Addition of ceramics materials to improve the corrosion resistance of alumina refractories

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Abstract

This review shows that adding ceramic materials such as ZrO_2 , $ZrSiO_4$, TiO_2 , SiO_2 , Cr_2O_3 , etc., to a base alumina refractory clay, improves the corrosion resistance under the presence of acid (H_2SO_4 , HCl , HNO_3 , etc) and/or alkaline ($NaOH$, KOH , Na_2O , $Ca(OH)_2$, etc) solutions. Characterization techniques, such as SEM, XRD, ICP-MS analysis, and weight loss show individual or correlated properties between apparent porosity, bulk density, mass loss, total number of eluted ions from alumina refractories, and the degree of corrosion resistance obtained by sintering. Static and dynamic (ASTM C-874) slag cup tests are performed by immersion or exposure to a continuous flow stream in acid and/or alkaline solutions. Results show that adding 81.6 mol% Cr_2O_3 , 4 mol% TiO_2 to the Al_2O_3 base reduces corrosion damage depth by SiO_2 - CaO - Al_2O_3 molten oxide. Sintered mullite matrix increases its corrosion resistance against alkaline vapors by adding 16% of $ZrSiO_4$. SiO_2 - Al_2O_3 refractory ceramics with mixtures of 67–23, 56–38, 25–71%, respectively, have 98% resistance against acid solutions. However, their resistance to alkaline solutions is 58% with 23% alumina and rises up to 88% with 71% of alumina. It is concluded that higher content of *corundum* (α - Al_2O_3) and mullite phases on refractory ceramic improves its resistance to acid and alkaline solutions and that the attack of degrading solution is preferential over impurities in the phases present near the grain boundaries.

Keywords Refractory ceramics · Acid and alkaline resistance · Mullite · Corundum

1 Introduction

The industry (energy or petrochemical) has the need to incinerate corrosive gases product of waste, which cannot be discharged to the atmosphere for ecological reasons. One of the major challenges of the industry is to increase the corrosion resistance of refractory materials and to improve the durability of combustion chambers in power plants used for waste incineration.

By increasing corrosion resistance of refractory materials, the durability of combustion chambers in energy plants used for waste incineration is improved. Ceramic materials such as Al_2O_3 have been used as refractories in

combustion chambers, despite the severe corrosion that occurs by fusing gases. Al_2O_3 is always the first option as a refractory because it is less expensive, easy to handle and produce. Latest developments in non-oxide refractory ceramics are SiC y MgO - C (*magnesia-carbon refractory*), but these materials cannot be used in highly oxidizing atmospheres, including combustion chambers, due to poor thermal shock resistance of MgO . Although Cr_2O_3 may be the first candidate as a compound for Al_2O_3 , diffusion rate of the Cr ion is smaller than any other metal ions; however, pure Cr_2O_3 reacts with the basicity of the slag, expelling toxic Cr^{6+} into the sintered body or slag [1].

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Refractory materials in operation are subjected to three types of loads: thermal, mechanical, and chemical. Chemical resistance of ceramics is defined as the ability to withstand destructive action in an aggressive environment. This resistance depends on the properties of the aggressive medium, chemical composition and structure of the ceramic, and the conditions of the corrosion process, especially the point of contact between the ceramic and the aggressive medium [2]. The life cycle of a refractory material is usually determined by the degree of corrosion it can withstand [3]. Corrosion may occur by a gas (inert or chemically active) or by a liquid (acid solutions, bases, salts, molten salts, glass, slag, metals, fresh or sea water, etc) [2].

A lot of scientific papers have been published on corrosion in alumina under several conditions, where corrosion behavior of highly pure alumina at room temperature for periods of 10 days, in a solution of H_2SO_4 at different concentrations (2 wt%, 10 wt% and 20 wt%), shows that impurities (MgO, CaO, SiO, Na_2O , etc.) present in the grain boundaries are the first cause of corrosion in high-purity alumina ceramics [4, 5].

