# LABORATORY AND INDUSTRIAL TESTING OF SILICA BRICKS FOR COKE OVENS

**M. A. Camerucci, A. G. Tomba M., A. L. Cavalieri[(1)]**
**M. de Córdova, D. Beltrán, R. Topolevsky[(2)]**

[(1)] Instituto de Investigaciones en Ciencia y Tecnología de Materiales (INTEMA)–Fac. Ingeniería, UNMdP/CONICET. Av. J. B. Justo 4302 (7600) Mar del Plata, Argentina.
[(2)] SIDERAR S.A.I.C.-Centro Siderúrgico Gral. Savio. P.O. Box 801 (2900) San Nicolás, Argentina.

## ABSTRACT

In order to determine the feasibility of reconstructing coke oven walls in a local steel mill, a set of laboratory and industrial tests was carried out on silica KN bricks. Two types of bricks that are currently used in oven construction were analyzed, with one being less expensive than the other. In addition, 25 year-old original oven bricks were also studied. The results of these laboratory tests were analyzed taking into account the requirements of DIN 1089 standards. Finally, a cost evaluation was carried out.

## INTRODUCTION

Because of its special thermal expansion behavior, excellent refractoriness, and chemical compatibility, one of the most common applications for silica bricks is in steelmaking coke ovens. Different qualities, KN, KD and KS, are standardized in DIN 1089 [1], which specifies their most important characteristics, admissible dimensional tolerances, and external appearance. This standard is accepted and applied in numerous coking plants.

Due to the deterioration of coke oven walls in some batteries in a local steel mill, a plan was developed to reconstruct the headers. As part of this plan, many wall headers were reconstructed with the same type of brick originally used for the oven's construction (original bricks[1]). An alternative supplier of KN silica bricks (alternative bricks[2]) with much lower prices was discovered. Keeping in mind the high cost of the original bricks, several laboratory tests were carried out in order to analyze the possibility of interchanging both types of bricks. Residual quartz content, cold crushing strength, apparent porosity, refractory under load, and creep in compression were considered. DIN 1089 standards [1], which set forth the specifications of silica bricks for coke ovens, were used for comparison.

In addition, original 25 year-old brick samples from decommissioned coke oven walls were studied to evaluate both the degradation of the brick properties and their compatibility with the alternative bricks. The brick properties evaluated were residual quartz content, apparent density, and cold crushing strength.

Furthermore, due to the positive results of laboratory tests, alternative bricks were purchased to reconstruct some of the wall headers. Two adjoining coke oven walls were reconstructed with original and alternative bricks respectively. In order to compare the behavior of both types of bricks during the reconstruction, start-up, and normal operation, the walls were tested and monitored during normal industrial activity for the following: the thermal evolution during the initial, post-repair heating, the expansion of the ovens' structure and the air-tightness of the walls both during start up and the ensuing months.

The aim of this work is to report the laboratory and industrial results obtained as well as the economic impact of using alternative KN bricks in the reconstruction of the coke oven walls.

[1] Otto, Germany
[2] Dengfeng Chunsheng Refractories Co. Ltd., China

## LABORATORY TESTS

The laboratory tests were performed in the Structural Ceramics Laboratory (INTEMA) [2] at the Engineering School, National University of Mar del Plata (Mar del Plata, Argentina). Some results are reported in **Table 1**: residual quartz content (%$Q_R$); cold crushing strength (CCS); apparent density ($\rho_a$); and apparent porosity ($\pi_a$). Alternative bricks were also tested in the Luoyang Institute of Refractories Research (LIRR) (Luoyang, China) in order to double-check the results.

### Residual quartz content (% $Q_R$)

The percentages of residual quartz in bricks shown in **Table 1** were determined using a spiking X-ray diffractometric method [3-5]. The unused original bricks exhibited higher %$Q_R$ than the alternative ones. Moreover, they showed a noticeable decrease in value after 25 years of operation [6]. The value of residual quartz content reported by the reference laboratory (unspecified method) for alternative bricks, 0.0 wt%, was close to that determined at INTEMA.

