Available online at www.elixirpublishers.com (Elixir International Journal)

Applied Chemistry

Elixir Appl. Chem. 127 (2019) 52601-52607

ISSN: 2229-712X

Formulation of Ceramic Crucibles for Fire Assays

Brahima sorgho[1*], Lamine Zerbo[1], Boubié Guel[1], Philippe Blanchart[2] and Moussa Gomina[3]

[1]Team of Physic, Chemistry and Technology of Materials, Laboratory of molecular Chemistry and Materials,
UFR-SEA, University Ouaga I Professor Joseph KI-ZERBO, 03 BP 7021 Ouaga 03, Burkina Faso.
[2]Institute of Research for Ceramics–IRCER., European Ceramic Centre (CEC), 87068 Limoges, France.
[3]CRISMAT Laboratory, ENSICAEN, 6, Bd. Maréchal Juin, 14050 Caen, France.

ARTICLE INFO

Article history:
Received: 24 December 2018;
Received in revised form:
06 February 2019;
Accepted: 18 February 2019;

Keywords
Refractory Crucibles,
Fire Assay,
Clays,
Burkina Faso.

ABSTRACT

The valorization of minerals requires physicochemical and thermal treatments, using various materials such as crucibles. They are refractory ceramics mainly based on clays. Crucibles must be resistant to chemical aggression by molten slag and metals at high temperature during repeated thermal cycles, to ensure the durability in use. Fire-assaying is extensively used in gold mining in Burkina Faso, needing a huge quantity of ceramic crucibles imported per year. In this study, we have characterized different clays to manufacture refractory ceramics for crucibles. Physical characteristics of laboratory made crucibles were compared to that of industrial crucibles. The behavior of an optimized composition with 25wt% of clay and 75wt% of chamotte (fired clay) was experimented to optimize the properties in use. Particularly, the adequate adjustment of all process parameters, as the paste plasticity, leads the control of the density and porosity of the fired ceramic. Fusion tests with copper and aluminum at temperature between 600°C and 1100°C proved the small penetration depth of the molten metal into the ceramic that reduce the corrosion phenomenon. Our work evidences the existence of both a scientific and a technological knowledge in the use of silico-aluminate mineral resources from Burkina Faso, for manufacturing refractory crucibles for the melting of both slag and precious metals.

Introduction

Refractories are structural materials, working at high temperatures in a severe environment, whether it is corrosion by molten metals or slags in the industry, or aggressive chlorinated atmosphere in incinerators. The strategic importance of these materials is considerable. They respond to a real demand of four important economic sectors : -the traditional sectors of high temperature processes, such as metallurgy, glass industry, ceramics and raw materials; -the environment and more particularly the field of treatment and energy recovery from wastes, as in the case of waste incineration; -the petrochemicals and the emerging sector of biofuels; -the new energy production of heat and electricity, such as hydrogen production or new generations of nuclear power plants [1].

The main characteristics of refractory ceramics are the high temperature level and therefore infusibility. Besides, materials must present complementary properties to withstand the stresses they undergo in service. Their behaviors are mainly governed by the corrosion phenomena, the chemical composition, the mineralogical characteristics of phases, and the microstructural characteristics [2]. Knowledge on the detailed characteristics are also essentials as the thermomechanical properties of materials under stresses, the improvement of the thermal shock resistance, and the reduction of the degradation at high temperature [3].

Most refractory materials are heterogeneous ceramics, which microstructure is mainly composed of relatively large grains forming a skeleton and a matrix phase that bound large grains.

Porosity always occurs within the microstructure, with either open and closed pores. Size distribution and possible heterogeneities of the larger grains, the matrix phase and pores are essential in physical behaviors of refractories [4,5,6]. The microstructural characteristics are controlled by the type of mineral materials, the fabrication process, and the sintering process [7].

Refractories classification is usually made from their chemical and mineralogical characteristics. The four main groups are: -silico-aluminate refractories based on refractory clays; -oxides refractories as alumina, mullite, silica and zirconia; -refractories based on magnesite, chromite or dolomite mineral compounds; -specific refractories based on carbon, carbides, nitrides, and spinel phases.

In the group of silico-aluminate refractories, clay refractories are extensively used for common applications in small and very large industrial kilns, and heating systems. They are very low-price materials most often composed of kaolinitic clays, for the matrix phase and chamotte or sand, for the granular phase. Different standard shapes of different sizes are manufactured to produce bricks for kiln walls, roofs, arches, tubes and circular apertures, etc. Refractories are also used to manufacture crucibles and molds for casting glass and metals. They are used by the iron- and steel-industry, and the metal casting sectors.

