Methods of assessing monolithic refractories for material selection in aluminium melt-hold furnaces

This article describes test methods used by major aluminium producers to assess and approve monolithic refractories for use in the key working zones of an aluminium-melting furnace. The results are assessed and compared and indicate where certain test techniques do not necessarily represent today's operating conditions, which have changed as manufacturers have worked to increase productivity through, in particular, increasing heat input to the furnace to melt the metal faster.

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

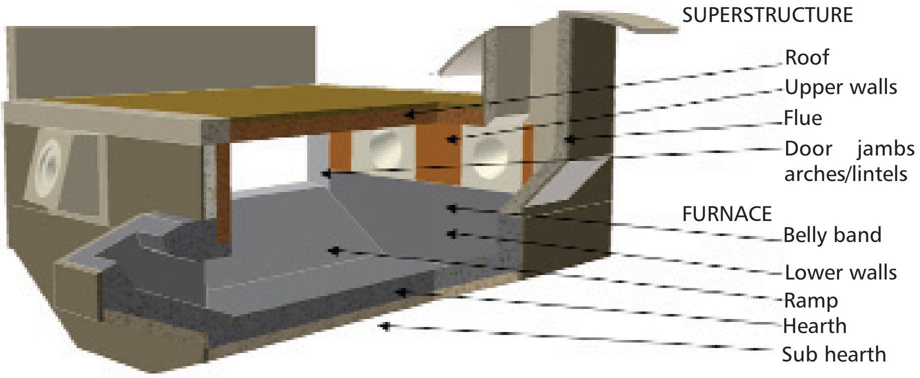

Fig 1 Furnace zones in an aluminium melt-hold reverb furnace

Background

Monolithics, such as those supplied by Morgan Thermal Ceramics, are used to line the metal and non-metal contact regions in typical melt-hold gas fired reverbatory furnaces. Each region is divided into sub regions as illustrated in **Fig 1** and each has a different set of operating conditions and hence 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 methods and frequency of cleaning, mean that diverse physical conditions can influence different parts of the furnace.

The diverse nature of the furnace environment means aluminium producers need to maintain a complex and lengthy testing scheme for furnace linings. This is to subject potential materials to the full range of conditions that they are likely to experience in service. As there is such a vast range of conditions it is not practical or cost effective to test materials for all types and so aluminium producers have developed a practical set of laboratory tests.

The 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 have developed tests to simulate these as part of their approval program.

The investigation of tests in this article focuses on the metal contact region, as this 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 aluminium reacts with free silica in refractories and this transformation leads to a very large expansion in volume, causing severe distortion and cracking of the lining.

$[4Al\ (l) + 3SiO_2\ (s)\ ?\ \ 2Al_2O_3\ (s) + 3Si]$

The most prevalent laboratory test for corundum growth resistance is the aluminium 'cup' test. The objective of this investigation is to understand how different test conditions affect the behaviour of the lining materials by evaluating how existing furnace lining materials behave when subjected to aluminium producers' contact 'cup' test methods.

