THERMOSHOCK FRACTURING IN ALUMINA REFRACTORIES: CRACK GROWTH AND PATTERN FORMATION IN A HETEROGENEOUS MEDIUM

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ABSTRACT HEADING

Multiple thermoshock experiments were performed on discs of an as-applied alumina (mullite) refractory. The objective was to characterize the progressive development of thermoshock fracturing in a real microstructure, to help identifying the way in which pre-existing microstructure influences thermoshock damage. The most important result from thorough microscopical characterization is that a disaggregation pattern develops that appears to be controlled by the extent, distribution, and shape of porosity in this mullite brick and it is suggested that realistic fracture dynamics modeling of thermoshock resistivity has to incorporate this controlling influence of material inhomogeneity.

1 INTRODUCTION

Aluminium-rich refractories are widely used in the metallurgical industry and damage by repeated thermoshocks is one of the main factors limiting their lifetime. Designing refractories with better mechanical thermoshock resistivity necessitates a detailed understanding of the fracture generation and growth responsible for thermoshock damage at the microscopical level. Current research frequently centers on fracture dynamical studies trying to establish physical models for fracture growth in heterogeneous media (Hasselman, 1969; Soboyejo et al. 2001) that pay relatively little attention to the ubiquitous presence of a preexisting realstruktur in refractory bricks in actual use. The approach followed here is to experiment on a refractory as used in service, and to try to identify the controlling microstructural parameters on fracture formation and eventual damage accumulation from a thorough microstructural characterization of samples after repeated experimental thermoshocking.

2 EXPERIMENTAL

Two mid-alumina, andalusite based bricks (58 resp. 59% Al_2O_3) have been used for experimentation. The two materials are produced by standard industrial processes involving high temperature sintering at ca. 1500 ºC from natural andalusite (kerphalite) as raw material. They

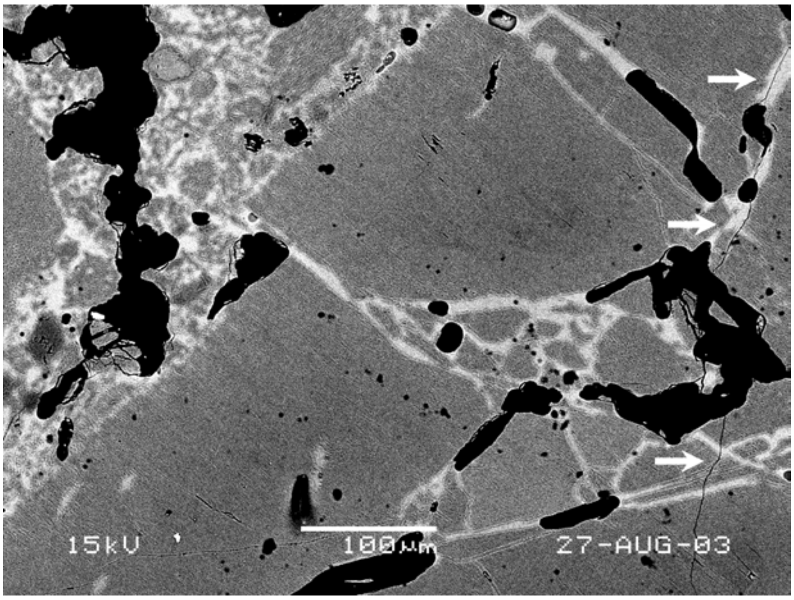

Fig. 1: (SEM image) Example of the general refractory microstructure: note the distinction between a blocky mullite glass symplectite pseudomorphs after original andalusite and intergranular crystal-melt mush (ceramic bond; melt is light-grey). Note the presence of porosity connected with extensive melt filled pre-thermoshock fractures. Arrows indicate a thermoshock-derived fracture connecting pre-existing porosity with disregard to melt or mullite.

contain minor additions of SiO_2 and corundum to steer the composition. The total amount of non aluminosilicate oxides is less than 3 %. Of each refractory, flat discs of 1 cm thickness and 5 cm diameter have been prepared and subject to downquench thermoshocks after thermal equilibration at temperatures ranging from 700 to 1300 °C. Discs were quenched in water and the process was repeated to up to 6 thermoshock cycles. The disc geometry assures that, apart from edge effects, the flat surfaces of the discs are planes of near-isotropical contraction, with the maximum tensile stresses during thermoshock lying in the disk surface planes, normal to which the formation of fractures by tensile failure can be expected. Therefore, these surfaces were observed after quenching in detail by reflected light optical methods and SEM. Identification of fractures and of connected porosity was facilitated by vacuum-impregnation of the observed surfaces with a coloured epoxy resin that yields a strong contrast under reflected light XPL illumination.