Very large quantities of crucibles are also used for the Pb fire-assay technology. Because of its precision and accuracy, it is the most employed method for gold analysis in geological materials.

Tele: +226 70 06 37 82
E-mail addresses: sobrah20@yahoo.fr

Table 2. Chemical and mineralogical compositions of RS 335 and RR 40 clays.

Sample	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	Na_2O	K_2O	loss of ignition	Kaolinite	Quartz
RS 335 (%)	62.4	24.3	1.4	1.4	0.4	0.4	< 0.1	1.2	8.6	≈ 70	30
RR 40 (%)	54.7	39.5	2.2	2.3	0.6	0.2	traces	traces	13.0	≈ 92	8

It is a key sector for industries related to gold mining in countries where gold resource is extensively available, as in Burkina Faso [8].

Fire assaying is a quantitative chemical analysis by which metals are separated and determined in ores and metallurgical products with the aid of heat and dry reagents. This technique is used since years to concentrate gold and other noble metals from ores or metallurgical products [9,10,11]. In a first step, a melt of at least two phases is formed by heating at about 1100°C a complex liquid borosilicate slag and a liquid lead phase of controlled quantity. The high degree of physical combination of gold and other noble metals in molten metallic lead and the great difference in specific gravity between the lead and slag allow the separation of the noble metals from the slag as a lead compound. In a second step, the removal of the lead as lead oxide in a porous magnesia cupel during a carefully controlled oxidizing fusion at about 950°C separates the lead from the noble metals. The remaining metallic bead is then quantitatively analyzed for the noble metals.

The crucibles in service undergo failure mechanisms that must be checked: -a chemical attack leading to corrosion from the flux addition; -a mechanical damage by the high temperature and the thermal shocks. To ensure a high standard of quality, it is necessary to develop tests to simulate failure, during laboratory experiments.

The article focuses on the optimization of clay refractory materials for specific crucibles that will be used for fire assay. The study also aims to realize tests in the metal contact region, where the most aggressive set of conditions occur.

Materials and methods

The clay raw materials for the study are from France (DAMREC Company). The two clays used are referenced RS 335 and RR 40. These clays were considered as reference clays since some laboratory studies at large scale were performed in France: - from preliminary studies, we evidenced the very similar composition and behavior of RS 335 and RR 40 clays with clays mined in Burkina Faso [5]. Particularly plasticity of paste, as drying and firing behaviors were studied.

The chamotte is a refractory clay that is calcined at a temperature between 1200 and 1400°C. It is milled and sieved to control grain size. The used chamotte is a commercial product from CERADEL Company, France. As for clays, the chamotte characteristics and thermal behavior is very similar to chamotte that have been obtained during small scale laboratory works in Ouagadougou University. The used chamotte is obtained from kaolinitic clay that is heat treated at 1300°C. It has controlled grain size and chemical analysis.

The raw clays were crushed and sieved at 315µm, to remove larger grain that reduces the mixture homogeneity. The clays (RS 335 or RR 40) and the chamotte with particle size of 200 µm were mixed homogeneously in a planetary mixer, and water was added progressively (21-28 wt%) to obtain a plastic paste for the pressing process. Compositions of mixtures reported in **Table 1,** and water content for clay-chamotte mixtures are within common ranges for industrial uses [3].

Pellets of 13 mm in diameter and 15 mm height were obtained by die pressing at 20 MPa in a metal mold. They are dried at temperature varying progressively from 40°C to 110°C during 8h. Firing is at 1250°C with a 30 minutes plateau.

Table 1. Compositions of clay-chamotte mixtures.

RS 335 (wt %)	RR 40 (wt %)	Chamotte (wt %)	Reference
50	0	50	C1
25	0	75	C2
0	50	50	C3
0	25	75	C4

The shaping process of crucible is a unidirectional die pressing of the plastic paste. It is performed in an aluminum die (**Figure 1**), with a small quantity of the plastic paste placed at the bottom and formed by a press flow during molding. It is intended to shape crucibles of 5 cm in diameter and 5 cm in height. To improve the process of plastic flow in the mold, the rheological properties of the plastic paste was characterized using a Vicat penetrometer apparatus. We measured the spot size of a cone under a constant load of 300 g on the flat surface of paste inside a cylindrical contained, and after the careful release of the cone [12]. The validity of this method was proved with different clay pastes for the characterization of the plasticity of clay–water systems, although it is a semi-quantitative characterization method which does not exactly fit with the theory of plasticity. For all plasticity measurement methods, a common procedure for all types of materials does not exist, and the most important methods are those that simulate the conditions of real processing [13].

