The 3rd International Postgraduates Seminar on Refractories

October 13th to 14th, 2020 (online)

Organized by

•Wuhan University of Science and Technology (China)
•Montanuniversitaet Leoben (Austria)
•Technische Universitaet Bergakademie Freiberg (Germany)

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•University of Science and Technology Beijing (China)
•AGH University of Science and Technology (Poland)
•University of Science and Technology Liaoning (China)
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Time schedule of the 3rd international postgraduates seminar			
Time	**Presenter**	**University**	**Presentation**
Tuesday 13th, Oct, 2020 Session A 15:00-17:40 (GMT+8)			
15:00-15:10	Welcome & Introduction		
15:10-15:30	Ilona Jastrzębska	AGH University of Science and Technology, Poland	Structure and microstructure evolution of hercynite after annealing treatment
15:30-15:50	Maciej Ludwig	AGH University of Science and Technology, Poland	Phase and microstructural characterization of recycled MgO-C aggregate and magnesia-carbon refractory bricks containing it
15:50-16:10	Ryszard Prorok	AGH University of Science and Technology, Poland	The effect of heat treatment on the properties of the basic castables with different microsilica content
16:10-16:20	Break		
16:20-16:40	Miriam Bach	TU Bergakademie Freiberg, Germany	Use of CFRP waste in carbon bonded refractories-a feasibility study on recycling
16:40-17:00	Mengke Qiao	Wuhan University of Science and Technology, China	Effect of nano zirconia on microstructure and high temperature properties of corundum castables
17:00-17:20	Sisi Zhang	Wuhan University of Science and Technology, China	M-S-H precursor synthesized by wet milling and its effects on the properties of magnesia based castables
17:20-17:40	Zheng Zhang	Université d'Orléans, France	Thermodynamical properties measurement by aerodynamic levitation
17:40-19:00	Dinner		
Tuesday 13th, Oct, 2020 Session B 19:00-21:10 (GMT+8)			
19:00-19:20	Benjamin Bock-Seefeld	TU Bergakademie Freiberg, Germany	Impact of slip- and flame-spray coated carbon-bonded alumina filters on the steel melt filtration and filter fabrication by means of additive manufacturing
19:20-19:40	Tony Wetzig	TU Bergakademie Freiberg, Germany	A new approach for filtration in continuous casting of steel
19:40-20:00	Yiran Man	University of Science and Technology Liaoning, China	Basic research on 3D printing technology and application of refractories
20:00-20:10			Break
20:10-20:30	Zhenglong Liu	Wuhan University of Science and Technology, China	In-situ grown silicon carbide whiskers onto graphite for application in Al_2O_3-C refractory
20:30-20:50	Zhe Chen	Wuhan University of Science and Technology, China	Microstructures and mechanical properties of lightweight Al_2O_3-C refractories using different carbon sources
20:50-21:10	Yang Yang	Wuhan University of Science and Technology, China	The feasibility of Cr_7C_3 as an antioxidant for magnesia carbon brick

Wednesday 14th, Oct, 2020 Session C 15:00-17:30 (GMT+8)			
15:00-15:20	Anastasia Kucheryavaya	Montanuniversität Leoben, Austria	The extent of zirconium oxycarbide solid solution range
15:20-15:40	Soheil Samadi	Montanuniversität Leoben, Austria	Determination of secondary creep stage parameters of a shaped alumina spinel refractory with the aid of genetic algorithm
15:40-16:00	Weiliang Du	Montanuniversität Leoben, Austria	Influence of aggregate shape on mechanical behavior of brittle disordered materials: a review
16:00-16:10	Break		
16:10-16:30	Zhenghuang Quan	Wuhan University of Science and Technology, China	Study on sintering behavior of activated magnesium aluminate spinel
16:30-16:50	Linchao Xu	University of Science and Technology Beijing, China	Effect of incorporation of nitrogen on calcium hexaaluminate
16:50-17:10	Xin Qi	University of Science and Technology Liaoning, China	Research progress on the application of high-silicon magnesite
17:10-17:30	Farid Asadi	Université de Limoges, France	DEM modelling of the quasi-brittle behavior of refractories by considering microcracks effect
17:30-19:00	Dinner		
Wednesday 14th, Oct, 2020 Session D 19:00-21:30 (GMT+8)			
19:00-19:20	Qingdong Hou	University of Science and Technology Liaoning, China	Lightweight design and thermal properties of magnesia-based refractories
19:20-19:40	Changkun Lei	Xi'an University of Architecture and Technology, China	One step synthesis and characterization of high aspect ratio network-like carbon nanotubes containing calcium aluminate cement composite powders
19:40-20:00	Yuchi Liu	Xi'an University of Architecture and Technology, China	The mechanism of preparation of lightweight corundum-mullite refractories by transient liquid phase method
20:00-20:10	Break		
20:10-20:30	Qiong Luo	Wuhan University of Science and Technology, China	Improve vapor corrosion resistance of in-situ $Ca_2Mg_2Al_{28}O_{46}$ ternary phase reinforced CA_6 materials
20:30-20:50	Kezhuo Li	Wuhan University of Science and Technology, China	Low-temperature catalytic preparation of SiC from polycarbosilane using iron catalyst
20:50-21:10	Hailu Wang	Wuhan University of Science and Technology, China	Preparation of novel reticulated prickly porous ceramics with mullite whiskers
21:10-21:30	Closing remarks		