Multiple processes contribute to the corrosion of refractory materials, but these processes are always based on the physicochemical properties of the corrosive agent and on the intrinsic properties of the refractory materials, such as interconnected porosity [6] and the presence of mullite phase $3Al_2O_3 \cdot 2SiO_2$ [7]. A high interconnected porosity allows the access of external agents and increases the surface of reaction, which favors material interaction and corrosion. Also, refractories are multiphase materials that enable preferential or selective attack [6].

For example, *mullite phase* [7] is the only stable crystalline phase in the aluminosilicate system under normal atmospheric pressure. The high levels of functional properties of aluminosilicate ceramics depend on the general content of *mullite* on its structural and morphological states [7], including resistance to acid and alkaline solutions [5]. Therefore, the industrial methods for producing aluminosilicate ceramics are based on the highest formations of *mullite* quantity. This is achieved by using the refractory clays with high amount of SiO_2 and alumina Al_2O_3 and SiO_2 [7].

2 Materials and methods

Eight of the most abundant elements in Earth are presented in Table 1. Silicon is never found in its elemental form, but in form of silicate minerals, and their structures

depend on the composition and formation conditions of the place where they are found. They can be contained in relatively pure mineral deposits or in deposits made up of one or more mineral species [8].

It is very common for aluminosilicates to be referred with the term “clay,” but dozens of minerals fall under the classification of clay, and a single clay deposit may contain a variety of individual clay minerals along with impurities. Therefore, clay minerals are classified as phyllosilicates, due to their layered structure. The most common clay mineral is kaolinite $Al_2Si_2O_5(OH)_4$, although others such as montmorillonite $(NaCa)_{0.33}(Al,Mg)_2Si_4O_{10}(OH)_2 \cdot nH_2O$, and vermiculite $[(Si_{3.04}Al_{0.92}Ti_{0.04})(Al_{0.11}Fe_{0.35}^{3+}Fe_{0.07}^{2+}Mg_{2.41}Mn_{0.003})O_{10}(OH)_2]Ca_{0.21}K_{0.05}Na_{0.10}$ are also abundant [8].

The type and proportion of clay minerals in individual sediments are related to sediments of source rocks, weather conditions, and transport mechanisms that occur in the material according to Biscaye [9], Liangbiao and Liu [10]. Also in the study by Kurkura Kabeto [11], sediments of the clay show the presence of kaolinite as main mineral, over a base of kaolin and other minerals associated with clay. These can be divided in to four types: kaolinite, microcline $KAlSi_3O_8$, kaolinite $3Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$, muscovite $KAl_2Si_3AlO_{10}(OH)_2$ –microcline $KAlSi_3O_8$ –kaolinite $3Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$. However, in their study, clays show other associated minerals such as SiO_2 quartz, vermiculite $Mg_{0.7}(Mg,Fe,Al)_6(Si,Al)_8O_{20}(OH)_4 \cdot 8H_2O$, albite $NaSi_3AlO_8$, calcite $CaCO_3$ and calcite magnesite in less quantity [11].

Clay minerals from different regions are added in certain compositions to formulate corrosion resistant alumina refractories. Mineral extraction is carried out in open sky. It is subsequently cracked to homogenization (ASTM C-702) and dried at 110 °C for up to 24 h. [12, 13]. Samples are reduced to microns (40–74 μm), using in some cases mixtures of different grain sizes [3, 7, 14] and using ball mills to produce superficial and structural changes to the solid material [15]. The ball and load ratio for ball mills varies between 10, 15 and 20–1, respectively, with rotation speeds between 250 and 480 rpm [16, 17]. They were sieved to obtain homogeneous particle size (ASTM C-136). Samples that are subjected to corrosion tests are formed into cylinders [3, 13], rectangles [14] or squares, mostly with variable dimensions by cold isostatic compaction using a hydraulic press at 10.12, 22, 40, 50, 75 and even 150 Mpa [3].