After firing, the densities of pellets were measured using a helium pycnometer (Micromeritics). For these measurements, the helium flow rate was set at the pressure of 1.7bar.

Microstructural observations were made using an optical microscope. At a smaller scale, we used a scanning electron microscope HITACHI SC-2500, to observe polished surfaces.

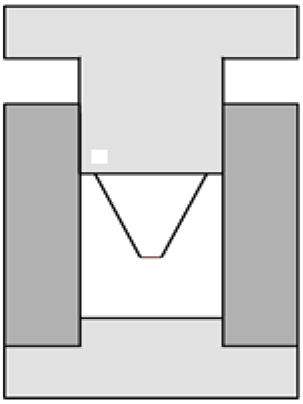

Figure 1. Desingn of the metal molds used for shaping crucibles.

Chemical and mineralogical compositions of mineral materials

Data of chemical analyses of the clays and the chamotte were obtained from the suppliers and reported respectively in **Table 2 and Table 3**. Chemical analyses indicate that silica and alumina are the predominant oxides in the RS 335 and RR 40 clay samples.

The silica mass ratio (SiO_2/Al_2O_3) of RS335 is about 2.56. This value is much greater than that of pure kaolinitic clay, for which it is about 1.18. It evidences the presence of a large amount of free silica under the form of quartz in the RS 335 clay. However, the silica mass ratio of RR40 is 1.38 that reflects the presence of a small amount of free silica and quartz [14].

The mineralogical compositions of the clays are in **Table 2**. It shows the high content of kaolinite mineral (RS 335 ≈ 70% and RR 40 ≈ 92%) although RS 335 contains a higher content of quartz (30%) than that of RR 40 (8%).

Chemical and mineralogical compositions have an important role on the ceramic properties during firing. However, the morphology and distribution of phases in the mixture and the type of the thermal cycle have also a predominant role. The complex interaction of all processes and compositional parameters are based on an extensive knowledge that is presented in ref [1]. For the used mineral materials, they have chemical and mineralogical compositions that fit with their potential use in refractory ceramics.

Table 3. Chemical analyze of the chamotte.

Oxide	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	Na_2O	K_2O
%	54.2	41.0	1.7	1.7	0.3	0.3	0.1	0.7

For the chamotte, (**Table 3**), from the high values of silica (SiO_2 = 54.2%) and alumina (Al_2O_3 = 41.0%), we expect the presence of heat transformed phases, that are recrystallized. From the silica-alumina phase diagram, mullite and silica are the predominant phases [15]. Depending on temperature and thermal cycle, silica is under the form of both the untransformed quartz and a recrystallized cristobalite phase. Besides, a vitreous silico-aluminate phase containing silica and alumina is combined with the minor oxides of iron, titanium, calcium, magnesium, sodium and potassium.

Composition of crucibles

Experiments on rheological properties of the plastic pastes were conducted with three different moisture values (22%, 25% and 28%). The compositions C1 and C3 (**Table 1**), containing the clay and the chamotte in equal proportions appear stickier than the compositions C2 and C4, in which the chamotte content is higher.

Figure 2 shows the results of the plasticity tests against moisture. It I seen that the cone penetration value increases from C3 to C4, C1 and C2, that is correlated to the increase of plasticity. However, the behaviors against plasticity is quasi-linear for C4 and C2, but non-linear with C3 and C1. It is in relation with the clay content that is 50wt% in C3 and C1 but decreases to 25wt% in C4 and C2 (**Table 1**).

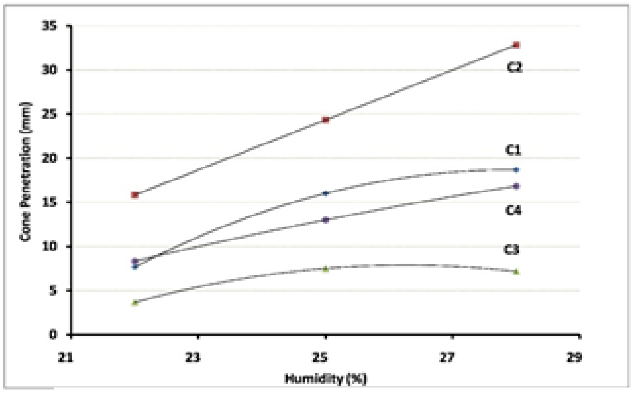

Figure 2. Plasticity experiments as a function of plastic pastes (wt%).

The behavior of C1 and C4 seems more suitable for pressing, since the plasticity value appears to be adequate for the pressing process but avoiding a too sticky behavior in the mold.