Presentation:
15 minutes Oral presentation and 5 minutes Q&A

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Abstracts

Session A 15:10-15:30 (GMT+8)

Structure and microstructure evolution of hercynite after annealing treatment

Ilona Jastrzębska *AGH University of Science and Technology* E-mail: ijastrz@agh.edu.pl

Compounds with the spinel structure have lately attracted much scientific and technological attention due to their beneficial physicochemical and mechanical properties, both at ambient and elevated temperatures. Hercynite, $Fe^{2+}Al_2O_4$, is one of the spinel compounds which reveals the potential to be used as the component of refractory material which positively influences the elastic behavior of the refractory and compactness of its microstructure. Its application is interesting in point of view of its formation during the production of basic refractories from raw materials with increased content of hematite. Thus, works concerning the application of the natural features of iron-enriched raw materials are essential, especially currently, from the perspective of the global issue of the decreasing purity of the strategic raw materials.

The work presents the route to obtain high-purity hercynite ($FeAl_2O_4$) and its solid solutions, high-temperature behavior of $FeAl_2O_4$ determined by numerous experimental methods, and the mechanism of its decomposition.

Hercynite, when subjected to thermal annealing decomposes step-wisely (Fig. 1).

The first step of annealing results in a formation of $FeAl_2O_4$-Fe_3O_4 s.s., magnetite, and γ-alumina.

In the next step, solid solution separates by exsolution of magnetite and γ-alumina. Then, oxides undergo oxidation or thermal transformation to more thermodynamically stable phases. Magnetite oxidizes into maghemite, which transforms into hematite, while γ-alumina transforms into the most thermodynamically stable α-alumina.

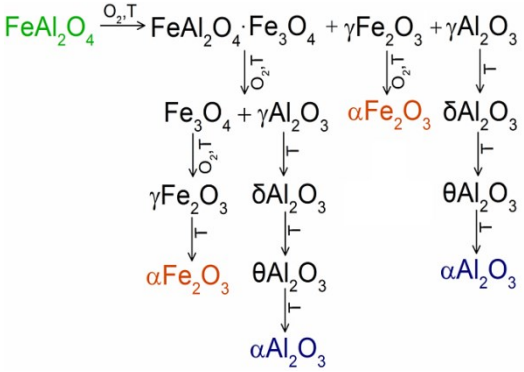

Fig. 1 Hercynite decomposition.

In conclusion, the final oxidation products of hercynite decomposition are α-alumina and hematite of non-typical scalenohedral morphology.

[1] Jastrzębska I., Szczerba J., Błachowski A., Stoch P. (2017): *Structure and microstructure evolution of hercynite spinel ($Fe^{2+}Al_2O_4$) after annealing treatment*, European Journal of Mineralogy, 29, 1, 63-72.

[2] Jastrzębska I., Szczerba J., Stoch P., Błachowski A., Ruebenbauer K., Prorok R., Śnieżek E., (2015): *Crystal structure and Mössbauer study of $FeAl_2O_4$*, Nukleonika, 60, 1, 45-47.

[3] Jastrzębska I., Szczerba J. (2019): *High temperature behavior of hercynite*, Refractories Worldforum, 1, 11.

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Session A 15:30-15:50 (GMT+8)

Phase and microstructural characterization of recycled MgO-C aggregate and magnesia-carbon refractory bricks containing it

Maciej Ludwig *AGH University of Science and Technology* E-mail: ludwig@agh.edu.pl

Refractory recycling becomes urgent issue due to the rapid increase of refractory raw materials prices, reduced raw materials deposits and regulations related to CO2 emission reduction. Magnesites and graphite which are necessary to produce MgO-C bricks for steel industry are nowadays established as critical for refractory materials market. Most of the research focused on recycling of MgO-C products while firing carbon from the material. Presented study focuses on testing and evaluation of MgO-C recycled aggregate omitting carbon removal step. This solution is more environmental, time and profit beneficial. A few different MgO-C refractory aggregates have been tested. The content of chosen aggregate in the composition of new type of MgO-C bricks was 0, 10, 20 and 30 wt.%. Samples were thermally treated at 200℃, 1000℃ and 1500℃. Phase characterization have been conducted using XRD method. Microstructural characterization was assessed using SEM method with analysis in micro areas (EDS) but also based on optical microscope observations. Obtained results indicated that MgO-C refractory aggregates were characterized by satisfactory purity. Phase composition of new magnesia-carbon bricks did not change drastically after implementation of recycled aggregate. Microstructural observation showed the differences between natural and recycled raw materials.