Table 1 Chemical composition of crust of Earth [8]

Element	O	Si	Al	Fe	Ca	Na	K	Mg	Others
Percent by weight	50	26	7.5	4.7	3.4	2.6	2.4	1.9	1.5

They are sintered in an oven at heating rates of 2–10 °C/min [7] and temperatures of 1600–1700 °C. The soaking times are ranging from a few hours up to few days, depending on the composition of the mixture and phases to be obtained [3]. The cooling is carried out inside the oven off. Static [18] or dynamic [13, 19] testing for corrosion resistance is made by chemical reaction, simulating conditions of a real application, at certain temperature, atmospheric pressure or vacuum, and with acid or alkaline substances, through methods such as cup slag test, drip slag test (ASTM C-768), gradient slag test, rotary slag test (ASTM C-874) and dip and spin test.

3 Result and discussion

A diffusion of ions (atoms) is common between a ceramic material and the aggressive medium. But it is not necessary to consider this interaction as destruction and subsequent loss of ceramic material. In ceramic corrosion, there are two main types of chemical resistance, which are acid and alkaline. Table 2 shows chemical resistance for alumina oxide, through mass loss under different reactive media [2].

Booklet of the Friedrichsfeld Company (Germany) reported the mass loss (Table 3) to measure chemical resistance of ceramics with 96.0 and 99.5% of Al_2O_3 subjected to several acid and alkaline media [2].

In a ceramic mixture of silica and alumina, corrosion resistance is higher under acidic than alkaline media, and mass loss under acidic is lower than alkaline medium, Table 4.

Long Huang, Luo and Li [20] studied Al_2O_3 alumina ceramics under acid and alkaline corrosion, sieved, uniaxially packed at 200 MPa and sintered at 1450 °C for 2 h. Then, he calculated mass loss in solutions of 50 ml HCl and NaOH. Huang et al. determined through the sample

Table 2 Chemical resistance of aluminum oxide ceramics in different acids and alkalis [2]

Reactant	Mass content wt%	Temperature treatment °C	Weight loss, mg (cm ² year) Densely sintered Al_2O_3 (99 wt% Al_2O_3)
H_2SO_4	98	100	65.0
HNO_3	70	100	7.0
H_3PO_4	85	100	< 1000
HCl	25	70	72.0
HF	53	25	20.0
HF + HNO_3	10 + 57	25	16.0
NaOH	50	100	75.0
KOH	45	100	6.0

Table 3 Chemical resistance of corundum ceramics in different acids and alkalis [2]

Reactant	Mass content %	Temperature treatment °C	Corrosion, mm/year, of corundum ceramics, less than	
			Frialit F-96 (96.0% Al_2O_3)	Frialit F-99.7 (99.5% Al_2O_3)
H_2SO_4	97	105	0.110	0.120
HNO_3	65	105	0.030	0.040
H_3PO_4	105	105	0.090	0.095
HCl	35	105	0.018	0.020
HF	40	20	0.450	0.100
$\text{C}_6\text{H}_8\text{O}_7$	25	105	0.010	0.014
CH_3COOH	98	105	0.011	0.010
HCOOH	98	105	0.048	0.050
NaOH	40	105	0.150	0.040

morphology that corrosion occurs mainly in grain boundaries of alumina ceramic materials (Al_2O_3) Fig. 1 [20] and that mass loss increases with increasing temperature (Fig. 2) [20].

Interaction between $\text{CaO-Al}_2\text{O}_3\text{-SiO}_2$ leads to the formation of phases with low melting point temperatures, such as *gehlenite* or *anorthite*. Nanomaterials in shaped refractories raise the surface energy making them more reactive. Thus, their resistance to corrosion and thermal shock can be improved if the interaction of nano-phases with the other particles is favorably adapted [12].

Maitra, Das and Sen [12] studied the effect of tiny titanium oxide particles on densification of molten refractory cement $\text{Al}_2\text{O}_3\text{-MgO}$. They observed that the addition of TiO_2 results in a positive influence in densification, spinel formation and reduction in grain sizes of spinel formed at high temperatures in this system, modifying physical properties such as contraction in cooking, apparent density, apparent porosity and actual density. Besides, the microstructure of the bodies with added TiO_2 became more uniform and with less content of vitreous phases. Thus, it was suggested that the addition of TiO_2 leads to densification and phase formation, having a direct influence in the increase in their properties [12].