After drying and firing, the densities and porosities of pellets of the various compositions are in **Table 4** that evidence the strong correlation between density and porosity data. Only the composition C4 has a satisfactorily low porosity value, which is similar to what is required for manufactured crucibles. Compositions C2 and C3 have values higher than that of C4. The composition of C1 is in between the composition of C4 and those of C2 and C3.

Table 4. Density and porosity of fired pellets of different compositions.

Sample	Density	Porosity (vol%)
C1	2.05	22.30
C2	1.84	28.12
C3	1.88	28.45
C4	2.05	18.51

To check the resistance of the pellets of different compositions in contact with a melted metal, tests were carried out with copper (**Figure 3**). Its melting temperature is 1085°C that is slightly higher than that of gold (1064°C). The pellets at the copper contact were tested at 1100°C for 30 min. After melting, penetration depths of copper were observed as a function of the composition. It is an indication of the resistance of different crucibles at high temperature in contact to the melt. Although, copper oxidation is observed due to its high oxidation kinetics at 1000°C, observations of microstructures on perpendicular section at the metal-refractory contact are significant. Optical microscopy observations are in **Figures 4, 5, 6, 7, 8**.

According to **Figures 4, 5, 6**, compositions C1, C2 and C3 have an impregnation depth of more than 2.5 mm. This is most probably related to the higher porosity of these compositions. From **Figures 4, 5** and **Table 4**, we notice that the RS335 clay is poorly adapted since the porosity and depth of impregnation are high. Moreover, the microstructure at the metal-refractory contact is very heterogeneous that is not suitable for durability in use at high temperature. From **Figure 7**, we observe a more homogeneous and compact microstructure at the interface. It results in a smaller penetration depth of molten copper that doesn't exceed 1.5 mm. This behavior is similar to that of a manufactured crucible, in **Figure 8**, which undergone a penetration depth of less than 1.5 mm.

Microstructural observations result in average penetration depths for all compositions that are reported in **Figure 9** against porosity. The very different behavior of C1, C2 and C3, and C4 compositions is mostly related to clay type and chamotte content. For RS 335 clay, that is the less refractory clay (Al_2O_3 content of 24.3wt% in **table 2**), an increase of chamotte ratio induces the increases of porosity and penetration depth. It is due to the fluxing behavior of the clay in-between chamotte grains at high temperature that favors the formation of large pores. With RR40 clay, the situation is different since porosity decreases drastically when the chamotte content increases. It results from the refractory behavior of RR40 clay (Al_2O_3 content of 39.5wt% in **table 2**) that closely bound chamotte grains, avoiding the formation of large pores.

Figure 9 evidences that the crucible with RR40 clay has optimized properties. In that case, the type of clay and the chamotte/clay ratio are suitable to obtain a behavior close to that of an industrial crucible.

As a result, the composition C4 (25% clay + 75% chamotte) will be used for advancing in crucible technology.

Figure 3. Pellet surfaces with copper after heat treatment at 1100°c.

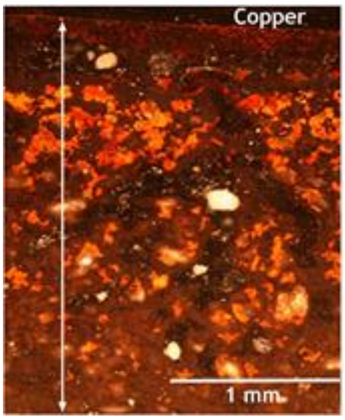

Figure 4. C1 material. Penetration depth > 2.66 mm.

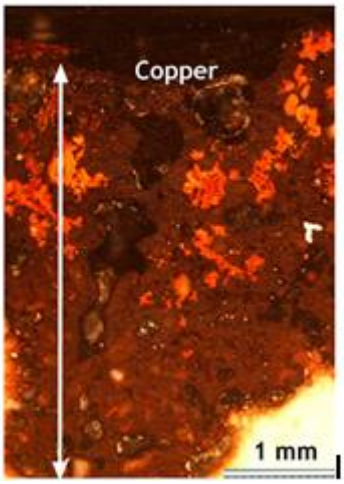

Figure 5. C2 material. Penetration depth > 2.66 mm.

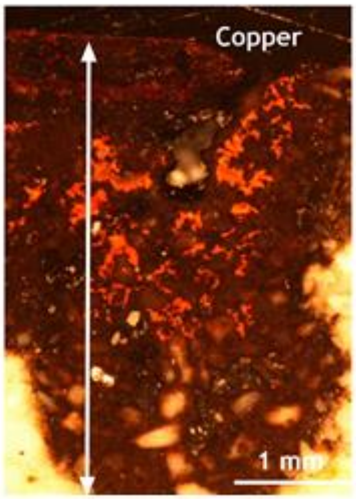

Figure 6.C3 material. Penetration depth = 2.66 mm.

