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INDUSTRIAL APPLICATION OF A REACTION SINTERED MATERIAL

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Abstract - A reaction sintering process developed earlier with TiO₂ as sintering aid is applied to produce dense mullite-zirconia components. These are embedded as reinforcement heads in refractory concrete blocks for walking beam furnace lining in steel rolling-mill. Physical and mechanical properties of the ceramic material are presented and discussed.

I - INTRODUCTION

Since several years, reaction sintering of zircon and alumina to produce zirconia toughened mullite ceramics has been investigated. Different routes to obtain these materials have been published earlier/1,2,3/, one of those has been developed by utilising reaction sintering aids to simultaneously perform zirconia and mullite formation and complete densification of the material. This process is based on a fundamental study on phase equilibria of the quaternary system ZrO₂-Al₂O₃-SiO₂-TiO₂/4, 5/ and is protected by a patent /6/.

The composition chosen for this application lies in the compatibility plane ZrO₂-Al₂O₃-SiO₂-Al₃TiO₅. The reaction occurring during sintering may be written in molar proportion as 2ZrSiO₄ + 4Al₂O₃ + TiO₂ → 2ZrO₂ + Al₃SiO₁₃ + Al₃TiO₅ and densification proceeds by a liquid phase sintering mechanism at temperatures between 1450 and 1500°C (eutectic point of the quaternary system at 1430°C). On a first step, zircon dissociates into zirconia and a transient liquid phase from which mullite crystallises. Titanium aluminate formation occurs later and the liquid phase
disappears when solid phases reach equilibrium. In this stage the first permanent liquid phase formation rises at 1595° C. Owing to its good thermal shock and high slag chemical resistances, application of this ceramic material as reinforcement head of walking beam furnace lining has been tried. Indeed refractory precast blocks are in contact with the oxidized surface of the steel slab and are chemically corroded by iron-oxide deposit. Moreover the temperature in the hot zone can be as high as 1200 to 1400° C when steel piece changes. A series of heads have been manufactured in our laboratory at Mons and later embedded under the surface of refractory blocks during their casting in Belref factory. Industrial tests in the rolling-mill division of a steelworks have just begun and first results are full of promise.

II - EXPERIMENTAL PROCEDURE

Raw materials for reaction sintering were:
- Opazir 5 of Quiminsa (Spain) as zircon
- RC152DBM of Reynolds Chemical Co (USA) as alumina
- R-SM2 of Tioxide (England) as titanium oxide.
A wet mixture has been mechanically stirred during ten hours at acid pH for dispersion purpose.
After drying cakes have been milled and sieved under 300 microns and 500 g powder batches have been isostatically pressed in cylindrical moulds.
Green heads have been fired in an electric furnace during 2 hours at 1500° C. Finally the upper surfaces have been machined and chamfered.
Dimensions of finished pieces (see picture 1) are 65 and 70 mm for upper and lower diameter and 50 mm height.

Picture 1 - Reinforcement ceramic head

Five heads have been embedded during casting under the upper surface of each monolithic refractory block. This one is made of a vibrable aluminous concrete (type : BELREF VIBRO REF - 606 M ; temperature limit = 1525° C) on the fire side and made of an insulating concrete
on the bottom (type: BELREF THERMISOL 2; temperature limit = 1250°C). The dimensions and weight of the blocks are 1120 X 775 X 495 mm and 946 kg.
Pictures 2 and 3 show respectively a general view of the block and some details of head location.

Picture 2 - Precast refractory block

Picture 3 - Location of the reinforcement head
III - PHYSICAL AND MECHANICAL PROPERTIES OF CERAMIC HEADS

Some heads have been used for characterization purpose. Bulk density and open porosity have been determined by the water immersion method. Modulus of rupture (σf) has been measured in three point bending and critical stress intensity factor (Kc) using the same device. Data published are mean values after eight determinations. Crushing strengths at room temperature and at 1200° C and refractoriness-under-load (2 kg/cm²) have been measured on cylindrical samples (diameter = 20 mm; height = 20 mm). All the data related to physical and mechanical properties of the mullite zirconia material are presented in table I.

Table I: Physical and mechanical properties

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bulk density (g/cm³)</td>
<td>3.57</td>
</tr>
<tr>
<td>Open porosity (vol %)</td>
<td>0.1</td>
</tr>
<tr>
<td>Modulus of rupture σf (MPa)</td>
<td>175 ± 13</td>
</tr>
<tr>
<td>Critical stress intensity factor (Kc) (MPa.m⁰.5)</td>
<td>2.4 ± 0.1</td>
</tr>
<tr>
<td>Crushing Strength R.T. (MPa)</td>
<td>400</td>
</tr>
<tr>
<td>Crushing strength 1200°C (MPa)</td>
<td>150</td>
</tr>
<tr>
<td>Refractoriness-under-load (R.U.L.)</td>
<td></td>
</tr>
<tr>
<td>10.5(°C)</td>
<td>1595</td>
</tr>
<tr>
<td>11.0(°C)</td>
<td>1605</td>
</tr>
<tr>
<td>15.0(°C)</td>
<td>1630</td>
</tr>
</tbody>
</table>

These data show that physical and mechanical properties of these heads with rather large dimensions are quite satisfactory for a mullite type compound sintered at relatively low temperature. In comparison on the laboratory scale best classically sintered mullite materials give σf and Kc values of 150 MPa and 1.8 MPa.m⁰.5 respectively. R.U.L. measurement gives the expected value from the quaternary system diagram. That means that transient liquid phase which accelerates reaction kinetic and densification at about 1450°C, disappears effectively before the end of the process. So, the material obtained by this route presents an initial permanent liquid phase formation at 1595°C which is an increase of about 150°C in comparison with its sintering temperature.

IV - CONCLUSION

A reaction sintering process based on a fundamental study of the quaternary system ZrO₂-SiO₂-Al₂O₃-TiO₂ has been applied with success in the manufacturing of ceramic reinforcement heads for walking beam furnace lining in steelmaking works. Despite the big dimensions of the components mechanical properties of the material are quite good when compared with those of mullite materials prepared by classical methods.

REFERENCES

/5/ PENA P. and de AZA S. - Science of Ceramics 12 (1983) 201
/6/ CRICB- Belgian Improvement Patent No 898.604 ; IC : C048 ; date : 84.01.31