Takehiko Hirata et al. [1] reported that corrosion depth in Al_2O_3 ceramics is reduced by adding Cr_2O_3 , reaching the lowest corrosion value with 81.6% Cr_2O_3 mol–14.4 mol% Al_2O_3 –4 mol TiO_2 in the mixture. This is caused by diffusion difference in Cr_2O_3 , because diffusion rate for the Cr ion is rather slow compared to other metal ions (Fig. 3). It was also reported that corrosion depth increased by increasing CaO ratio contained in the slag, on the ceramic matrix formed by $\text{CrO}_2\text{-Al}_2\text{O}_3$ (Fig. 3) [1]. Since solubility and viscosity properties change in the slag as CaO content

Table 4 Acid and alkalis resistance of ceramics and porcelain [2]

Parameter	Chemical resistance of porcelain mixtures containing oxides %					
	No.1		No.2		No.3	
	SiO ₂	Al ₂ O ₃	SiO ₂	Al ₂ O ₃	SiO ₂	Al ₂ O ₃
	67.00	23.00	56.60	38.32	25.00	71.00
Resistance acid	98.3		98.72		98.94	
Resistance alkaline	58.12		72.19		88.59

Fig. 1 SEM image of Al₂O₃ before and after NaOH corrosion [20] **Fig. 2** Mass loss of Al₂O₃ with different solution temperatures (0.5 M NaOH), exposed for 5 h [20]

changes [21]. The dissolution process in ceramics can be controlled by the diffusion of oxide components, through the limiting layer of slag existing in the front side of the oxide surface, or by dissolution rate of oxides in the slag [1].

Liu et al. [18] studied corrosion resistance by adding ZrO₂ to a refractory Al₂O₃-C. Pointing that, adding ZrO₂ ion gives anticorrosive characteristics to Al₂O₃-C base, especially in FeO castings with concentrations higher than 6%, where carbon graphite Al₂O₃-C content is converted

in reducing agent of iron oxide, which is present in the casting. Figure 4 shows the relationship between FeO iron concentration and degree of corrosion [18].

M Serhat and Kara [3] reported that the addition of zirconia ZrSiO₄ to the mullite sintered matrix 3Al₂O₃ 2SiO₂ improves mechanical and corrosive properties upon alkaline vapors (Na₂O). Even considering that alkaline vapor corrosion is very severe for alumina-silicate refractories, since new phases produce stress and cracking. This is because of densification in *mullite* by adding zirconia to the mixture. Densification in the mixture reduces infiltration of alkaline vapors with reduction in porosity in the refractory material. Thus, when zirconia is added to the alumina-silica SiO₂ mixture (Fig. 5), it is possible to reduce apparent porosity to 8.9% increasing its corrosion resistance to alkaline vapors [3]. Porosity depends on the distribution of grain size for the sintered mixture. A structural ceramic material should have a porosity < 2%, whereas for a refractory it should be above 20% and between 50 and 70% for refractory insulation [22].

An EDX analysis in the densified area shows that the chemical composition at the reaction zone is different from that of the green material, where atoms of sodium, aluminum, silicon and oxygen were found in the reaction areas, and sodium was not found in the affected part [3].

Fig. 3 **a** The relationship between the depth of corrosion and CaO content in the CaO–SiO₂–Al₂O₃-based molten oxide. **b** Content of CaO [1]

Fig. 4 FeO content in melts and the corrosion rate of the refractories, test temperature 1773°K. [18]