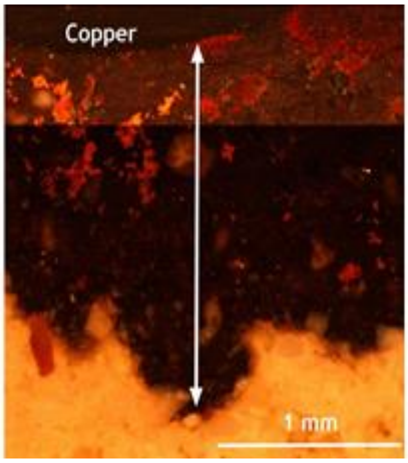

Figure 7. C4 material. Penetration depth = 1.83 mm.

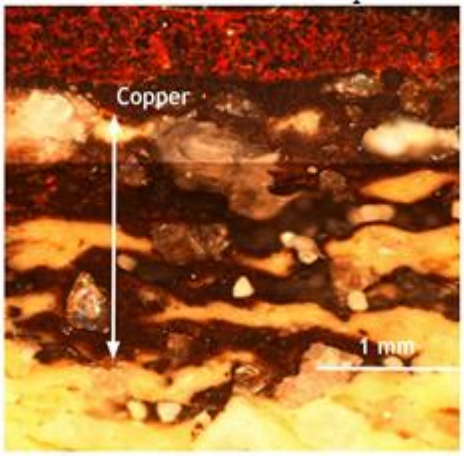

Figure 8. Reference crucible. Penetration depth = 1.85 mm.

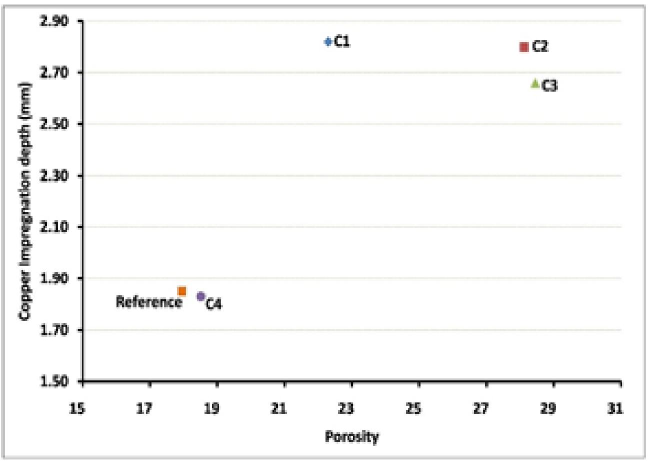

Figure 9. Average penetration depth of copper from surface of refractory ceramics, as a function of porosity (Vol%).

Shaping the crucible by pressing

To carry out an optimal pressing process with C4 composition, it is necessary to optimize both the quantity of pressing paste that must be placed into the mold, and also the moisture content of the clay-chamotte plastic paste.

With the aluminum mold of Figure 1, a first test with 130g of paste was rather satisfactory but tests with 150g made it possible to obtain crucibles with a more compact texture and with a smoother appearance. Regarding the moisture adjustment, preliminary tests showed an optimal humidity between 22 and 25% of water (Figure 2). With C4 paste, we performed complementary experiments with the Vicat penetrometer apparatus and results are in Figure 10.

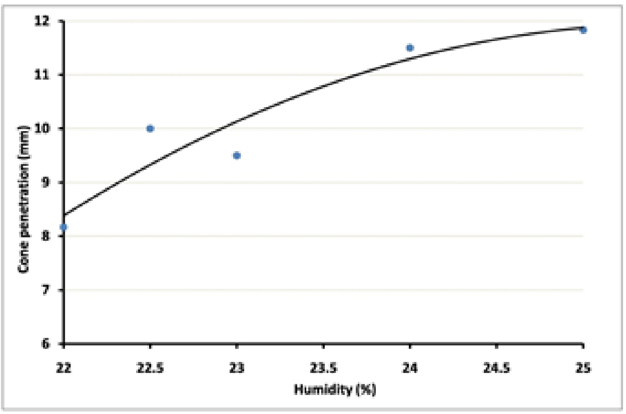

Figure 10. Plasticity measurements with C4 composition against paste humidity (wt %).