The tests

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

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*Thermal Ceramics

Fig 2 Mould for sample preparation – method 1

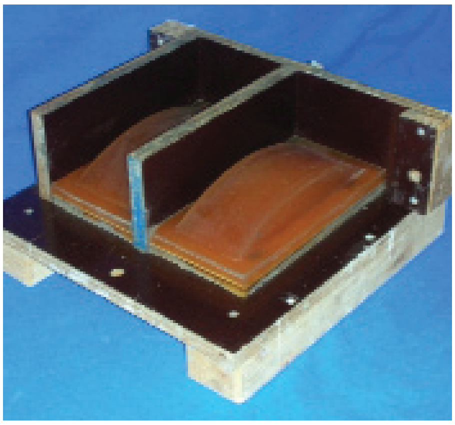

Fig 3 (right) Mould for sample preparation – method 2

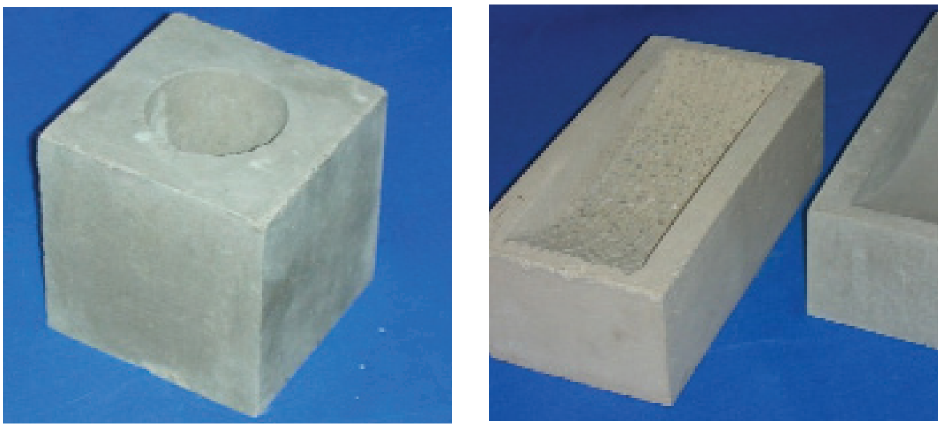

Fig 4 Samples prepared by method 1 (left) and method 2 – as fired (right) and after surface roughing (middle)

Method 1

Sample preparation

A series of 100mm cubes are cast from compositions mixed at standard water addition (mould and cubes can be seen in **Fig 2** and **4**.) Each cube has a 50mm deep, slightly tapered hole (55mm diameter at top, 53mm at base). Samples are set overnight, then de-moulded, cured and dried at 230°F for 18 hours. Half the dried sample cups produced are then pre-fired to 2192°F for five hours. Lids of the same material (25mm thick) are also made to minimise loss of volatiles.

Test procedure

Typically 7075 alloy is used for testing, supplied as 52mm bar and cut to 50mm 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 pre-fired samples are tested at the same time for comparison.

The assembled cups are placed in a kiln, heated to 1832°F at a rate of 302°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 or ease of removal of the aluminium and photographed.

Method 2

Sample preparation

Following the supplier's mixing recommendation, a standard brick size (230mm height x 114mm width x 76mm depth) of the test material is cast into a mould that incorporates a curved face to form a cup shape with a maximum depth of 32mm for holding the alloy. The mould is shown in **Fig 3**. After the recommended curing time, the sample is fired according to the supplier's recommendation to 1499°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.

Test procedure

The cup sample is raised to 1499°F in a furnace at a rate not exceeding 302°F/hr. Meanwhile 7075 alloy is melted in a silicon carbide crucible, heated to 1499°F and sampled for analysis. The molten alloy is then ladled into the brick cavity at 1499°F to about 3mm 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 alloy from the cup is taken for analysis.

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 centre (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

Samples are prepared according to the supplier's recommendations and cast into the same moulds as used as method 1. Following the same setting, curing and drying process, half the dried sample cups are pre-fired to 1472°F for five hours and half to 248°F.

Test procedure

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 ± 41°F. 160g of pure aluminium (>99.8%) is ladled into the sample hole and the cups are held at 1472°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 inspected for penetration and reaction with metal and photographed.

Results

Three monolithic materials as characterised were tested using the three 'cup' test methods to assess how the different test conditions used by the aluminium producers affect the outcome of the test results.

As shown in **Fig 5**, none of the materials tested using method 1 show any significant corundum growth, as would be expected since all three materials are routinely used in aluminium furnaces. Material C, which has been pre-fired to 2192°F, does show a thin layer of corundum formed at the interface with the metal and this suggests that corundum resistance begins to degrade as firing temperature increases. This behaviour would have performance implications in service when furnaces are operated more aggressively.