The research was supported by the found of the project of The National Centre for Research and Development No POIR.01.02.00-00-0175/16.

Session A 15:50-16:10 (GMT+8)

The effect of heat treatment on the properties of the basic castables with different microsilica content

Ryszard Prorok *AGH University of Science and Technology* E-mail: rprorok@agh.edu.pl

The basic castables (based on MgO) are refractory materials with large practical application, but there are still several issues that inhibit its large scale use. The major problem is the ability of the basic oxides to hydration. This problem can be avoid/reduce by use agents that can influence on hydration process like microsilica or chelating compounds. In result there is obtain bonding system that entrap a large amount of water. During heat treatment this water is released and take part in shaping properties of the basic castables. The aim of this work was to evaluate base properties of the castables with different microsilica content after different temperatures of the heat treatment. Samples of the castables were tested on cold crushing strentgth, cold modulus of rupture, Young's modulus, apparent density and open porosity. The phase composition as well as microstructure were also evaluated. It has been shown that the amount of microsilica strongly influence on castables properties. The temperature of the heat treatment also changes the properties of the castables but the scale of this effect depends on content of microsilica.

This work was partially financially supported by the statutory funds of the Faculty of Materials Science and Ceramics, AGH Kraków number 16.16.160.557.

Session A 16:20-16:40 (GMT+8)

Use of CFRP waste in carbon bonded refractories-a feasibility study on recycling

Miriam Bach *TU Bergakademie Freiberg, Germany* E-mail: Miriam.Bach@ikgb.tu-freiberg.de

The increasing use of carbon fiber reinforced polymer (CFRP) composites in recent times has resulted in a vast amount of recyclable material being available today, e.g. from end-of life components such as aircraft or wind turbines. Recycling approaches for carbon fiber composites are currently being investigated with the aim of preserving the high-quality properties of the original composite material. However, this study focuses on the reuse

of CFRP in carbon-bonded refractories. For this purpose, suitable treatment procedures were established in order to investigate the influence of CFRP size and shape, adherent polymer matrix and replacing graphite as raw material to physical, mechanical and thermo-mechanical properties of the refractories. Thus, the potential of CFRP composites used in refractory materials could bring economic and environmental benefits to the refractory, carbon source and carbon fiber composite industries in the near future.

Session A 16:40-17:00 (GMT+8)

Effect of nano zirconia on microstructure and high temperature properties of corundum castables

Mengke Qiao *Wuhan University of Science and Technology, China* E-mail: 2551507503@qq.com

The effects of nano zirconia addition on the microstructure and properties of corundum castables were studied by using corundum, kyanite, alumina powder, gel powder and nano-ZrO_2 as raw materials, FS10 as water reducer and citric acid as retarder. The results show that the addition of 1.2(wt)% nano-ZrO_2 which heated at 110℃ could reduce the median pore diameter (D50) from 480 nm to 200 nm. After heated at 1100℃, the cold modulus of rupture (CMOR) of the samples increased by 29.95% to 24.71 MPa and the cold compressive strength (CCS) increased by 50.32% to 116.37 MPa. The hot modulus of rupture (HMOR) increased by 114.95% to 3.59 MPa after being treated at 1500℃. The high surface energy of nano-ZrO_2 can increase the interface energy and bonding strength between adjacent particles.

Session A 17:00-17:20 (GMT+8)

M-S-H precursor synthesized by wet milling and its effects on the properties of magnesia based castables

Sisi Zhang *Wuhan University of Science and Technology, China* E-mail: 1220460413@qq.com

Magnesia castables have been widely used in tundish and converter attributing to its high refractoriness and simultaneous purification effect of molten steel. The main binder in magnesia castable is MgO-SiO2-H2O system which effectively solve the problem of crack, spalling or explosion brought by formation of Mg(OH)2 during drying step. However, high content of microsilica(usually 6 wt.%) will adversely affect high quality steel and lead to a depression on high temperature properties of magnesia castable. Our work aimed to reduce the content of SiO2 in magnesia castable without sacrificing its properties. Hence, the M-S-H (MgO-SiO2-H2O) precursor was synthesized via wet milling process, which stimulated the dissolution of Mg^{2+} and $HSiO^{3-}$, thus promoted the formation of M-S-H in magnesium castable and lead to a better bonding effect. Consequently, the workablility and mechanical properties of magnesia castable at both room and high temperatures got effectively enhanced. By this way, the castable with only 2 wt.% microsilica can satisfy the requirements of early strength, and mechanical properties after high temperature treatment. Additionally, the HMOR of the newly designed castables were obviously improved.