We note a non-linear and continuously increased plasticity when the water content increases. As for first plasticity measurements (Figure 2) with a common procedure, we observe that for 22.5wt% to 23wt% of moisture the paste is within an accurate plasticity range. As consequence, pressing gives a crucible with a good surface appearance. Below 22.5% of moisture, the paste is not sufficiently plastic, while above 23%, it becomes too deformable.

Aluminum melting in crucibles

For melting tests in optimized crucibles, aluminum (melting temperature of 660°C) was chosen in place of copper to reduce the oxidation process at high temperature [16]. Melting of aluminum was carried out in an elevator furnace and the process is the following:

The crucible was first heated in the kiln at 800°C. Once a stable temperature is attained, 15 g of aluminum was added into the crucible. Fusion is then operated for 30 minutes, and the melted metal is poured to cool in air (Figure 11). At the end, the cooled crucible is observed to investigate the extend of the metal-refractory interaction process.

Figure 11. Aluminum melting in C4 crucible.

Aluminum interactions on surface of crucibles

The melting experiments with aluminum were carried out with three types of crucibles:

- the C4 uncoated crucible (**Figure 12**); With this C4 uncoated crucible, there is a fine black deposit at the interface of the metal and the crucible, and a part of metal is sticking on crucible surface. The uncoated crucible sample clearly reveals some remaining metal that adhere to the crucible surface. It can be supposed a significant role of the high surface roughness, clearly evidenced in **Figure 12**. However, aluminum melt did not penetrate the open pores at the surface vicinity.
- a C4 glazed crucible with a silico-aluminate glass melting at 1150°C; With this glazed crucible (**Figure 13**), a very thick black deposit is present on the refractory surface, but the metal tends to come off the surface of the crucible. In **Figure 13** for the glazed crucible, a vitrified coating is on refractory surface, with an average thickness of 0.6 mm. This coating is not porous and presents a smoother surface than that of the ceramic. It reduces drastically the interaction between the molten metal and the ceramic, and after cooling, there no residual adherence of the metal on the surface.
- a C4 crucible preliminary impregnated with coal tar that is a by-product coke and coal gaz. With this crucible impregnated with coal tar (**Figure 14**), no trace of adhesion of metal on refractory surface can be observed. We also note that the three crucibles were not damaged by thermal shock during 10 repeated thermal cycles.

To observe microstructural characteristics of metal-refractory interface, we cut the crucibles used for melting. Representative samples were specifically polished for scanning electron microscopy (SEM) observations.

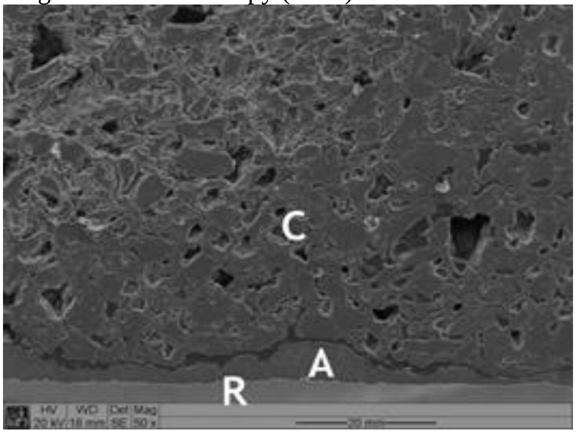

Figure 12. SEM image of the interface between aluminum (bottom) and crucible. C: crucible, A: aluminium, R: polymer used during the polishing process.

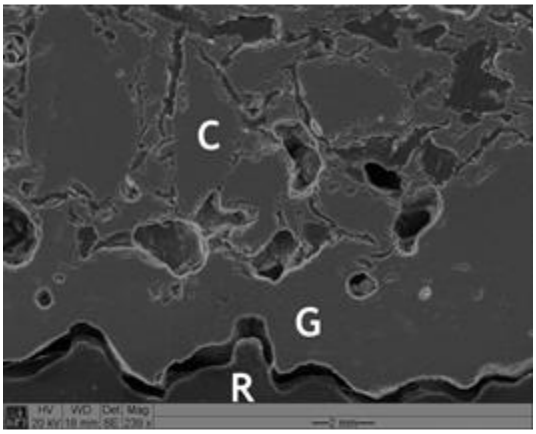

Figure 13. SEM image of the glazed crucible. The dense glazed coating is in-between the surface and the ceramic. C: crucible, R: polymer used during the polishing process.

