also determined with a scanning electron microscope with EDS. Creep tests were carried out at temperatures higher than 1500 °C according to EN and ISO standards.

At low temperature, pure silica exhibits some differences such as a higher density and a lower porosity compared to "classical silica bricks". The crystallographic composition is also different. The main phase contains over 90 % cristobalite instead of the 30 – 40 % found in classical silica bricks. In addition the microstructure is more homogeneous, the material contains only one phase and pores are smaller. Because of the absence of lime and the lower level of impurities, the refractoriness is very high. The evolution of the mechanical properties such as bending strength versus time exhibits several phenomena which can be related to the low to high temperature cristobalite phase transformation. There is a strengthening at moderately high temperature and a decrease at very high temperature. The microstructure consists of large grains with prismatic cristobalite crystals radiating from the centre. Expansion versus temperature and time measurements, and creep tests at temperature ranging between 1500 – 1600 °C, demonstrate that shrinkage and expansion appear simultaneously in the material. The shapes of the resulting creep curves are due to superimposition of the two phenomena. The development with increased temperature is not very linear and there is a large scattering of results between different samples tested at the same temperature.

In the third session the keynote speech was **"Influence of Microstructural Characteristics on the Fracture Toughness of Refractories"**. Ana García-Prieto, Pilar Pena and Carmen Baudin of Instituto de Cerámica y Vidrio, CSIC, Madrid/ES with Manuel Dos Ramos-Lotito, and Delia Gutiérrez-Campos of Universidad Simón Bolívar, Department of Ciencia de los Materiales, Caracas/VE, were the authors. The objective of this work has been to establish basic relationships between the microstructure, the texture of refractories and their toughness. A series of different commercial materials were chosen in order to highlight the influence of microstructural characteristics on fracture behaviour and associated toughness. Silica, aluminosilicate and silica-alumina-zirconia based shaped refractories and a calcium aluminate cement bonded castable were chosen to study.

Extensive microstructural characterization was performed using a combination of techniques, including chemical analysis by X-ray fluorescence, X-ray diffraction, reflected light optical microscopy and scanning electron microscopy with analysis by dispersive energies. The use of three point bending tests were chosen to characterise the toughness of the various different refractories. When carefully performed using the high-performance test machines available nowadays it was possible to establish displacement controlled tests as routine lab tests for stable fracture. For the typical microstructural characteristics of the commercial refractories which were studied here, a standard size specimens of 25 mm × 25 mm × 150 mm tested with a span of 125 mm and

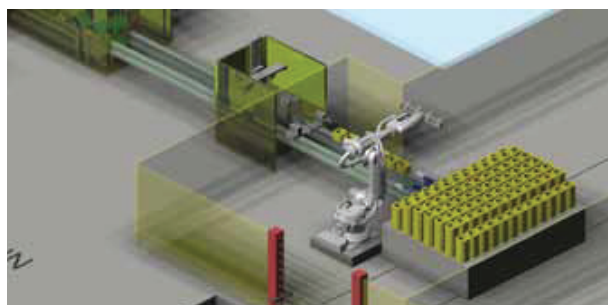
relative crack notch gave different distinct toughness values for different microstructures.

The main microstructural features that influenced the resistance of materials to the initiation of fracture are different from those that regulate crack propagation within the material. The characteristics of the aggregates used in the refractory determine the toughness to resist crack initiation while, for high values of work of fracture the presence of microstructural features which are capable to arrest cracks or at least deflect them are needed.