Session A 17:20-17:40 (GMT+8)

Thermodynamical properties measurement by aerodynamic levitation

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Calcium aluminate melts are critical systems in the field of refractories and cement, their thermophysical properties are of great interest in understanding their physical and transport behavior. Owing to heterogeneous nucleation at the melt-crucible interface, it is difficult to obtain accurate physical properties of melts using conventional measurement. To avoid the heterogeneous nucleation, the melts with 50wt%CaO-50wt%Al_2O_3 were heated by the laser-heated aerodynamic levitation furnace to measure the density, viscosity and surface tension in the temperature range of 1600 to 2300℃. With increasing the temperature, the density, surface tension and viscosity decreased from 2.75 to 2.58g/cm^3, from 0.72 to 0.58 N/m, from 310 to 19.3 mPa·s, respectively. But as the

temperature rose further, their decreasing trend became gradually flattened.

Session B 19:00-19:20 (GMT+8)

Impact of slip- and flame-spray coated carbon-bonded alumina filters on the steel melt filtration and filter fabrication by means of additive manufacturing

Benjamin Bock-Seefeld *TU Bergakademie Freiberg, Germany* E-mail: benjamin.bock@ikgb.tu-freiberg.de

Solid, non-metallic inclusions considerably impair the mechanical properties of casted steel products. In order to remove these inclusions from the steel melt, carbon-bonded alumina filters have been developed, which interact with molten steel. As a result of this interactions, inclusions are entrapped in an in-situ formed structure on the filter surface and are removed from the steel melt. A further opportunity for the steel melt purification consists in the application of a dense flame sprayed coating, which exhibits the same chemical composition as the inclusions and leads the deposition of inclusions on the filter surface due to attraction forces.

In order to evaluate the steel melt purification mechanisms, carbon-bonded alumina filters with and without a flame sprayed alumina coating were immersed in molten steel and analyzed with regard to the structural changes of the filter surface. Furthermore, the fabrication of additive manufactured filters was examined, which should enable the production of reproducible filter geometries with improved mechanical properties and higher steel melt purification rates.

The results revealed that the application of ceramic filters led to a significant deposition rate of inclusions on the filter surfaces. Moreover, the additive manufactured filters provided a great potential for the steel melt filtration.

Session B 19:20-19:40 (GMT+8)

A new approach for filtration in continuous casting of steel

Tony Wetzig *TU Bergakademie Freiberg, Germany* E-mail: tony.wetzig@ikgb.tu-freiberg.de

Ceramic foam filters, which are applied in metallurgical and foundry operations to improve the purity of cast metal melts, are traditionally manufactured by the replicating polymer foam templates. Since the first introduction of the replication technology in the 1960’s, billions of filters were produced and utilized to enhance the performance of metal products. To adapt the technique for the continuous steel casting, the filter material and manufacturing technique have to be tailored to withstand the severe conditions and to ensure component reliability over a long casting duration. In the present study, a combined approach using centrifugation, dip and spray coatings was investigated to produce large-scale carbon-bonded alumina filters with special geometry. The filigree filter structures survived the immersion in an industrial tundish for 45 minutes, despite exposure to thermal shock, corrosive slags, constant melt flow and buoyancy at high temperatures. The special design of the filter material combines the thermal shock resistance of carbon with the corrosion resistance of alumina. Clogging, which is considered harmful for carbon-bonded submerged entry nozzles, helped in the case of the filtration by protecting the core material from further decarburizaion while reinforcing the structure mechanically and removing harmful inclusions and dissolved elements from the melt.

Session B 19:40-20:00 (GMT+8)

Basic research on 3D printing technology and application of refractories

Yiran Man *University of Science and Technology Liaoning, China* E-mail: ymmanyiran@163.com

Refractories with grid structures have been widely used in molten metal filtration and high temperature flue gas filtration. However, the existing molding methods have some insufficiencies, such as complex molding process, long time-consuming, uneven pore size and distribution. At the same time, the properties of the products need to be improved (e.g. Filtration results, corrosion resistance and thermal shock resistance). The advantage of 3D printing

technology is that it can produce complex grid structures. The emergence and development of 3D printing technology provides a new idea for the preparation of refractories with grid structures. In order to prepare high-performance refractories with grid structures with low cost and high efficiency mullite high-temperature flue gas filter with high thermal shock resistance and high corrosion resistance magnesia alumina spinel molten metal filter prepared by 3D printing technology was investigated, respectively. In the work, effects of composition of raw materials, type and quantity of additives, firing process and grid type on mechanical and thermal properties were studied. The printing effect, drying effect, linear shrinkage, bulk density and porosity, phase composition, microstructure, thermal shock resistance, corrosion resistance and mechanical strength were characterized, respectively. The regular pattern of preparing refractories by 3D printing was also discussed.