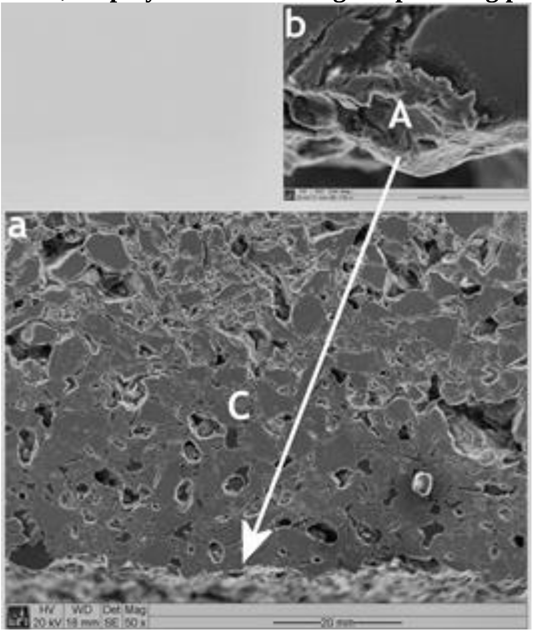

Figure 14. SEM image of the coal tar-impregnated crucible. a: General view of the cross section; b: closer view of the interface where aluminum is still bonded. C: ceramic; A: aluminium on surface.

In Figures 14a and 14b, the crucible impregnated with coal tar shows a behavior similar to that of the glazed crucible, and no molten aluminum is inside open pores. However small layers of aluminum are locally bonded to crucible surface. It can be explained by the specific behavior of coal tar. It is a very complex mixture of aromatic compounds that are quite stable and resist breaking even at the relatively high temperature. It resists to intrusion from liquid phases and does not break down even at the relatively high temperature of 600°C. The high temperature form of coal tar contains carbon-based phases but may contain inorganic compounds. They should be combined with the molten aluminum to form chemical bounds onto the ceramic surface. Besides, the surface roughness is an essential feature to avoid crucible corrosion. The crucibles coated with a surface layer of coal tar are less damaged by the metal in contact, since it cannot infiltrate into the pores on surface.

From Figures 12, 13, 14, it is seen that the corrosion resistance of ceramics is affected by both the chemical composition and the processing conditions. To avoid infiltration by the molten metal, the ceramic should be free of porosity and any constituent which is prone to dissolve in molten metal [17].

To improve the corrosion resistance, the mechanisms involved must be studied. In general, molten aluminum alloys attack the refractories by redox reactions in which silica and silicates in the refractory are reduced to form elemental silicon while metallic aluminum forms aluminum oxide.

$$4Al\ (liq) + 3SiO_2(s) \longrightarrow 2Al_2O_3\ (s) + 3Si$$

This transformation leads to a large expansion in volume, causing cracking of refractories.

The extend of corrosion depends on the silica content of the refractory, and especially that of the matrix phase. During the redox reaction, while aluminum and alloying elements diffuse into the refractory, silicon is released by the redox reaction and counter diffuses into the molten phase. A reaction layer containing alumina grows at the interface and acts as a barrier against further melt penetration. To improve the corrosion resistance of refractories, the wetting process of melt can be controlled with the reduction of surface roughness and with a coating barrier on surface.

With molten gold, the processes are different since gold doesn't react with oxide phases in silico-aluminate refractories. However, in a highly reducing condition a eutectic reaction may occurs in the gold-silicon system. The eutectic temperature is 363 ±2°C and the eutectic composition was found to be 19.0 ±0.5 at. % silicon. Such phenomenon can be prevented with an oxidizing atmosphere in the kiln.

The situation is different with molten slags [18]. Corrosion of refractory in contact with slags is from three major processes: - a chemical process of dissolution and diffusion; -penetration into open porosity from the surface; -erosion from the slag movement that is slow in small crucibles.

In general, slag viscosity is an important factor that affects corrosion. At the interface, the slag progressively dissolves some refractory oxide that increases the viscosity. It reduces the rate of further attack by the slag since a diffusion mechanism becomes predominant. Consequently, clay refractories are fairly resistant against molten slag. At the same time, slag resistance is also affected by the refractory porosity. A porous refractory is easily corroded by the slag, which soaks into the pores, whereas a dense refractory dissolve only slowly. However, the binding matrix of the refractory is more rapidly attacked than the solid grains, causing them to become loose and dispersed in the slag without dissolving.

These descriptions of mechanisms involved in the corrosion resistance of refractories further explain the role of the surface roughness, the porosity and the coating in C1, C2, C3 and C4 crucibles.