**Table 1. Results of laboratory tests**

| Brick | %$Q_R$ (wt%) | CCS (MPa) | $\rho_a$ (g/cm$^3$) | $\pi_a$ (%) |
|---|---|---|---|---|
| Original, unused | 3.50 | 34 | 1.79 | 22.96 |
| Alternative, unused | 0.50 | 66 | 1.84 | 20.48 |
| Original, used[a], PS-oven side | 1.44 | 52 | 1.80 | 17.90 |
| Original, used[a], PS-burner side | 0.00 | 91 | 1.90 | 17.00 |
| Original, used[a], CS-oven side | 1.71 | 66 | 1.90 | 17.60 |
| Original, used[a], CS-burner side | 0.12 | 41 | 1.80 | 19.80 |

[a] after 25 years

The residual quartz content of unused silica bricks indicates the degree of phase transformation during their production due to thermal effects. It affects the expansion behavior of the body at temperatures higher than 1300°C. A $\%Q_R$ >6 wt% produces dangerous contraction-expansion in post-expansion behavior [7-9]. A content <6 wt% of residual quartz is specified in the DIN 1089 standards for KN silica bricks. For practical purposes, contents between 2 and 3 wt% are recommended in the literature [7-9]. The unused original bricks, the used original bricks and the alternative unused bricks analyzed met this standard. In original bricks, the decrease of $\%Q_R$ in used refractories with respect to the value of the unused ones (from 3.50 to 0-1.71 wt%) was mainly attributed to silica phase transformations, depending on both the contamination and the raw materials impurities, due to the high temperatures and lengthy times in service.

## Cold crushing strength (CCS)

Cold crushing strength was determined according to DIN 51067 (EN 993-5) standards [10] and the obtained values are reported in **Table 1**. The mechanical resistance of the alternative bricks was discovered to be higher than the original bricks. Moreover, an increment in the CCS values of these last bricks after 25 years of operation [6] was recorded. The mechanical strength value for the alternative bricks agreed with that obtained in the reference laboratory: 76 MPa.

The cold crushing strength is an important mechanical parameter because it affects impact resistance during normal operation. DIN 1089 standards establish a minimum strength of 28 MPa as an acceptable value for silica bricks in coke ovens. The mechanical resistance of every tested brick was higher than this limit.

## Apparent density ($\rho_a$) and apparent porosity ($\pi_a$)

The determination of apparent densities and porosities was carried out according to DIN 51067 part 1 (EN 993-5) standards [11] and the results are shown in **Table 1**. The reference values (LIRR) for apparent density and porosity for alternative silica bricks, 1.88 g/cm$^3$ and 18.00% respectively, were close to those determined by INTEMA. The $\rho_a$ value of original bricks was lower than that of the alternative ones. An increase in the apparent density was registered for original bricks after 25 years of use. Similarly, a higher apparent porosity in the original bricks with respect to the alternative ones was also discovered, with original brick porosity decreasing in relation to the amount of years in service.

The values of apparent densities and porosities, such as the $\%Q_R$, help evaluate several features: degree of brick transformation during production, post-expansion under load, and thermal conductivity. Literature in the field [12] report a correlation between these brick parameters; for example, there is a correlation between a very good degree of transformation and an apparent density <2.35 g/cm$^3$, a $\%Q_R$ <6 and a dilatation (T >1000°C) post-expansion <1%. On the other hand, DIN 1089 standards give the following minimum values for KN silica bricks: apparent density <2.35 g/cm$^3$, and apparent porosity <24.5%. All the bricks studied, including those used for 25 years, showed values lower than the standard limits for good performance, and agreed with the correlation above mentioned.

In addition, it was verified that the densities obtained for unused silica bricks were within the allowed range in relation to thermal conductivity for KN bricks [7], while those of the original used bricks were slightly higher than the allowed range.

The relationship between the mechanical resistance and the apparent porosity [6] is plotted in **Figure 1**. It agrees with previously reported data for similar coke oven silica bricks [13].

## Refractoriness under load (RUL)

The refractoriness under load (RUL) of unused original and alternative bricks was determined by compression based on DIN 51064 standards [14]. A modification to the maximum temperature (1450°C) and an indirect determination of the sample's dimensional variations from the actuator displacement [15], were employed. The deformation values determined (**Table 2**) have comparative validity.

