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Thermal Shock Testing of Monolithic Refractories

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ABSTRACT
A simple technique for evaluating the thermal shock resistance of monolithic refractories at laboratory scale was implemented ad-hoc. The aim of this contribution is to describe the main features of this technique. The tested material is a monolithic refractory commonly used in electric arc furnace roofs of the steelmaking industry. The proposed methodology is based on the air-cooling of cylindrical specimens and the determination of their residual mechanical resistance by diametrical compression tests.

1 Introduction

The thermal shock resistance of refractory materials can be determined by means of standard tests. The mostly used standards are ASTM C 1100-88 [1] and C 1171-91 [2] for bricks and monolithic materials, respectively. In order to produce thermal shocks, blowers insufflate air being heated with a line burner, on the hot face of sample bars (23 cm and 15 cm in length for bricks and monolithic materials, respectively). This procedure can be repeated several times. The equipment which is needed for these operations commonly is used in industrial laboratories. The degree of damage is evaluated by measuring the before- and after-shock modulus of elasticity, sonic velocity, or modulus of rupture (MOR).

The water quenching test that is also standardized [3] is the most frequently used test at laboratory scale in the evaluation of the thermal shock resistance of bricks and monolithic refractory materials. Since water has a heat transfer coefficient value (h) being approximately 100 times higher in comparison to air, it provides a more severe quench-

ing medium which implies some additional disadvantages [4–8].

Although these tests are useful for materials selection and comparison, they show some limitations when a more basic analysis of the thermal shock behavior of refractory materials is required. The thermal shock resistance depends on a variety of variables, among them the shape and size of the specimen and, especially, the heat transfer conditions between the quenching medium and the specimen. This in turn depends on several factors (geometry and surface finish of the specimen, temperature, quenching medium) [4–10]. This contributes to the fact that even normalized results of testing cannot directly extrapolated to service conditions.

The roof of electric arc furnaces (EAF) is opened for metallic charge loading at least one time per heat during its campaign. Special refractory materials are needed in the central part of the roof (delta), which is in close contact with the heating electrodes. These refractory materials should withstand high temperatures and permanent thermal cycling between 700–800 °C and up to 1,600 °C. For this reason, wear and damage by thermal shock usually is observed in these materials during operation.

In this contribution, a simple methodology was designed ad-hoc for the laboratory eval-

uation of the thermal shock resistance of monolithic refractories used in the construction of EAF roof taking into account the material's specific conditions of use.

2 Experimental

The material under consideration is a commercial monolithic refractory material that is used for the construction of EAF roof in the steelmaking industry. It is an alumina/chromium based material with hydraulic bond and organic fibres.

For the evaluation of its thermal shock behaviour at laboratory scale, a method that reproduces the operation conditions of the material as near as possible was developed. It includes the application of a controlled thermal shock which can be repeated several times followed by the quantification of the damage by means of the measurement of material's properties being sensitive to this type of degradation (destructive and non-destructive techniques). Preliminary trials to determine the appropriate experimental conditions (quenching medium and quenching time, specimen geometry and method for damage evaluation, among others) were required.

Keeping in mind the service conditions of the studied concrete, thermal shock by cooling was selected with an initial specimen temperature fixed at 1,600 °C according to

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Fig. 1 • Thermal shock test for air-quenched samples
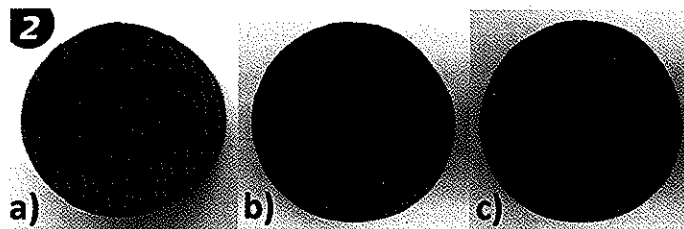

Fig. 2 • Fired samples: a) before thermal shock; b) quenched in air; c) quenched in water

the EAF operation temperature. For the thermal treatment, an electric furnace with heating elements consisting of silicon carbide was used. In order to minimize the transformations occurring in the material during the furnace heating (sintering and/or chemical reactions [11–12]), the specimens were inserted into the furnace when the furnace temperature (1,600 °C) has stabilized. The samples were placed into the furnace separately, but in only one furnace opening. The same procedure was followed when the cylinders were removed from the furnace to the quenching medium.

