



Chemical interaction between Ba-celsian ($\text{BaAl}_2\text{Si}_2\text{O}_8$) and molten aluminum

M.N. Ibarra C^{b,*}, J.M. Almanza R^a, D.A. Cortés H^a, J.C. Escobedo B^a, R. Martínez-López^a

^aCINVESTAV-Unidad Saltillo, Ave. Industria Metalúrgica 1062, Parque Industrial Saltillo-Ramos Arizpe, CP 25900 Ramos Arizpe, Coahuila, México

^bCorporación Mexicana de Investigación en Materiales, S.A. de C.V. Ciencia y Tecnología No. 790, Fracc. Saltillo 400, Saltillo, Coahuila CP 25290, México

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Abstract

Wetting and corrosion experiments by molten aluminum were performed on mullite and Ba-celsian ($\text{BaAl}_2\text{Si}_2\text{O}_8$) substrates. The $\text{BaAl}_2\text{Si}_2\text{O}_8$ was synthesized from a mixture of alumina, silica and barium carbonate at 1450 °C for 10 h. The formation of pure $\text{BaAl}_2\text{Si}_2\text{O}_8$ was corroborated by X-Ray Diffraction analysis. Corrosion tests were performed by an immersion test of samples in molten pure aluminum or 319.0 alloy at 900 °C for 24 h. The wetting angles were measured using the sessile droop test at 900 °C for 2 h using the same Al alloys. The mullite sample showed a severe corrosion after the test, while the $\text{BaAl}_2\text{Si}_2\text{O}_8$ sample showed a corrosion layer of about 100 μm. The wetting angles were 109° and 127° for mullite in contact with pure Al and 319.0 alloy, respectively, while for $\text{BaAl}_2\text{Si}_2\text{O}_8$ were 153° and 149°, showing a no wetting behavior.

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1. Background

Aluminum industry is constantly growing and there is a constant demand of better refractory materials [1]. There are several requirements for refractories in contact with molten aluminum, such as thermal shock resistance, chemical stability and some other physical characteristics such as low porosity. The increase in the production amount and quality of aluminum alloys demand the use of higher quality refractories [2]. Silica based ceramics are widely used due to their low cost and availability [3]. The aluminum reduces SiO_2 to form alumina, which has a higher expansion coefficient than mullite.

This alumina layer breaks down due to thermal fluctuations allowing to the aluminum to infiltrate and then, the corrosion is accelerated [4,5]. Silicoaluminates based refractories with high amount of impurities (Na_2O , Fe_2O_3 ...) shows poor corrosion resistance to molten aluminum [4] due to the formation of glassy phases that accelerates the corrosion [6]. The addition of alkaline salts to the molten aluminum bath increases the degradation of SiO_2 based refractories due also to the formation of a glassy phase. The energy saving measurements, such as the use of low thermal conductivity materials in the furnace exterior increase the inner temperature, lowering the viscosity of aluminum and increasing the penetration towards the refractories. This penetration depends in great amount on the porosity and pore size of the refractories [2]. The decrease in porosity and pore size in refractories is a difficult task due to the nature of refractories fabrication. On the other hand, one of the most important requisites for a refractory in contact with molten aluminum is the inertness of the material in order to maximize the useful life [7]. Several materials are inert to

*Corresponding author. Tel.: +52 844 2059675/4381100.

E-mail addresses: marcelanohemi@gmail.com,
marcela.ibarra@comimsa.com (M.N. Ibarra C),
manuel.almanza@cinvestav.edu.mx (J.M. Almanza R),
dora.cortes@cinvestav.edu.mx (D.A. Cortés H),
jose.escobedo@cinvestav.edu.mx (J.C. Escobedo B),
ricardo.mtzl@cinvestav.edu.mx (R. Martínez-López).

aluminum attack such as alumina or SiC, but there are expensive materials. Among other factors that affect corrosion, the wetting of the material by the molten alloy, plays an important role. In order for the corrosion to start the molten metal has to wet the refractory. Wetting depends on a great number of factors, such as temperature, rugosity, reactivity, etc [8]. In an experiment of the sessile drop test [9,10], contact angles higher than 90° means no wetting and therefore as far as corrosion resistance concerns, these are the goal in the development of nonwetting refractories by aluminum alloys.

In order to reduce the wettability of refractories by molten aluminum, several nonwetting additives have been used. It has reported the use of Ce₂O₃ [4], AlF₄ [1,3,11,12], CaF₂ [1–3,7,11,13,14], AlPO₄ [15], V₂O₅ [16], clinker white cement [14], B₂O₃ [17] and BaSO₄ [1–3,7,11,12,18]. Refractories that contain barium sulfate showed the formation of BaSi₂Al₂O₈ after burning at high temperatures and this material is considered the responsible for the non wetting behavior of refractories by molten aluminum; however, there are not reports of wetting angles for this compound. The BaAl₂Si₂O₈ has appropriate properties, such as low thermal expansion coefficient ($2.29 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$) and high melting point (> 1700 °C), these make this material a good candidate to be used as a refractory material [19–22].