The fourth keynote speech was **"Refractory Solutions for Aluminium Furnaces"**, an overview by Roberto Caballero, Víctor Mendoza, and Jon Andoni Agirre of Refractory Solutions Insertec S. L. U., Bilbao/ES. The growth of aluminium applications due to its combination of beneficial mechanical properties and a relatively low density in comparison with steel, have required advances in refractories technology especially because of the many different alloys needing to be manufactured under cost-effective processes. These industrial processes involve aluminium confinement units such as both melting and holding furnaces which operate in a relatively low to medium range of temperatures from a refractory point of view. It might therefore be supposed that this would present little challenge compared to other metallurgical processing in the iron and steel industries, in which refractories play a very important role. The lecture, outlined the



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most relevant requirements for refractories, especially those in contact with molten aluminium and its many alloys. Especial attention was paid to their ability to resist high corrosion from metals, slags and salts as well as the influence of other diverse factors in the process. Current state of the art refractory solutions were discussed to cover the various types of furnace commonly used. Different laboratory tests which were designed to predict the final behaviour of refractories in service were also presented and their high degree of relevance in choosing the right correct materials for each service condition was outlined.

This was an extremely comprehensive review especially with regard to the information tabled on the underlying mechanisms of corundum formation in refractories in the aluminium industry.

This reaction is extremely complex and damaging to refractories in service and the information presented was extremely valuable in understanding and minimizing the risk.

Technical sessions

The other papers presented in the refractories symposium were equally interesting and covered a very wide range of refractories topics including as shown by the papers listed:

- Impact of Nucleation and Growth of $\text{Mg}(\text{OH})_2$ Crystals in the Development of MgO-Bonded Refractories

- Effect of Sodium on the Thermomechanical Properties and Microstructure of Calcium Aluminate Cement-Bonded Silica Containing Castables
- Influence of Zirconia Phase Transformation on the Thermomechanical Behaviour of High Zirconia Refractories
- A Novel Idea of Impact Resistant MgO-C Bricks for Steel Ladle at Rourkela Steel Plant
- New Additives System for Silica Fume Containing Castables with Enhanced Robustness to Raw Material Variations
- The Recycling of Foundry Waste Materials
- The Influence of Coking Temperature on Properties of Self-Glazing Al_2O_3 -C Components
- Porosity and Temperature Dependence of Young's Moduli of Mullite-Alumina Composite Ceramics Prepared by Starch Consolidation Casting
- The Influence of Microstructural Characteristics on the Fracture Toughness of Refractories
- Process Control in Recycling of Refractory Waste from a Steel Induction Furnace
- Calcium Zirconate – a Promising Refractory Material for Titanium-Based Melts
- Improvement of the Wedge Splitting Test for Small Refractory Specimens
- A New Binder System in Fireproofing Materials
- An Innovative Refractory Paint for the Protection of Metallic Anchors and Vessels from Alkali Corrosion
- A Study on Corrosion Mechanisms and Thermomechanical Behaviour of an Alumina-Magnesia Spinel Refractory Castable
- The Development of Drying Test for Refractory Concrete Linings
- Influence of Fibres on Refractory Concrete Properties and Their Relation with the Drying Behaviour of Refractory Concretes
- Sustainability Leads to the Globalisation of Corrosion Processes – a Case Study of Urban Waste Recycling Using a Fluidised Bed Incinerator
- Effects of Grain Size Distribution on Densification and Microstructure of Lightweight Al_2O_3 -MgO Castable Matrix Based on Close Packing Model
- Corrosion of Refractory MgO-C in an Induction furnace
- The Influence of Sintering Parameters of Aluminium Titanate (TiAl_2O_7) on the Microstructure and Thermomechanical Properties
- Wettability and Infiltration of Liquid Silicon on Several Substrates
- Characterization and Recycling of Spent Refractory Wastes in Aluminosilicate (Geopolymer) Matrixes at Low Environmental Impact
- Novel Multilayer Refractories Derived from Cast Ceramic Tapes
- Conductive Ceramic Composite Based on Magnesia Spinel.

Both the presentations and posters were very well received by the delegates attending the symposium some of whom also took part in the ANFRE meeting. The concurrent meetings therefore satisfied the most academic participants as well as those from the equally important sectors of commerce and the installation fields of the European refractories industry. There will be separate publication of most of the papers by ECeS in their own and associated journals and details may be obtained from Instituto de Cerámica y Vidrio, CSIC (E-mail: info@icv.csic.es) by those requiring more comprehensive information.

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