At first, two quenching media of different severity were selected: air and water. Both media were used at room temperature.

These media were selected because they are commonly used in thermal shock testing of refractory materials and easily available, and they have no operative inconveniences such as manipulation, environmental, or toxicity risks. Keeping in mind the aim of reproducing the thermal shock conditions in service as near as possible, the temperature change in the specimen tested in air was registered with a K thermocouple connected to a digital thermometer.

The possibility to apply thermal shock cycles before evaluating damage was considered. A total of 5 cycles with a dwell time in the quenching fluid of 5 min was selected.

Two methods were considered for damage evaluation: firstly the qualitative evaluation of cracks by ocular inspection, and secondly the determination of residual mechanical strength by measurement of before- and after-shock values.

In the first instance, the inspection using an unaided eye was evaluated, keeping in mind the possibility of using a magnification device (glass or microscopy) in the case of being insufficient.

For the determination of the mechanical resistance, a test with a low dependence on the directionality of damage is desirable in order to be effective as a quantification method. For this reason, the flexure of bars was discarded due to its strong dependence on the orientation of damage with regard to that of the applied load. On this sense, diametral compression of cylinders [13] was considered as a more suitable mechanical test. Although the crack orientation effect could not be completely discarded in this test, it was considered that the randomness of the load application line on the lateral face of the cylinder minimized this effect. Furthermore, this configuration allows the use of specimens with aspect ratio around 1, conversely to the flexure of bars. This favors the homogeneity of:

a) the temperature distribution of the cylinder in the furnace, and

b) the thermal stresses generated during quenching.

On the other hand, this test has the advantage of requiring a smaller contact area between the specimen and the compression platens with regard to the uniaxial compression. In turn, this reduces the negative effects of the contact friction [14]. In diametral compression test, the friction is evenly minimized using a material pad between the specimen and the platens that also favors the appropriate distribution of the applied load [13].

A servo-hydraulic universal testing machine (Instron 8501) with high stiffness load frame was used. The tests were carried out in air at room temperature using steel loading platens. MoS_2 paste was placed between the specimen and the hard platens as a material pad. The tests were carried out in displacement control and previous trials were performed to determine a suitable rate. A value of 0.2 mm/min was settled down in order to minimize subcritical crack growth effects and to achieve an appropriate initial accommodation of the system.

The dimensions of the cylindrical specimens were selected taking into account the maximum level of the load system and the mechanical resistance of the studied material reported in the materials data sheet. A diameter (D) of 30 mm and a thickness (L) of 25 mm (aspect ratio of D/L = 1.2) were established.

For the analyzed material, green bodies were prepared by ramming of material-water mixtures in an amount that was selected under consideration of

1) the technical data sheet,

2) the amount used in the plant, and

3) the obtainment of an appropriate consistency of the mixture for the application of the forming technique at laboratory scale.

After forming, cylinders were thermally treated for curing and firing. In both cases, the thermal cycles were designed taking into account the procedure used to prepare and to install the material in plant. Curing was carried out in a range of temperatures between 100 and 200 °C and firing was performed at a temperature of 1,400 °C. This last value is in the range of temperatures used in the plant at the different stages of preparation, installation, and operation of the material (between 600 and 1,600 °C).

3 Results and discussion

In Figure 1, the air-quenching of the specimens is presented. Figure 2 shows the visual aspect of the fired samples before and after the thermal shock test.

The quenching in air was carried out by removing the cylinders from the furnace to
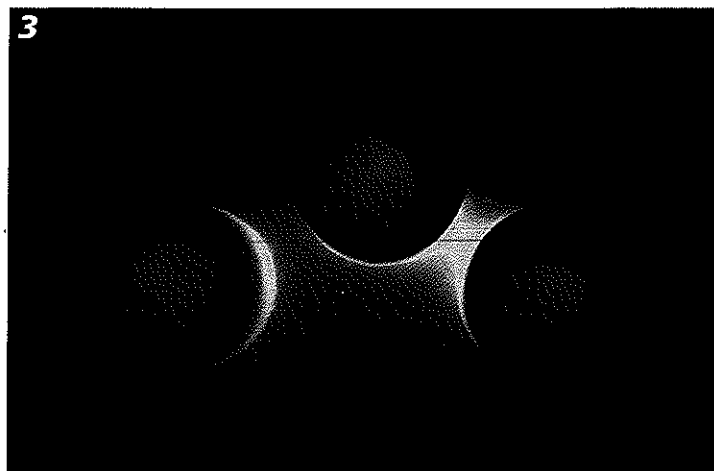
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Fig. 3 • Refractory plate holding samples just after their removal from the furnace (air)

the laboratory atmosphere at a temperature of 21–24 °C. For water quenching, an aluminum vessel containing nearly 3 liters of the liquid at a temperature of 18 °C was used. The specimens were submerged into the fluid immediately after taking them off from the furnace.