3 MICROSTRUCTURES

The microstructure of the two refractories is derived from the raw material andalusite grains during sintering. Both refractories are composed of a package of former andalusite grains of angular to euhedral shape, with grain sizes of ca. 1 mm median. These former andalusite grains are converted into symplectites of mullite + SiO_2-rich glass to variable degrees; in one of the two refractories, a notable proportion of andalusite remaines unconverted in the grain cores, encapsulated in thick symplectite shells. The symplectites have individual crystallite dimensions of 1-5 mm with variable coarsening during sintering. Theses symplectic andalusite pseudomorphs are bound together (ceramic bond) by interstitial aluminosilicate composition ranging from 74-78% SiO_2, coincident with the SiO_2-mullite cotectic at 1300 degrees. This glass is densely loaded with

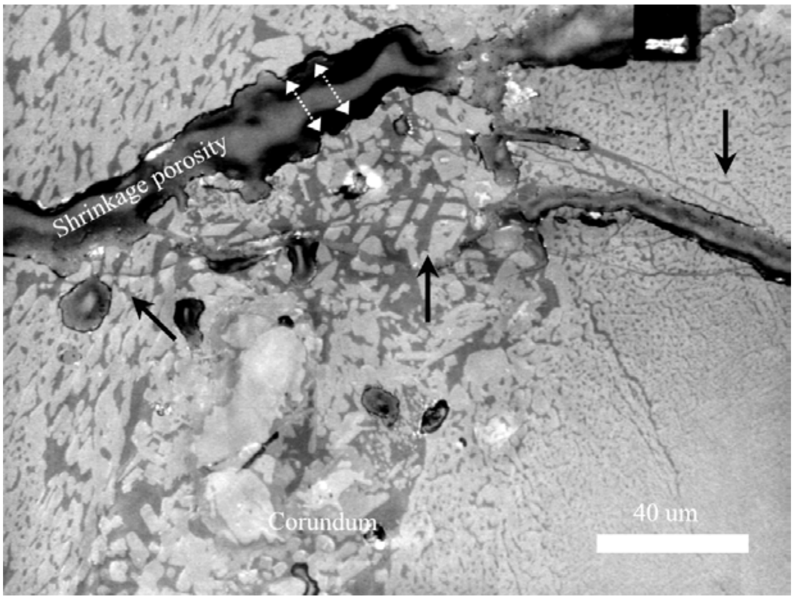

Fig. 2: Example of thermoshock-induced microcrack splay (arrows) relieving stresses between open elongated pores but refilled by melt (healed) during a subsequent thermal equilibration cycle. Note net dilatation of segmented individual mullite microcrystals. Reflected Light, glass is darker grey.

mullite microprecipitates and fragments of symplectic silicate. Strong reaction gradients surround minor refractory constituents (cristobalite, corundum) that are out of equilibrium, but only on a local scale. This overall microstructure did not perceptibly change during repeated thermal equilibration at 1300 degrees as inherent in the thermoshock experiments.

The microstructure of the refractories displays moreover an abundant array of pre-experimentation weaknesses. First, there is a high amount of porosity: the overall porosity is 20%, of which 16% is connected, and the other part closed, e. g. in frequent foam-like bubbles in the intergranular glass, or in pre-production original voids of the andalusite raw material. Secondly the porosity is very inhomogeneously shaped and distributed. A large amount of open cracks is present in the refractories before experimentation, and the cracks range in genesis all the way from natural cracks in the andalusite grains through cracks produced during sintering to cracks formed at the last cooling of the production sintering. All these cracks are present in various stages of healing by melt infill or recrystallization, and they effectively constitute a part of the open (connected) porosity. The crucial distinction between crack formation from the thermoshock experiments and these pre-existing cracks is possible only by detailed microscopical observations.

4 THERMOSHOCK FRACTURE FORMATION

It was found that microscopically identifiable microcracks attributable with reasonable certainty to the thermoshock experimentation were rare compared to the preexisting total crack density. The thermoshock cracks show a progression of morphological forms. The smallest, least evolved thermoshock cracks without noticeable crack separation (<0.5 μm, no displacement) ubiquitously originated at porosity surfaces and are oriented on the microscale distribution of porosity, growing in splays to connect individual pores to each other. Such cracks were found to transgress mullite-