**Table 2. Refractoriness under load (RUL)**

| Unused bricks | Maximum Expansion (temperature) | Contraction at 1450°C |
|---|---|---|
| Original | 0.70% (938°C) | 0.26% |
| Alternative | 0.79% (789°C) | 0.28% |

Both types of bricks showed similar behavior although the contraction of the alternative bricks began at a lower temperature.

Another parameter, the value of the equivalent pyrometric cone (EPC), was used to determine the refractoriness of the bricks. Even though this parameter is not included in DIN 1089 standards for silica bricks KN, it is part of Japanese JIS specifications and Chinese standards. Equivalent pyrometric cones of samples obtained from the powders of ground bricks were determined according to IRAM 12505 standards [16] using Orton patterns. The same EPC value was obtained for both the unused original and alternative bricks: >31 (1680°C). These values were near to those of JIS standards (>32) and Chinese standards (1690°C), and they were also in agreement with the findings from the RUL mechanical tests.

The reference laboratory (LIRR) followed the YB/T 370-1995 standard [17] to carry out RUL tests (stress: 0.2 MPa, deformation: 0.2%, cylindrical specimen: 36 mm in diameter x 50 mm in height) on the alternative bricks. A value of 1700°C was obtained according to standard admitted values: ≥1700°C (YB/T 370-1995) and >1640°C (DIN 51064).

The RUL test evaluates the softening of the bricks in conditions of increasing temperature and constant mechanical loading. The result is

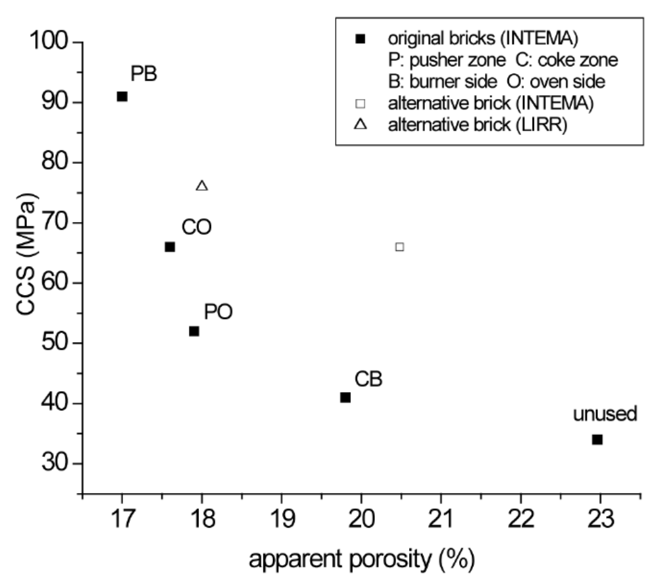


**Figure 1.**

affected by the content and distribution of low melting point phases. The reference laboratory (LIRR) measured a content of low melting point agents 3 wt%, lower than that specified in DIN 1089 standards (<4.25%).

## Creep in compression

Creep test evaluates the permanent deformation of a refractory subjected to constant high temperature and compression during long time. Tests of creep in compression for both unused original and alternative bricks were carried out (INTEMA) based on DIN 51053-2 (EN 993-9) standards [18], but with modifications particular to the local steelmaking industry; the deformation values therefore had validity only for comparative aims. The results obtained are shown in **Table 3**, reported as [18]:

- Z (5 -25): deformation between 5 and 25 hours
- Z (25): deformation at 25 hours

**Table 3. Creep in compression**

| Unused bricks | Z (5-25) | Z (25) |
|---|---|---|
| Original | 0.42% | 0.48% |
| Alternative | 0.10% | 0.12% |

The results obtained showed better creep behavior in the alternative unused brick compared to the original unused one, with the latter showing higher permanent deformation.

The reference laboratory (LIRR) used the same standard without modifications to measure creep in compression of the alternative bricks and obtained the following values: Z (5-25) = 0.05% and Z (25) = 0.33%. These results were somewhat different in relation to those measured on similar alternative bricks at INTEMA laboratory. In both cases, this type of brick exhibited values lower than those specified by the DIN 1089 [1] standards: Z (5-25) <0.12% and Z (25) <0.35%, while the values of the original bricks were higher.

