Monolithic refractories are well established as linings for a range of melting and holding applications during aluminum processing because they provide optimum productivity and cost effectiveness. A wide range of products is available and suppliers must be able to offer specifically tailored material solutions as aluminum furnaces have their own unique set of operating conditions compared to other refractory applications.

This article describes test methods used by major aluminum producers to assess and approve monolithic refractories for use in the key working zones of an aluminum melting furnace. Test results indicate that certain test techniques do not necessarily represent today’s operating conditions, which have changed as manufacturers strive to increase productivity through, in particular, increasing heat input to the furnace to melt the metal faster.

Monolithics, such as those supplied by Morgan Thermal Ceramics, are used to line the metal and nonmetal contact regions in typical melt-hold gas-fired reverberatory furnaces. Each region is divided into subregions as shown in Fig. 1, and each has a different set of operating conditions and environment for the furnace lining. Therefore, a variety of refractories are required for a complete furnace lining.

End users are melting and holding a variety of fluxing materials, so the monolithic products need to cope with the specific chemistry present in the furnace. In addition, different operating practices with respect to furnace management (for example, cleaning methods and frequency) mean that diverse physical conditions can influence different parts of the furnace.

Due to the diverse nature of the furnace environment, aluminum producers must maintain a complex, lengthy testing scheme for furnace linings. This is done to subject potential materials to the full range of conditions that they are likely to experience in service. Because there is wide range of conditions, it is not practical or cost effective to test materials for all types of conditions, so producers developed a practical set of laboratory tests.

Two main failure mechanisms that limit service life are chemical attack (corundum growth or corrosion from flux addition) and mechanical damage (ingot loading, cleaning practices, or thermal shock). Producers developed tests to simulate these as part of their approval program.

This article focuses on the investigation of tests on the metal contact region; this region produces the most aggressive set of conditions and represents the most demanding part of the furnace in terms of lining performance. Corundum growth is the most significant threat in this area, and, therefore, receives the most attention when designing and testing furnace lining materials.

Corundum forms when liquid aluminum reacts with free silica in refractories described by

$$4\text{Al (liq)} + 3\text{SiO}_2 (s) \rightarrow 2\text{Al}_2\text{O}_3 (s) + 3\text{Si}$$

and this transformation leads to a very large expansion in volume, causing severe distortion and cracking of the lining.

The most prevalent laboratory test for corundum growth resistance is the aluminum “cup” test. The objective of this investigation is to understand how different test conditions affect the behavior of the lining materials by evaluating how existing furnace lining materials behave when subjected to contact cup test methods used by aluminum producers.
Types of refractory tests

The standard metal contact “cup” tests of three large aluminum producers are outlined below. These procedures are routinely used to assess the suitability of monolithic refractories for use in melt-hold furnace linings.

**Method 1: Sample preparation and test procedure**

A series of 100 mm cubes are cast in molds (Fig. 2) from compositions mixed at standard water addition. Each cube has a 50-mm deep slightly tapered hole (55 mm in diameter at top, 53 mm at base), as shown in Fig. 3. Samples are set overnight, then demolded, cured, and dried at a temperature of 230°F for 18 hours. Half of the dried sample cups are prefired to 2190°F for 5 hours. Lids of the same material (25 mm thick) are also made to minimize loss of volatiles.

Typically, 7075 aluminum alloy is used for testing, supplied as 52 mm bar and cut to 50 mm lengths. The cut alloy sample is inserted into the hole in the sample cup and the lid is placed on top, unsealed. Both as-dried and prefired samples are tested at the same time for comparison.

The assembled cups are placed in a kiln, heated to 1830°F at a rate of 300°F/hour, and held at temperature for 100 hours. This is followed by natural cooling in the kiln. After cooling, the samples are sectioned vertically, dried, visually assessed for the degree of metal penetration, corundum growth, and ease of removal of the aluminum and photographed.

**Method 2: Sample preparation and test procedure**

Following the supplier’s mixing recommendation, a standard brick size (230 mm high × 114 mm wide × 76 mm deep) of the test material is cast into a mold (Fig. 4) that incorporates a curved face to form a cup shape with a maximum depth of 32 mm for holding the alloy (Fig. 5). After the recommended curing time, the sample is fired according to the supplier’s recommendation to 1500°F with a 10 hour hold and left to cool naturally in the kiln. The curved cup section is then roughened using a diamond saw to expose the refractory grain.

The cup sample is heated to 1500°F in a furnace at a rate not exceeding 300°F/hour. Meanwhile, 7075 alloy is melted in a silicon-carbide crucible, heated to 1500°F, and sampled for analysis. The molten alloy is ladled into the brick cavity at 1500°F to about 3 mm below the top of the brick and held at temperature for 72 hours.

The alloy is raked every half hour for the first three hours to remove the oxide film barrier at the metal/refractory interface. After 72 hours, the oxide formed on the top of the molten alloy is cleaned and a sample of the alloy from the cup is analyzed.