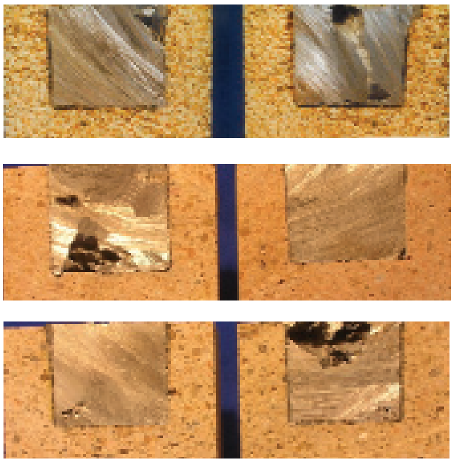

Fig 5 Materials A, B and C tested by method 1 – dried (left) and pre-fired to 2192°F (right)

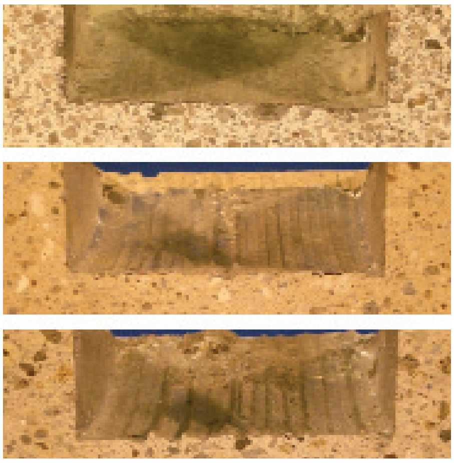

Fig 6 Materials A, B and C tested by method 2

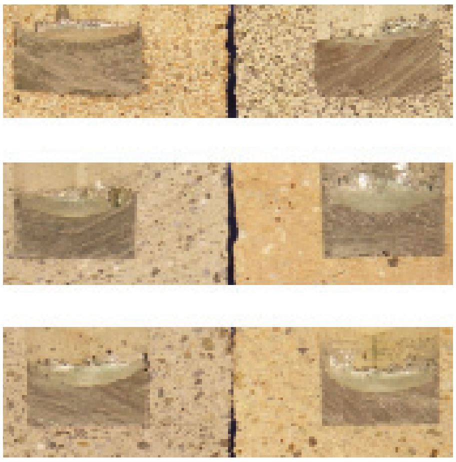

Fig 7 Materials A, B and C tested by method 3 – pre-fired to 1472°F (left) and pre-fired to 2195°F (right)

The results of method 2 as seen in **Fig 6** show no corundum growth on any sample at lower temperatures of 1499°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 (as shown in **Fig 8**) 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 and as silica content increases, more Si is detected in the alloy. 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. The results indicate that testing at 1832°F accelerates the corundum reaction and that pre-firing the sample at higher temperatures can cause the non-wetting additive to react with other material constituents and to lose its effectiveness.

As all the materials studied are already in use in many furnaces, we would expect that all the materials tested would pass these cup tests and for most test conditions studied that has been observed. However, as the severity of the test conditions increases, more metal/refractory interaction has been observed, specifically in material C.

This matches general operational observations where it has been 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 1832°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 aluminium 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 aluminium holding temperatures, are now more valid.

Fig 8 Sub-hearth Gunning material tested by method 2

In particular, corundum growth is often seen to start at hot spots in the furnace, where temperatures can be measured in excess of 1832°F. This situation is exacerbated by exothermic reactions from salt and dross build-up 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 aluminium test practices, the testing temperatures of methods 2 & 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 volatilisation. Methods 1 and 3 use relatively small alloy samples, which also suffer from volatilisation, 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 have shown that resistance to corundum growth can alter considerably in the presence of salts and further investigation on this subject is being carried out.

Conclusion

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

Aluminium producers have worked to increase 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 oxidisation and increased stirring of the metal to achieve homogenisation. Given the increasingly challenging environment the refractory lining has to work in, aluminium 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. ■