## INDUSTRIAL TESTS

The reconstruction of one wall (W1) with alternative silica bricks and an adjoining wall (W2) with original silica bricks, up to the sixth flue of both the pusher and coke oven zones were carried out. The behavior of both walls was evaluated by means of: a) its evolution during the initial post-repair heating, b) the expansion of the structure, and c) the airtightness of the walls during start-up and the ensuing seven months. A relative cost analysis was also performed.

## Initial heating

After rebuilding walls W1 and W2, an auxiliary burner was installed in both oven zones (pusher and coke) to carry out the initial heating. Afterwards, thermocouples were placed in the upper flues of the reconstructed area. Temperature values were registered every 4 hours during the entire heating period (5 days).

In **Figure 2** the heating curves of both the pusher and coke oven zones are plotted. As one can see, both walls (W1 and W2) evolved similarly, and in accordance with the standard curve. This corresponds to the relationship between the apparent density and the thermal conductivity.

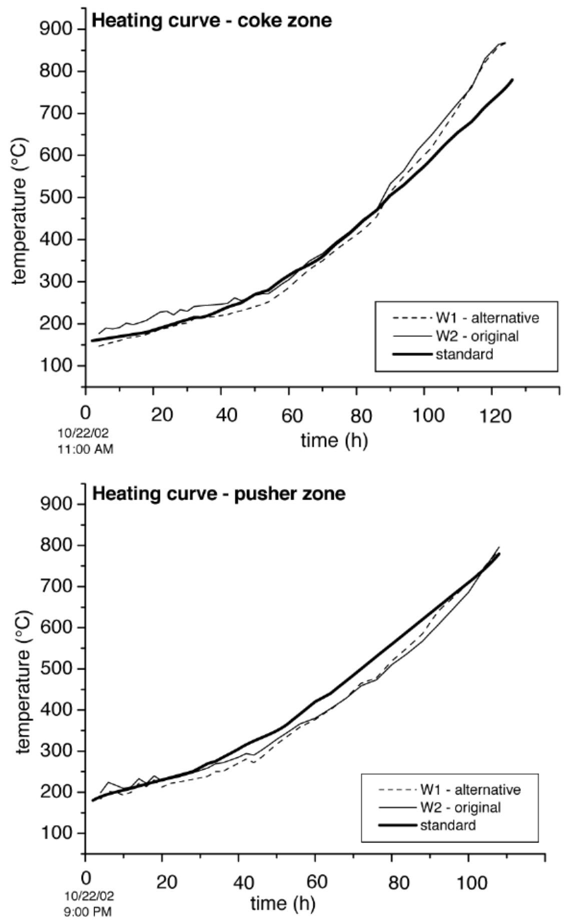


Figure 2.

## Expansion during heating

The distance between the reconstructed wall end and a reference thread soldered to the external face of an "L" support was taken as the "cold measurement". Expansion was then measured once per day during the entire heating start-up process (**Figure 3**). It had to reach a value of 40 mm as defined by the oven's design.

A slightly smaller expansion of wall W1, reconstructed using the alternative bricks, was measured in both the pusher and coke oven zones. This fact agreed with laboratory results based on a dilatometric analysis of the unused original and alternative bricks up to 1400°C [19] and the results of RUL tests (INTEMA).

## Airtightness of the walls

This property was measured using the crude gas filtration index from the oven to the combustion chambers which was determined by visual inspection and empirical calculations following a method developed by NSC (Nippon Steel Corporation) and applied in many coke ovens [20]. **Figure 4** shows the filtration index of both ovens, 1 and 2, that share the wall W2 (original bricks). W1 and W2 belong to oven 1 while both walls of oven 2 are constructed with original bricks.