In the air quench test, the specimen temperature decreased to 1,000 °C in a very short time interval (<<1 min), and reached a value of nearly 700 °C after a time period of 1 min. Although the specimen temperature was not registered in water (due to experimental drawbacks), it was assumed that the rate of decreasing the temperature in water was much higher than in air due to the larger heat transfer coefficient in the liquid. A boiling of water was observed in the proximity of the specimens in the first seconds after being introduced into the fluid. It has been reported that this effect can vary the rate of heat transfer locally in more than one order of magnitude [6]. As a consequence, the water quench test conditions become difficult to control and to reproduce. Keeping in mind the difficulty in the control of the rate of heat transfer in water, and the actual thermal variations of the material in service, air was considered a more suitable medium than water in order to reproduce results and to correlate them with in-service conditions.

After 5 thermal cycles (5 consecutive thermal shocks under equal conditions), it was possible to observe macroscopic cracks by naked eye in every surface (plane and lateral) in longitudinal and traverse directions. The degree of damage was visibly higher in the cylinders quenched in water than in the air-cooled ones. The ocular inspection also allowed an identification of differences in cracks depth among specimens which depend on the order of removal from the furnace. The first removed cylinders exhibited deeper cracks than the later quenched ones.

This fact was attributed to the diminution of the initial temperature of the specimens still located in the furnace as a consequence of the temperature decrease into the chamber. Therefore, the thermal shock of these cylinders was less severe.

The obtained results showed that the ocular inspection by naked eye was an appropriate method for qualitative evaluation of thermal shock damages. Furthermore, the results evidenced that the specimens should be introduced in the furnace at the same time in one furnace opening in order to be tested under the same experimental conditions. The removal of the specimens from the furnace should be done in the same way.

Considering the mechanical results, fired specimens without thermal shock exhibited diametral compression strength of 5.0 ± 0.3 MPa. Although this magnitude was low, it was clearly higher than those values corresponding to samples damaged by thermal shock in both quenching media. The cylinders cooled in air retained 64 % of the initial resistance (3.2 ± 1.1 MPa) while those quenched in water retained only 52 % (2.6 ± 1.3 MPa) of its original value. Additionally, an increment of the mechanical strength scattering was observed when the specimens were subjected to thermal shocks. This increase was expected due to the interaction between thermal shock cracks, randomly oriented, and the applied load in the diametric compression test. However, the relative order of the individual values correlated with the order of introduction and removal of the test specimens from the furnace. Because of this, differences in the values of the mechanical resistance among cylinders quenched in the same medium but removed at different times were evident, as was previously observed by ocular inspection.

In this way, the sensibility of the residual mechanical strength measured by diametral compression for the quantification of thermal shock damage was inferred from the obtained results, and the need to modify the way of introduction/extraction of the specimens was confirmed.

To overcome the latter problem, the employment of a plate to support the cylinders and to place them simultaneously into the furnace (minimizing the furnace openings), was introduced (Figure 3). Due to the severity of temperature changes involved in the introduction and the extraction of specimens (somewhat lower than 1,600 °C), several materials were evaluated. Silica refractory was the most suitable material since the plate did not exhibit thermal spalling. A second set of trials (three samples per plate) introducing this improvement showed a reduction of mechanical strength data scattering: fired specimens (without thermal shock) = 4.7 ± 0.2 MPa, cylinders cooled in air: 70 % of the initial resistance (3.3 ± 0.8 MPa), cylinders quenched in water: 53 % of the original value (2.5 ± 0.5 MPa).

Based on the results mentioned above, a minimum of three specimens (simultaneously subjected to thermal shocks using the plate support) was required to obtain reliable data.