Session B 20:10-20:30 (GMT+8)

In-situ grown silicon carbide whiskers onto graphite for application in Al_2O_3-C refractory

Zhenglong Liu *Wuhan University of Science and Technology, China* E-mail: lzl2016@wust.edu.cn

To get highly clean steel, low carbon materials become more important in the field of metallurgy. But as the graphite content decreases, the thermal properties of the carbon containing refractories will inevitably deteriorate. For these reasons, there is an urgent need to find a method that can improve the performance of carbon containing refractories, while also reducing the graphite content. In our work, SiCw@graphite composite powders were prepared by salt-assisted synthesis from Si powders, graphite, and a molten salt medium (NaCl and NaF). The effect of in situ generation of silicon carbide whiskers coating on the wettability and oxidation resistance of graphite was investigated, and first time add to Al2O3-SiC-C refractory castables products. The presence of the silicon carbide whiskers coating significantly improves the wettability of the graphite, while preventing the rapid oxidation of the graphite. Hence, the oxidation resistance and the slag corrosion resistance of the castables material is significantly improved against. Meanwhile, the strength of the castables material is significantly improved due to the formation of ceramic bond between the whisker and the matrix and aggregate.

Session B 20:30-20:50 (GMT+8)

Microstructures and mechanical properties of lightweight Al_2O_3-C refractories using different carbon sources

Zhe Chen *Wuhan University of Science and Technology, China* E-mail: chenzhe2019@wust.edu.cn

Al_2O_3-C refractories containing dense corundum aggregates have been widely used to control the flow of molten steel during continuous casting. We previously proposed an original strategy to strengthen Al_2O_3-C refractories by using microporous corundum aggregates instead of dense corundum aggregates. In present study, two lightweight Al_2O_3-C refractories containing microporous corundum aggregates were fabricated by using flake graphite and microcrystalline graphite as the carbon source, respectively. The effect of different carbon sources on the microstructures and mechanical properties of lightweight Al_2O_3-C refractories with silicon additive was investigated. Compared with the specimens using flake graphite, more SiC whiskers formed in the microporous corundum aggregates in the specimens using microcrystalline graphite, because microcrystalline graphite had a higher reaction activity than that of flake graphite. It resulted in a more intertwined and compact microporous aggregate/matrix interface structure reinforced by SiC whiskers. Due to a better microporous aggregate/matrix interface together with the SiC whiskers, the crack propagation along the aggregate/matrix interface was suppressed, whereas the percentage of cracks propagating within the aggregates was enhanced. Therefore, compared with that using flake graphite with a higher price, the lightweight Al_2O_3-C refractories using microcrystalline graphite had a higher strength, a higher fracture energy and a somewhat higher toughness.

Session B 20:50-21:10 (GMT+8)

The feasibility of Cr_7C_3 as an antioxidant for magnesia carbon brick

Yang Yang *Wuhan University of Science and Technology, China* E-mail: wustwind@163.com

Magnesia carbon (MgO-C) refractory is commonly used in the steelmaking system. The presence of graphite brings improvements in the thermal shock resistance and slag corrosion resistance of the refractory. However, the oxidation of graphite carbon in a MgO-C brick usually leads to the deterioration of material properties.At present, a lot of researches have been done to improve the oxidation resistance of magnesia carbon bricks. Common antioxidants are metal powders, carbides, and borides. The metal carbide Cr_7C_3, as a by-products of aluminum chromium slag recycling smelting, might also have the possibility to be an antioxidant for magnesia carbon brick. In this report, we will analyze the feasibility of Cr_7C_3 as an antioxidant from its effect on the oxidation resistance, physical and mechanical properties as well as slag resistance of magnesia carbon brick.

Session C 15:00-15:20 (GMT+8)

The extent of zirconium oxycarbide solid solution range

Anastasia Kucheryavaya *Montanuniversität Leoben, Austria* E-mail: anastasia.kucheryavaya@unileoben.ac.at

Zirconium oxycarbides have been investigated since decades due to their good refractoriness, physical properties, and corrosion resistance. Variety of ZrOC compounds were reported, which in turn makes chemical composition determination a crucial step in the ZrOC materials characterization. Solid solution range is traditionally represented with the formula ZrOxCy, where $x+y \leq 1$. New methods of analyses as well as improved synthesis procedures allowed to provide new perspective towards the solid solution range. Different methods were traditionally applied for obtaining chemical composition of these materials, including combustion of carbon, elemental analyses of C and O, assignment of chemical composition to cell parameter a calculated from X-Ray Diffraction (XRD) patterns. These procedures include assumptions and calculations, influencing the results. Scanning electron microscopy (SEM) in combination with Energy dispersive X-ray spectroscopy (EDS), Transmission electron microscopy (TEM) investigations with EDS or electron energy loss spectroscopy (EELS) have been applied. This work includes and interprets obtained results of XRD patters, SEM figures and EDS spot analyses.