Conclusion

The study shows that high performance refractory crucibles can be obtained from mixtures of clay and chamotte. It is a very reliable way for achieving the characterization of precious metals in mineral resources by the fire assaying method. The crucibles have a suitable resistance to both the high temperatures, to the thermal shocks, and to damages from melted fluxes and metals. Our investigations on the mineral materials properties lead to an optimized composition of a plastic paste that can be shaped to form crucibles. The process parameters were optimized at all stages of the fabrication process. Particularly, the surface roughness and the open porosity of the fired crucibles can be improved by different coatings, proving their role in reducing the interactions between refractories and the molten metal and slags.

Realistic and successful tests were conducted with copper and aluminum that were melted in crucibles at temperatures between 600 and 1100°C.

The study evidences the possible use of inexpensive mineral resources and of simple fabrication processes to manufacture refractory crucibles. It also proves that scientific and technological knowhow on refractory crucibles are effectively available, to be used in fields of the mining industry of Burkina Faso.

Acknowledgments

This work was funded by the "FRITTE" project of the National Fund for Research and Innovation for the Development (FONRID) of Burkina Faso and Ouagadougou University. The authors express many thanks for the material and financial supports that this work has received during years.

References

[1]Poirier J. Refractory Materials, Ceramic Materials, Wiley Ed, 2010.

[2]Du Toit J, Cromarty R.D, Garbers-Craig A.M. Matte-tap-hole clay refractory brick interaction in a PGM smelter. The journal of the Southern African Institute of Mining and Metallurgy (2016) 339-342.

[3]Poirier J. Les céramiques réfractaires de l'élaboration aux propriétés d'emploi. Verres Céramiques & Composites (2011) 1, 28-42.

[4]Sorgho B, Ayouba Mahamane A, Guel B, Zerbo L, Gomina M, Blanchart P. Removal of Cd2+, Cu2+ and Pb2+ with a Burkina Faso clay. St. Cerc. St. CICBIA (2016) 17 365-379.

[5]Sawadogo M, Seynou M, Zerbo L, Sorgho B, Yameogo A, Millogo Y, Ouedraogo R. Densification behaviour of chamotte grog for refractory bricks: mineralogy and microstructure. J. Soc. Ouest-Afr. Chim. (2016) 041, 1- 10.

[6]Laibi A.B, Gomina M, Sorgho B, Sagbo E, Blanchart P, Boutouil M, Sohounhloule D. Caractérisation physico-chimique et géotechnique de deux sites argileux du Bénin en vue de leur valorisation dans l'éco-construction. Int. J. Biol. Chem. Sci. (2017) 11 499-514.

[7]Azzou A, Blanchart P. Predicting the sintering curve of porcelain by support vector regression. Journal of the American Ceramic Society (2011) 94 3768-3773.

[8]Djangang C.N, Elimbi A, Melo U.C, Lecomte-Nana G.L, Nkoumbou C. Sintering of clay-chamotte ceramic composites for refractory bricks. Ceramics International (2008) 34 1207-1213.

[9]Bugbee E.E. A Textbook of Fire Assaying, third ed. Colorado School of Mines Press, 1981, Colorado.

[10]Petrovic N, Bubelan D, Cokic S, Nesic B. The determination of the content of gold and silver in geological samples. J. Serb. Chem. Soc. (2001) 66 45-52.

[11]Cerceau C.I, de Freitas Carvalho C. Silveira Rabelo A.C, Gouvea dos Santos C, Mayra Dias Gonçalves S, Vieira Varejao E.V. Recovering lead from cupel waste generated in gold analysis by Pb-Fire assay. Journal of Environmental Management (2016) 183 771-776.

[12]Vijayananda M.D. An Alternative Method for Assessment of Consistency of Cement Paste. International Research Journal of Engineering and Technology (IRJET) (2016) 03 608-613.

[13]Andrade F.A, Al-Qureshi H.A, Hotza D. Measuring the plasticity of clays: A review. Applied Clay Science (2011) 51 1-7.

[14]Konan K. L. Interactions entre des matériaux argileux et un milieu basique riche en Calcium, Thèse de Doctorat de l'Université de Limoges (France), 2006.

[15]Klug F.J, Prochazka S, Doremus R.H. Alumina-Silica Phase Diagram in the Mullite Region. Journal of the American Ceramic Society (1987) 70 750-759.

[16]Wynn A, Coppack J, Steele T. Assessing Monolithic Refractories for use in Aluminum Melt-Hold Furnaces. Advanced Materials & Processes (2011) 6 28-31.

[17]Yan M, Fan Z. Durability of Materials in Molten Aluminum Alloys. Journal of Materials Science (2001) 36 285-295.

[18] Xian Jian Guo, Ruiqing Li, Ralph Harris. Kinetics of Oxygen Transfer through Molten Silver Cupellation Slag. Canadian Metallurgical Quarterly (1999) 27 333-41.