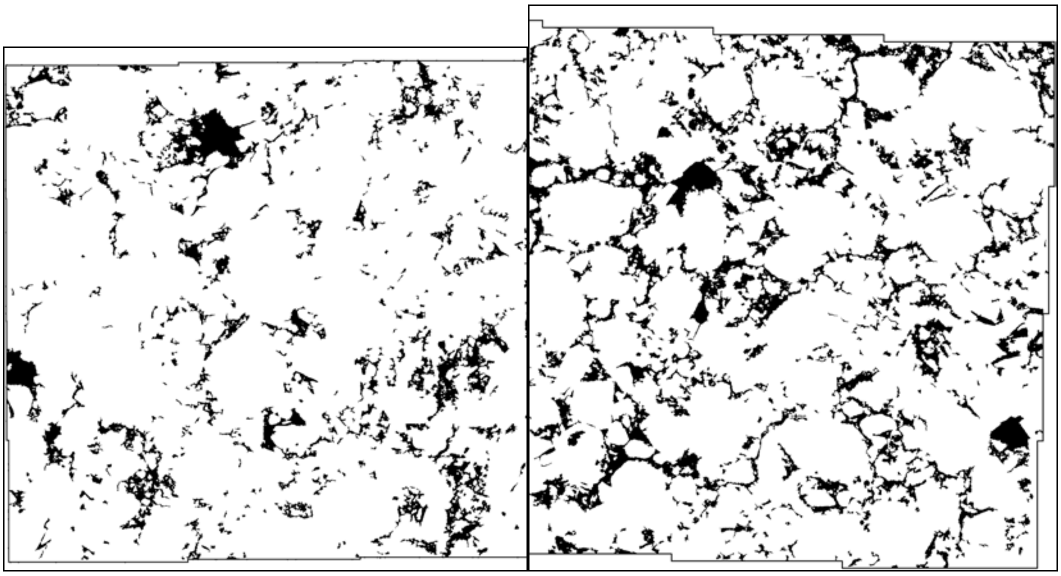

Fig. 3: Tracing of connected porosity (black) from image analysis. View onto the surface of the discs. Left: Original material; Right: After 6 downquench shocks from 1300 ºC. Each image is 11 mm wide. Area fraction of connected porosity is 9.8 % in the left image, 13.2 % in the right image. Note the increase in connectivity of large pore complexes in the after-shock image.

glass interfaces without any stress refraction, implying that their mechanical differences were negligible at the stage of crack formation within the shock temperature interval for controlling crack nucleation and growth compared to the porosity vs. brick matter contrast. Fig. 2 gives an example of such a porosity-connecting microcrack splay. In a further stage of evolution, thermoshock-derived cracks can be traced through the microstructure jumping from pore to pore, and thereby mechanically decoupling crack sides, and frequently using considerable lengths of porosity in the total effective crack length (see Fig. 1 for an example). It was impossible to track individual microcracks through multiple thermoshock cycling of the disks, but careful examination of multiply shock-cycles discs showed that thermoshock induced cracks could be re-entered by melt in a subsequent thermal cycle, partly or fully rehealing them (see Fig. 2 for an example), preserving net dilation.

In multiply shock-cycled disks it was observed that the progressive accumulation of such pore-connecting cracks leads to a change in the configuration of connected vs. unconnected porosity. It was found that relatively wide throughgoing cracks develop with considerable crack openings (10s of μm) that cut through the microstructure and disassemble the microstructure into blocks of a characteristic dimension. This is shown here in Fig. 3, contrasting the connected porosity tracing as found from image analysis using the coloured epoxy impregnation, in a 6-times shocked versus original refractory. The overall proportion of connected "porosity" (including cracks) increases substantially, and the outlined blocks lose their coherent ceramic bond. These blocks may consist of individual andalusite pseudomorph grains, or of assemblages of several of them, or of fragments of them. The local occurrence of pre-existing porosity, where favourably oriented to integration into macrocracks by relatively short interstitial pore-jumping microcrack steps, appears to be more important for this disaggregation process than the detailed mullite-glass or pseudomorphs-matrix configuration. Though a characteristic grain size of this system of disaggregated blocks appears to develop, at current we do not have sufficient data to quantify a link between specific shock conditions and specific disaggregation grain sizes. It is important to emphasize that the porosity and disaggregation development as viewed in Fig. 3 is seen in a surface parallel to the maximum tensile thermoshock stresses. At present, we do not yet have investigated how these dismembered blocks in multiply shocked surfaces transit into the unfragmented original refractory at depth, i. e. along the gradient of decreasing shock intensity. It is possible that they develop basal decollement fractures caused by tangential stress or that the developed open fractures at the surface dissipate into microcrack splays at depth.

5 CONCLUSION

Concluding, we find that the pre-existing microstructure, above all the microscale abundance, shape, and orientation of porosity appears to exert the dominant control on the actual thermoshock damage formation in a refractory as used in the metallurgical industry. We suggest that it might be beneficial to an understanding of thermoshock damage formation in refractories with a pre-existent realstruktur if efforts are directed to modeling the micoscale stress redistribution and focusing enforced by this inhomgeneity, to better assess the true stress conditions to be covered by fracture dynamical modeling of individual microcracks.

6 REFERENCES

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