Session C 15:20-15:40 (GMT+8)

Determination of secondary creep stage parameters of a shaped alumina spinel refractory with the aid of genetic algorithm

Soheil Samadi *Montanuniversität Leoben, Austria* E-mail: Soheil.samadi@unileoben.ac.at

Creep is one of the major causes of irreversible strains in refractory linings at high temperatures. The corresponding Norton-Bailey creep parameters of ordinary refractory ceramics can be inversely identified with the Levenberg-Marquardt algorithm. Nevertheless, in most cases, the experimental creep curves are rather fluctuant and diverse. For some cases, the inverse identification could fall into a local minimum if a good initial guess of creep parameters fails. In the current presentation, the genetic algorithm is introduced to overcome the local minima of inverse identification. A shaped alumina spinel refractory was used to manifest the benefits and disadvantages of the combination of the genetic algorithm and the Levenberg-Marquardt algorithm.

Session C 15:40-16:00 (GMT+8)

Influence of aggregate shape on mechanical behavior of brittle disordered materials: a review

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Refractories and concretes are often composed of aggregates and fines. The shape of aggregates is diverse for

different raw materials. Experimental researches reveal that it plays a role in the mechanical behavior of refractories. For finite element and discrete element mechanical modelling, it is necessary to consider aggregate shape explicitly. This presentation briefly reviews the state of the art of numerical and experimental researches of aggregate shape impact on mechanical behavior of brittle disordered materials.

Session C 16:10-16:30 (GMT+8)

Study on sintering behavior of activated magnesium aluminate spinel

Zhenghuang Quan *Wuhan University of Science and Technology, China* E-mail: qzh2270663905@163.com

Magnesium aluminate spinel ($MgAl_2O_4$) which have a series of great properties is a promising candidate for high-temperature applications. How to use lower energy consumption to prepare spinel products with better quality is still the most urgent problem to be solved in current industrial production.

At present, solid-phase sintering is still the most widely used method for mass production of alumina-magnesia spinel in industry, which is divided into two main methods of one-step sintering and two-step sintering. One-step sintering will cause 5%-8% volume expansion due to the spinel reaction in the process of sintering, resulting in lower density of materials. However, the two-step sintering eliminates the influence of volume expansion effectively by the chemical reaction of activated spinel firstly and crushing, repressing the activated spinel in the secondary sintering process, thus obtaining spinel materials with higher density. Due to incomplete spinelization reaction, activated magnesium aluminate spinel has the characteristics of small grain size, large specific surface area and so on, shows certain sintering activity during secondary sintering process. Therefore, it is necessary to study sintering behavior and the influencing factors of activated spinel.

Session C 16:30-16:50 (GMT+8)

Effect of incorporation of nitrogen on calcium hexaaluminate

Linchao Xu *University of Science and Technology Beijing, China* E-mail: xlc1250824875@163.com

CA_6 has the characteristic of preferentially forming platelet or plate-like grains，the resulting relatively porous microstructures lead to the infiltration of slag into the refractory matrix easily which limits the application of CA_6 in metallurgical and petrochemical to a large extent. Ions doping is regarded as an efficient approach to improve the density of calcium hexaaluminate (CA_6). In this study, N-doped CA_6 has been successfully synthesized by pressureless sintering under N_2 atmosphere at 1750°C using CaO, Al_2O_3 and Al fine powder as the raw materials. The reasonable doping amount and the promotion mechanism of N^{3-} on the thickness of CA_6 along c axis were also discussed by means of first-principle calculation and Rietveld refining analysis.The results show that the structure formula of the N-doped CA_6 is $CaAl_{12}O_{18.58}N_{0.28}$ and it is preferential for N^{3-} to substitute O(3) ions near Al(2) in the spinel block. Such a substitution occurs in the Al-O (Al2-O3) bond parallel to the c-axis, and the bond lengths for tetrahedral Al-N bonds are larger than those of Al-O bonds, showing a promising potential of N^{3-} for increasing the crystallization thickness of CA_6 along c axis.

Session C 16:50-17:10 (GMT+8)

Research progress on the application of high-silicon magnesite

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Magnesite is one of superior mineral resources in China. It has been widely used in metallurgy, building materials, chemical industries etc. High-silicon magnesite is one of low-grade magnesite with high content of SiO_2. High-quality magnesia products can be prepared from high-silicon magnesite by flotation technology and thermal separation technology. By direct calcination of high-silicon magnesite with reasonable ingredients, high value-added refractory materials such as magnesia silica sand, forsterite and composite powder materials can be

prepared. Important raw materials such as building materials and chemicals can also be prepared from high-silicon magnesite. Hence high-silicon magnesite has high economic and scientific research value. In the presentation, the purification processes of high-silicon magnesite at home and abroad were reviewed, the technical indicators and purification principles were expounded, and finally the comprehensive utilization methods of high-silicon magnesite were introduced.