The filtration index values were discovered to be high and outside of the acceptable limit ("objective" line in graph) for the

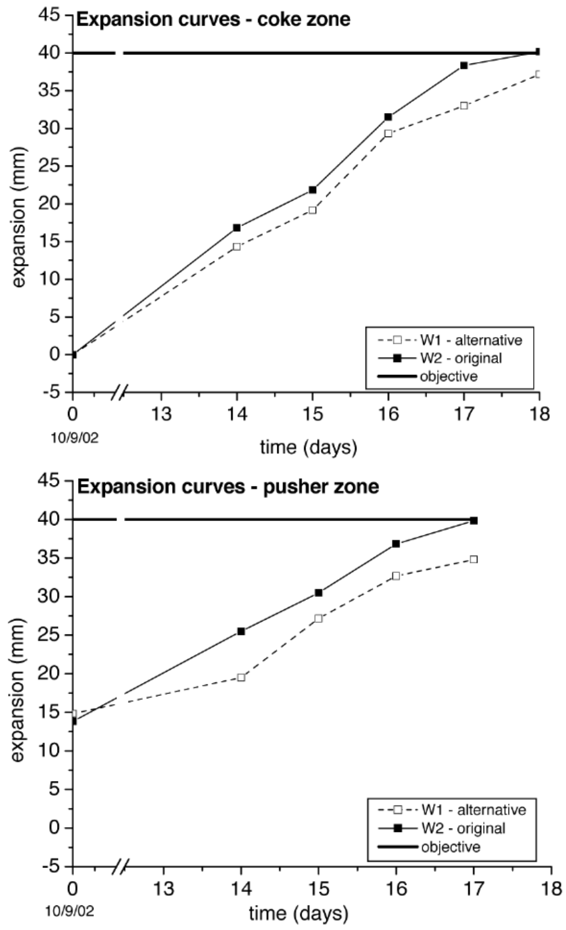


**Figure 3.**

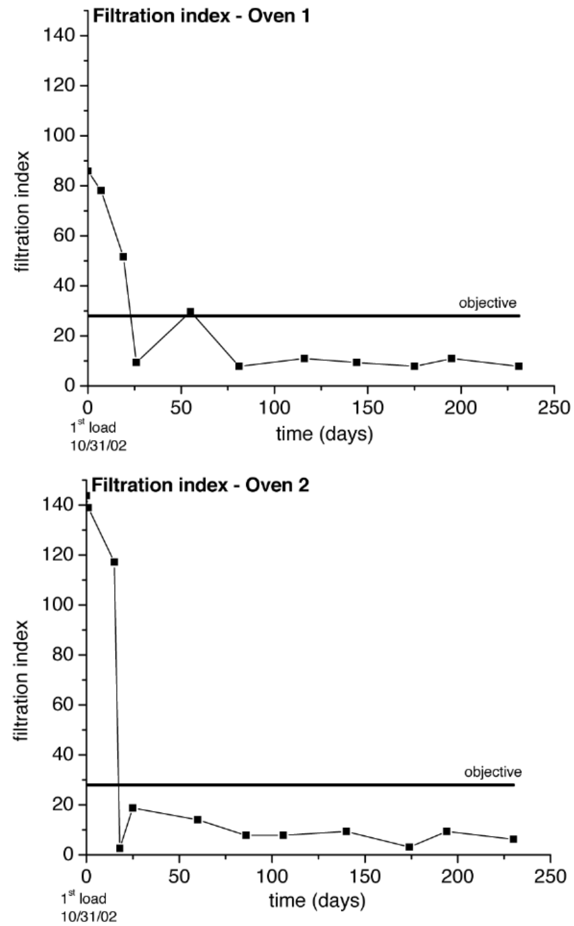


**Figure 4.**

first loads of both ovens, but normalized within three months of operation. The filtration index for the reconstructed wall headers of empty ovens was high in the first loads because the wall lost the graphite layer that provided appropriate airtightness during normal operation. Their values normalized after the third or fourth loads when the wall was under normal operating conditions. The similar results plotted in **Figure 4** for both ovens 1 and 2 indicate that the filtration index for the wall headers W1 and W2 (oven 1) showed similar behavior with the headers of the other oven in normal operation (oven 2).

## EVALUATION OF COSTS

The following items were considered in the cost evaluation: bricks, manual labor, auxiliary materials, and equipment.

Costs for manual labor, auxiliary materials and equipment in the reconstruction for walls W1 and W2 are the same for either alternative or original bricks. However, using alternative bricks (W1) instead of original ones (W2) generates a total savings of 54%.

## CONCLUSIONS

According to the results obtained in both laboratories (INTEMA and LIRR), all the bricks—the alternative unused KN silica bricks, as well as the used (25 years) and unused original bricks—satisfied the requirements set forth in the DIN 1089 standards.