Any remaining metal is poured off and the cup surface is cleaned with a Superwool blanket pad. The cup is air cooled and sectioned through the center (along the short axis) to assess degree of metal attack. The initial and final chemical analyses of the alloy are compared to determine pickup of silicon and iron.

**Method 3: Sample preparation and test procedure**

Samples are prepared according to the supplier’s recommendations and cast into the same molds as used as Method 1. Following the same setting, curing, and drying process, half of the dried sample cups are prefired to 1470°F for five hours and half to 250°F.

Four test pieces are heated simultaneously in an electric furnace alongside a quantity of the test alloy in a crucible at 50°F/min to 1472°F ±40°F. A 160-g sample of pure aluminum (>99.8%) is ladled into the sample hole, and the cups are held at 1470°F for 72 hours. The melt is stirred daily to break the oxide film formed and afterwards is left to cool naturally in the furnace. It is cut diagonally and the cut face is inspected for penetration and reaction with metal and photographed.
Test results

Three monolithic materials shown in Table 1 were tested using the three cup-test methods to assess how the different test conditions used by the aluminum producers affect the outcome of the test results.

As shown in Fig. 6, none of the materials tested using Method 1 show any significant corundum growth, as would be expected, because all three materials are routinely used in aluminum furnaces. Material C, which was prefired to 2190°F, shows a thin layer of corundum formed at the interface with the metal, which suggests that corundum resistance begins to degrade as firing temperature increases. This behavior would have performance implications in service when furnaces are operated more aggressively.

The results of Method 2 show no corundum growth on any sample at lower temperatures of 1500°F, despite roughening of the contact surface to try to promote reaction. However, the alloy analysis reveals that silicon pick-up increases going from material A to B to C. Cup-test failures are normally accompanied by increased concentration of silicon and iron in the alloy after testing.

The trend in increasing silicon pick up matches the reduction in alumina/silica ratio in the material; as silica content increases, more Si is detected in the alloy (Table 2).

Despite the low testing temperature of Method 2, material C is close to the failure threshold for maximum allowable silicon pick up of 0.5%.

As with Method 2, the results of Method 3 show no visible signs of corundum growth on any sample (Fig. 8). The results indicate that testing at 1830°F accelerates the corundum reaction and that prefiring the sample at higher temperatures can cause the nonwetting additive to react with other material constituents and to lose its effectiveness.

As all the materials studied are already in use in many furnaces, one would expect them to pass these cup tests; for most test conditions studied, this was the case. However, as the severity of the test conditions increases, more metal/refractory interaction was observed, specifically in material C.

This matches general operational observations where it was noted that material C starts to suffer from corundum growth in more aggressively run furnaces. According to these laboratory tests, metal contact performance appears to start deteriorating as temperature increases to 1830°F.

In the past, such high test temperatures were considered unrealistic as holding temperatures tended to be well below this level. However, in more recent times, as aluminum furnaces continue to be pushed harder, chamber temperatures have risen and conditions have become more aggressive for the refractory lining. Therefore, test conditions that accelerate the reactions involved, by increasing temperature above traditional aluminum holding temperatures, are now more valid.
In particular, corundum growth is often seen to start at hot spots in the furnace, where temperatures can be measured in excess of 1830°F. This situation is exacerbated by exothermic reactions from salt and dross buildup on the lining. As industry needs have changed, so the furnace environment has changed, and, therefore, the material test methods need to evolve to reflect this.

In light of modern aluminum test practices, the testing temperatures of Methods 2 and 3 appear too low, as they do not accelerate corundum growth reactions adequately. Additionally, the high melt-surface area in Method 2 promotes excessive dross formation and volatilization. Methods 1 and 3 use relatively small alloy samples, which also suffer from volatilization, but this can be controlled to improve test repeatability by covering the sample cup with a refractory lid of test material.

Cup test results are further complicated when salts are introduced into the metal contact cup tests. These studies show that resistance to corundum growth can alter considerably in the presence of salts, and further investigation on this subject is being carried out.

**Conclusions**

The metal contact cup test methods used by three aluminum producers for furnace lining selection were investigated using monolithic materials currently in use in several melt-hold furnaces around the world.

Aluminum producers are increasing productivity to remain competitive. This is normally achieved by increasing heat input to the furnace using more powerful burners to melt the metal faster. However, this leads to increased metal losses as a result of surface oxidation and to larger heat gradients across the metal, leading to segregation of alloying elements and a reduction in metal quality.

These effects are countered by increased use of fluxes to suppress surface oxidization and increased stirring of the metal to achieve homogenization. Given the increasingly challenging environment the refractory lining has to work in, aluminum producers must ensure that their material assessment tests also reflect these changes in conditions. Otherwise, the tests will produce unrealistic results and material selection may be compromised.

The results of this investigation suggest that those cup tests using lower temperatures are not aggressive enough for assessing lining materials in today’s furnace environment. In the past, such test conditions were adequate, but the test methods have not evolved in line with the furnace conditions which they are trying to simulate.

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