Session C 17:10-17:30 (GMT+8)

DEM modelling of the quasi-brittle behavior of refractories by considering microcracks effect

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This study is a part of ATHOR project, which is focusing on the numerical simulation of refractory ceramics by considering their microstructure and its influence on the fracture mechanics. The objective of this study is to use the Discrete Element Method (DEM) for simulating the fracture mechanisms in refractory ceramics that exhibit brittle behaviour and crack branching. Fracture propagation requires a large number of discontinuities and Finite Element Method (FEM) is not easily capable of managing this kind of microstructural effects. Using DEM to model fracture mechanisms in a pseudo-continuum media, especially in the ceramics field, is a new and promising approach under active development. This study focuses on the randomisation of local fracture criteria in the virtual sample using Weibull distribution. This approach can model the microcracks and quasi-brittle behaviour of the sample under direct tensile tests, cyclic loadings and wedge splitting test. As a perspective, the proposed approach promotes a new numerical way that may be used for designing and simulating an optimal microstructure in order to reduce brittleness in refractory ceramics.

Session D 19:00-19:20 (GMT+8)

Lightweight design and thermal properties of magnesia-based refractories

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In recent years, there was a huge demand for basic lightweight insulation materials in the metallurgical industry, especially magnesia based refractories with low thermal conductivity. Therefore, the lightweight magnesia based refractory products were prepared through the synthesis of lightweight aggregate and the different grain compositions. The microstructure (pore size distribution and phase composition), thermodynamics (coefficient of thermal expansion and thermal conductivity) and sintering properties of the lightweight magnesia based refractory products sintered at 1600℃ were characterized. The original salt pseudomorph produced by the thermal decomposition of magnesite fine powder (porogenic agent) provides a uniform porous structure for the synthesis of lightweight aggregates. The porous morphology of the periclase phase was controlled by adjusting the content of porogenic agent. The average pore size of lightweight magnesia based refractory ranged from 1.5 μm to 4.2 μm, and the apparent porosity increased from 29.88% to 32.46%. In the same time, the thermal conductivity increased from 0.037 W/(m·K) to 0.217 W/(m·K), indicating that the introduction of homologous porogenic agents could produce lightweight alkaline refractories with high porosity and low thermal conductivity.

Session D 19:20-19:40 (GMT+8)

One step synthesis and characterization of high aspect ratio network-like carbon nanotubes containing calcium aluminate cement composite powders

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The poor water dispersion and oxidation resistance of carbon has restricted the widely industrial application of carbon containing castables. High aspect ratio network-like carbon nanotubes containing calcium aluminate cement composite powders were synthesized by carbon-bed sintering method using calcium acrylate and activated alumina

as raw materials, and nickel nitrate as catalyst. Meanwhile the effects of catalyst content on the structure and morphology of carbon nanotubes were also studied. The synthesized products were characterized by X-ray diffraction, field-emission scanning electron microscopy, high-resolution transmission electron microscopy and Raman spectroscopy. The results indicated that the phase compositions of composite powders were similar to that of commercial Secar71. The length of carbon nanotubes was ca. 25 um, and the diameter of carbon nanotubes was 55 nm. The aspect ratio of carbon nanotubes is ca. 450. The formation process and mechanism of carbon nanotubes was studied by thermogravimetric analysis coupled with Fourier transform infrared spectroscopy. The growth temperature of carbon nanotubes was 700℃, and the growth of carbon nanotubes agrees with vapour-solid (V-S) model. In addition, the thickness of floating layer, floating ratio and oxidation ratios of the synthesized product were lower than those of the same carbon containing Secar71 cement (S71CB) composite powders, implying that the water dispersion and oxidation resistance of carbon nanotubes were improved because the high aspect ratio network-like carbon nanotubes were not easily separated from calcium aluminate cement and the carbon nanotubes were encased in calcium aluminate cement.

Session D 19:40-20:00 (GMT+8)

The mechanism of preparation of lightweight corundum-mullite refractories by transient liquid phase method

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The lightweight corundum-mullite refractory, which can be directly used for the working lining of the transition zone, with high refractoriness, high compressive strength, and low thermal conductivity has been manufactured by utilizing the phenomenon of transient liquid phase diffusion in the sintering process. The mechanism of the transient liquid phase diffusion method at high temperature has been illuminated through the analysis of thermodynamic simulation software results and the variation of the samples' microstructure. The effects of quartz particles on the mechanical and thermodynamic properties of the sintered samples have been studied by comparing the results of X-ray diffraction, microstructure, refractoriness under load, and the thermal conductivity. It is worth to notice that the prepared lightweight corundum-mullite refractory gains a low volume density, low thermal conductivity, high compressive strength, and high refractoriness under load with controlling the quartz particles' granularity, addition, and the sintering temperature.