The behavior of W1 and W2 wall headers reconstructed using alternative and original bricks respectively was similar during reconstruction, oven start up, and after seven months in operation with regards to the initial heating, expansion, and wall airtightness.

The use of alternative bricks points to a savings of approximately 54% compared to the cost of original bricks.

## REFERENCES

1. DIN 1089 (1970, 1990), "Refractory Building Material, Coke Oven Bricks, Line-Bonded Silica Bricks, Requirements and Test Method".
2. A.G. Tomba M., M.A. Camerucci, A.L. Cavalieri, "Mechanical Testing and Research Laboratory-Ceramics Division at Intema UNMdP/Conicet-Argentina", *RA&N*, **9** [4] 11-13 (2004).
3. G. Goswani, J.D. Panda, P.K. Panigrahy, "Accurate Determination of $SiO_2$-Polymorphs in Silica Refractories by X-ray Diffractometry"; Vol. III, 1549-1558 in Proceedings UNITECR '97, ( New Orleans) 1997.
4. R.L. Snyder, "Materials Science and Technology: Characterization of Materials, Part 1", Ed. R. W. Cahn, P. Haasen, E. J. Kramer, Vol. 2A, (1992).
5. M.F. Muñoz, "Lifetime of Silica Refractory Bricks Used in the Construction of Coke Ovens in Steelmaking", Undergraduate thesis, National University of Mar del Plata, Mar del Plata, Argentina, 2004.
6. M.A. Camerucci, A.G. Tomba Martinez, A.L. Cavalieri, M. de Córdova, R. Topolevsky, "Post-mortem Analysis of Silica Bricks for Coke Ovens", *TARJ*, **25** [3] 189-94 (2005).

7. H.J. Koschlig, C.O. Feuerfest, "Refractory Materials for Modern Large-Capacity Coke Oven Batteries", UNITECR '89, Vol. 1, 244-261 (1989).
8. K.E. Lepere, E. Overkott, R. Beckmann, L. Escher, "Requirements with Respect to Silica Bricks as Building Material for the Next Coke Oven Generation", UNITECR '91, 315-318 (1991).
9. F. Brunk, "Silica Brick For Modern Coke Oven Batteries", Cokemaking Internacional, 2, 37-40 (2000).
10. DIN EN 993-1, "Methods of Test for Dense Shaped Refractory Products, Part 1". (DIN 51067. Part 1), February 1995.
11. DIN EN 993-5, "Methods of Test for Dense Shaped Refractory Products, Part 5: Determination of Cold Crushing Strength", (DIN 51067. Part 1), February 1995.
12. Didier Fuerfest Technick, Issue 1, pg.16-103 (1954).
13. Yuito Narita-Hiroshi Ueda, "Alteration of Silica Bricks in Coke Ovens", *TAJR*, **21** [1], 35-40 (2001).
14. DIN 51064 (translation of German standard), "Testing of Raw Materials and Ceramics. Determination of Hot Resistance in Compression (DFB) in Refractory Bricks", June 1981.
15. P. Méndez, "Characterization and Mechanical Behavior at High Temperature of Refractory Materials for Construction and Repairing of Coke Ovens", Undergraduate thesis, National University of Mar del Plata, Mar del Plata, Argentina (1998).
16. IRAM 12507, "Refractory Materials. Method for the Determination of Equivalent Pyrometric Cone", August 1990 (Argentina).
17. YB/T 370-1995, "Test Method for Refractoriness Under Load of Refractory Products (Non-differential With Rising Temperature", 1995 (China).
18. DIN 51053-2 (EN 993-9), "Method of Testing Dense Shaped Refractory Products-Part 9, Determination of Creep in Compression", January 1973.
19. L. Zamboni, "Dilatometry of Two Samples of Refractory Material of Coke Oven Wall", Technical Report, DYTEMA-UTN, San Nicolas, Argentina (1998).
20. I. Komaki, T. Matsuo, Y. Kogushi, K. Nishimoto, H. Yamamoto, "Coke Oven Diagnosis and Repair Techniques", Ironmaking Conference Proceedings, Volume 44, 595-606 (1985).