The main objective of this work is to evaluate the wettability and corrosion resistance of BaAl₂Si₂O₈ by aluminum alloys and to compare the results with those of mullite ceramics.

2. Experimental

The BaAl₂Si₂O₈ samples were prepared from a stoichiometric mixture of Al₂O₃ (Aldrich reagent grade), SiO₂ (silica, 99.8 wt%, Possehl) and BaCO₃ (Aldrich reagent grade) which was homogenized in a ball mill using acetone as dispersion medium for 4 h in spinning rods. The mixture was dried at 60 °C for 24 h and then grounded in a mortar to eliminate agglomerates. Cylindrical samples with a diameter of 4 cm and 0.5 cm in height were prepared using uniaxial pressing at 78 MPa. Samples were sintered at 1450 °C for 10 h. Samples after sintering were analyzed by Scattering Electronic Microscope (SEM) and X-Ray Diffraction (XRD). Additionally, a sample of mullite powder (Virginia K, mesh 325) was prepared following the same procedure and sintered at 1450 °C for 6 h. For these experiments, non-stoichiometric mullite (57.72 Al₂O₃, 39.33 SiO₂, 0.76 Fe₂O₃, < 0.010 K₂O, < 0.012 Na₂O, 0.085 CaO, < 0.008 MgO, wt%) was used in order to ensure sinterability of the substrates due to the SiO₂ excess content. Sintered mullite was also analyzed by SEM. Pure aluminum (99.74% purity) and an Al–Si alloy 319.0

containing about 6.5 wt% Si as the major alloying element were used in this study of the molten alloy in contact with the ceramic. Table 1 shows the chemical composition and melting point of pure aluminum and the Al alloy used in the corrosion and wetting experiments.

The corrosion tests for mullite and BaAl₂Si₂O₈, were carried out by immersion of the samples (4 cm in diameter) in molten aluminum alloy. The test was performed at 900 °C for 24 h. After testing, samples were cross-sectioned, grounded using 80, 120, 320, 500, 800, 1000, 1200 and 2400 grit SiC papers, then they were finally polished using a 1 μm diamond paste and analyzed by SEM.

Contact angle for each sample was measured using the sessile drop test under an argon atmosphere [9,10]. Before the test, cubes of 1 cm per side of pure aluminum or aluminum alloy were obtained and grounded using SiC papers and then cleaned with acetone. These cubes were placed over the ceramic substrate (4 cm in diameter with no superficial modification) and placed inside the tube furnace at room temperature. Additionally, Zr sponge was also placed inside the tube to consume the residual oxygen. Tests were carried out at 900 °C for 2 h using 10 °C/min rates for heating and cooling. In order to study the wetting behavior of the samples under severe conditions, a temperature of 900 °C was selected. To assure equilibrium, a 2 h holding time was chosen. All wetting experiments were performed under a high purity argon (99.999%) atmosphere with a continuous flow rate (5 L/min). The tube furnace was equipped with a transparent window at one end which allows to record video and measure the contact angle formed between the ceramic substrate and the drop of molten aluminum or alloy. The contact angle measurement was carried out based on images acquired at an interval of 5 min from video recorded during 2 h of testing. Contact angles were determined drawing tangent lines in the contact zone between the liquid drop and the ceramic substrate in both sides of the drop. In a previous work [24], sessile drop tests were carried out using this assemble.

3. Results and discussion

Fig. 1 shows the SEM image and EDS spectra for mullite sample sintered at 1450 °C for 6 h. This SEM analysis confirms the presence of mullite, beside some impurities of Ti and Fe in the sintered substrate.

Fig. 2a shows the XRD pattern of the Al₂O₃–SiO₂–BaCO₃ mixture after sintering at 1450 °C for 10 h. The only phase detected was hexagonal Ba-celsian (BaAl₂Si₂O₈). Fig. 2b shows the SEM analysis for that sample, it can be detected some alumina particles and some white needles that contains

Table 1
Chemical composition (wt%) and melting point of pure aluminum and Al alloy.

Chemical composition	Melting point (°C) [23]	Al	Si	Mg	Cu	Fe	Pb	Mn	Zn	Others
Pure Al	660	99.74	0.02	0.0009	0.0003	0.10	0.06	0.002	0.005	0.06
Al–Si	605	85.77	6.5	0.04	3.23	0.95	0.04	0.28	1.85	1.34

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