Session D 20:10-20:30 (GMT+8)

Improve vapor corrosion resistance of in-situ $Ca_2Mg_2Al_{28}O_{46}$ ternary phase reinforced CA_6 materials

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Coal catalytic gasification technology is one of the most effective process ways for producing SNG. The working conditions of the coal catalytic gasification furnace require the refractory lining to be stable under high-temperature reducing atmosphere, and have excellent resistance to alkali vapor and water vapor erosion. This research introduces the improvement of the structural strength of CA_6 materials by the bridging structure of plate-like $Ca_2Mg_2Al_{28}O_{46}$ formed in the matrix. The optimized pore structure increases the permeation resistance of vapor, good bonding of aggregate-matrix and high structural strength inhibit the formation of continuous cracks during high temperature vapor erosion, exhibits excellent resistance to alkali vapor and water vapor erosion. The continuous scouring of high-temperature water vapor causes the precipitation of SiO_2 in the silicon-rich liquid phase, then dissolves in water vapor and produces volatile gas phase, resulting in the special microscopic features of concave holes and terraced stripes appear on the surface of some grains.

Session D 20:30-20:50 (GMT+8)

Low-temperature catalytic preparation of SiC from polycarbosilane using iron catalyst

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Owing to its advantages of high thermal conductivity, low thermal expansion coefficient, high corrosion resistance and radiation resistance, silicon carbide (SiC) was widely used in the fields of machinery, aerospace and refractories. At present, the commonly used methods to prepare SiC include carbothermal reduction, sol-gel, arc discharge and precursor pyrolyzed method, among which, the last one attracts a lot of attention; and SiC with simple preparation process, low synthesis temperature and high product quality can be prepared via the method. In addition, it is well known that the introduction of catalysts can reduce the activation energy of chemical reactions so that the reactions can proceed smoothly at lower temperatures. However, the research on the preparation of SiC powder via catalytic precursor pyrolysis of polycarbosilane is not investigated detailly at present.

Thus, this study prepares SiC powder through the precursor pyrolyzed method using iron as the catalyst and polycarbosilane as the precursor, the effects of pyrolysis temperature, content of iron catalyst and holding time on the formation of SiC were studied. The results showed that the synthesis temperature of SiC could be reduced from 1273 K to 1023 K by adding 2.00 % iron catalyst, and the yield of SiC increased from 66 % to 80 % at 1123 K.

Session D 20:50-21:10 (GMT+8)

Preparation of novel reticulated prickly porous ceramics with mullite whiskers

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Porous materials are widely used in heat exchangers, sewage treatment, electromagnetic shielding, thermal insulation, gas adsorption, photocatalytic due to their high specific surface area. The specific surface area of materials plays a pivotal role in them. It can be enhanced by increasing the porosity of the material, but the cost of this improvement is reducing the strength of the material. In order to improve performance, it is necessary to increase its surface area without reducing the strength of the material. In this work, mullite porous ceramics with mullite whisker on the inside and outside surfaces structures, which known as prickly porous ceramics(PPCs). They were fabricated using polyurethane foam coated with slurries as the pore-forming agents, sintered after secondary impregnation with silica sol and ammonium fluoroaluminate. The the sintering temperature as well as slurry composition of secondary impregnation were tuned to tailor the strength and surface structures of the PPCs. In addition, the potential of PPCs as high-temperature catalyst supports was demonstrated. Overall, the PPCs demonstrated large surface areas and high mechanical strength. This study paves the way for the fabrication of high-performance porous ceramics.

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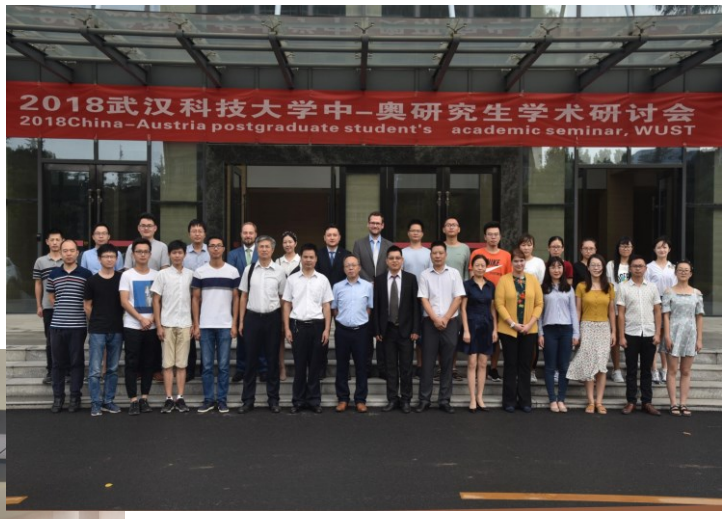

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