LidijaČurković et al. [4] determined the correlation between the number of ions eluted from Al₂O₃ ceramics and time of immersion at different concentrations of H₂SO₄. This ratio shows the number of ions dissolved after corrosion process containing ions of Al^{3+} , Mg^{2+} , Ca^{2+} , Na^{+} , Si^{4+} and Fe^{3+} . This shows that impurities play an important role in corrosion process of ceramic alumina, since corrosion degree in Al^{3+} , Mg^{2+} , Ca^{2+} , Na^{+} , Si^{4+} and Fe^{3+} increases with corrosion time. Kinetic corrosion of high-purity Al₂O₃ ceramics at different concentrations (2, 10 and 20%) of H₂SO₄ at room temperature showed high

ion separation within the first 24 h, and for prolonged periods above 250 h, the effect remains stable Figure 6 [4]. Chang Woo Lee, Kang, Chang and Hahm [23] performed similar work for a porous alumina membrane subjected to HCl, HNO₃, H₂SO₄ acid solutions and NaOH, Ca(OH)₂ alkaline solutions independently highlighting the influence of temperature raise (20, 30 and 40 °C), concentration (0.001, 0.01, 0.1 N) and exposure time (30–180 min) in these solutions [23].

In 1981, Irwin and Amónof Westinghouse Electric Corporation [19] identified structural materials from an acid vaporizer to assess corrosion compatibility with the process stream. They subjected ceramic materials to sulfuric acid (H₂SO₄), under temperatures of 361–452 °C, and increments of 250–1000 h. It was found that silicon and materials containing significant amounts of silicon, such as silicon carbide and silicon nitride, had the highest resistance to boiling sulfuric acid. In another corrosion study carried out by Fernanda Coen-Porisini [19], ceramic samples were subjected to 800 °C in two different vapors containing certain decomposition products of sulfuric acid. Both tests did not show significant changes for Al₂O₃ alumina, 3Al₂O₃ 2SiO₂ mullite, and ZrSiO₄ zirconia, only small deposits on the surface. Tiegs [19] identified silicon carbide as the best ceramic exposed to temperatures between 1000 and 1225 °C in sulfuric acid, simulating a decomposition environment. In 2004, Shintaro Ishiyama [19] applied for a patent of a high-temperature corrosion resistant ceramic; his results show a percentage change in weight and corrosion rate for the samples after 100 h of exposure to boiling sulfuric acid under high pressure. Based on these studies,

Fig. 5 Outer surface of the test cups after alkali attack test [3]

Fig. 6 Total amount of eluted ions (Al^{3+} , Mg^{2+} , Ca^{2+} , Na^+ , Si^{4+} and Fe^{3+}) from Al_2O_3 ceramics in different concentrations of H_2SO_4 solution [4]

we can say that SiC silicon carbide, Si_3N_4 silicon nitride and Al_2O_3 alumina are the most suitable to withstand corrosion testing [19].

4 Conclusions

The corrosion resistance of high-purity alumina ceramics immersed in acid solutions (H_2SO_4 , HCl , HNO_3) and alkaline solutions (NaOH , KOH , Na_2O , $\text{Ca}(\text{OH})_2$) is determined by the degree of solubility of impurities contained within the grain limits (MgO , CaO , SiO , FeO and Na_2O). Due to the corrosion mechanism produced is a type of intergranular corrosion with preferential attack at the grain boundaries, where most of its impurities are found.

Also concentration of acid or alkaline solutions and the exposure temperature of either acid ceramic (SiO_2 ,

Al_2O_3) or alkaline ceramics (MgO , CaO , Cr_2O_3) are important factors to determine the corrosion resistance.

The addition of zirconium dioxide (ZrO_2), zirconium silicate (ZrSiO_4), titanium oxide (TiO_2), chromium oxide (Cr_2O_3), or even micro-silica (SiO_2), to alumina ceramics, improves corrosion resistance, even more than by only exposing high-purity alumina ceramics to acidic or alkaline solutions. Due to this, sintering has structure such as Al_2O_3 - MgO refractory cement with higher densification and reduction in grain size than spinel phase alumina (Al_2O_3). The high produced densification causes less infiltration of corrosive medium due to low porosity. Depth corrosion is even reduced by adding amounts of Cr_2O_3 - Al_2O_3 ceramics, because of delayed speed with which the slag diffuses through the material.

The phases that offer greater resistance to acid or alkaline solutions are achieved by sintering the alumina base ceramic material trying to obtain the higher content of corundum α - Al_2O_3 or mullite $3\text{Al}_2\text{O}_3\cdot 2\text{SiO}_2$ phases.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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