With respect to the failure mode of the cylinders in diametral compression tests, an effect of the thermal shock cracks pattern was observed. In fired specimens without thermal shock, the fracture occurred through a central crack running diametrically, just as it is usually reported for this configuration [13]. However, in some of the cylinders subjected to thermal shock, this crack was displaced from the central line in some cases. Moreover, secondary cracks were observed [13] generally parallel to the central one, although some of them exhibited different orientation.

Keeping in mind the results described above, the conditions for the thermal shock testing of the concrete under study were established as follow.

- Geometry of specimens: cylinders of aspect ratio of nearly 1, whose preparation conditions have to be consistent with those used in a plant.
- Initial temperature of specimens: selection based on the maximum temperature supported by the material in service. A statistical number of specimens (at least 3) placed on a plate support (silica refractory could be used for the plate) have to be introduced simultaneously into the furnace previously stabilized at the desired temperature.

- Quenching medium: air at room temperature, with a dwell time in air of 1–2 min and a variable number of cycles (tested: 5 cycles); the exposure to quenching medium has to be carried out by removing the specimens (supported on the plate) from the furnace to the laboratory environment.
- Damage evaluation: performed after determining the number of established cycles of thermal shock by:
  a) visual inspection with the naked eye to observe pattern and depth of cracks, and
  b) residual mechanical strength determined by diametral compression.
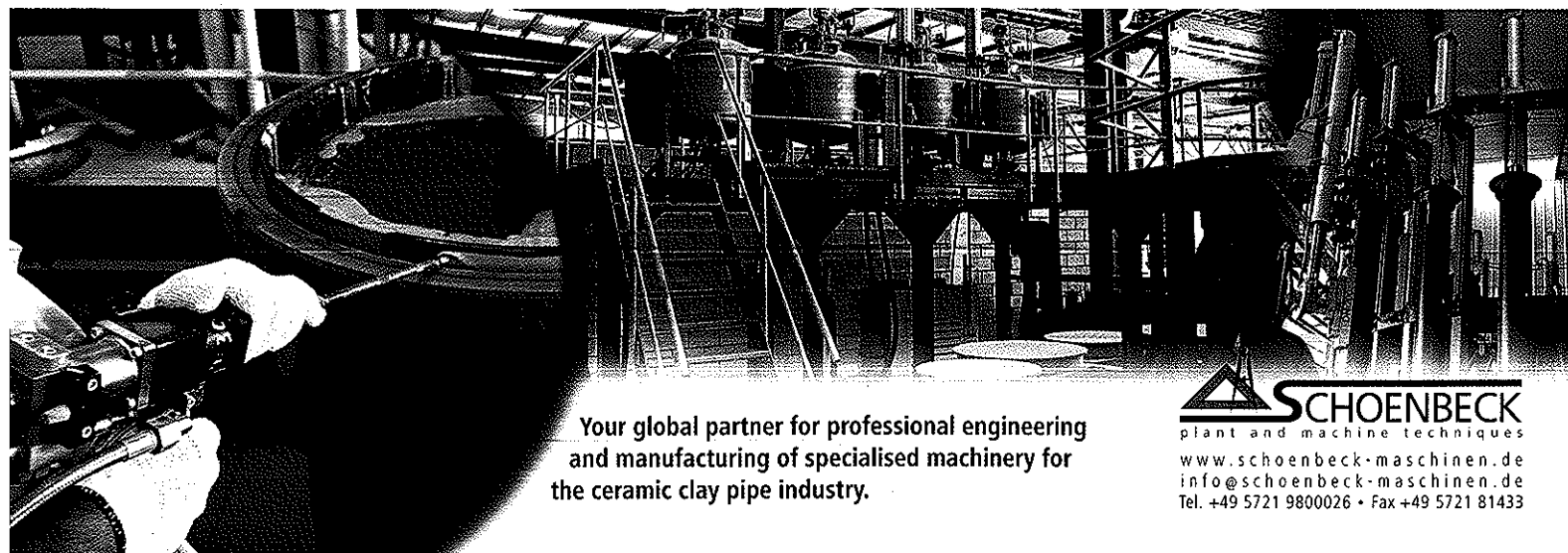
4 Conclusions

The methodology described in this contribution is a simple tool to evaluate the thermal shock resistance of a monolithic material simulating the in-service conditions of preparation, installation and operation as close as possible. Moreover, its application field can be extended to the evaluation of other monolithic materials of similar characteristics (i.e. bond type and granulometric parameters), introducing pertinent modifications (initial temperature, number of cycles, specimen dimensions).

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