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Chasing the link between processability and texture in multiphase materials

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Abstract. Using concrete recycling process design as an illustrative example, this paper supports the concept that efficient comminution and separation process design should be based on understanding, and then exploiting, known links between textural properties and processing performance criteria.

Keywords: processability; texture; concrete recycling; fracture porosity.

INTRODUCTION

This work is concerned with unraveling the relationship between texture, and changes in texture, with process design for complex multiphase materials. The material of interest here is concrete. Concrete is interesting in that it is a manmade multiphase material, not too dissimilar from natural ores, but whose texture can be changed through controlled manufacturing in the laboratory. Beyond the broad scope issue of linking texture and processability of multiphase materials, this work has direct practical application for selective liberation between aggregates and cement and development of concrete waste recycling process routes.

In the context of concrete waste processing, the textural property of interest is porosity, which is manipulated through heating. Concrete is heated either externally, with an oven, or using microwaves [1]. In the context of this paper, these heating modes are used to produce different fracture porosity patterns, thereby producing distinct concrete textures with varying degrees of embrittlement. The concept that underlies this work is the conviction that textural properties dictate the overall processability of the material, hence understanding these textural properties is a promising approach for developing efficient processes.

In the context of concrete recycling, processability is measured in terms of aggregate liberation, comminution product fineness and mechanical strength of the material.

MATERIALS AND METHODS

Concrete samples were cast from a mixture of CEM 1 52.5 Portland cement and narrowly sized 2-2.5mm siliceous aggregate particles. The sample preparation and testing protocol is represented in Fig. 1.

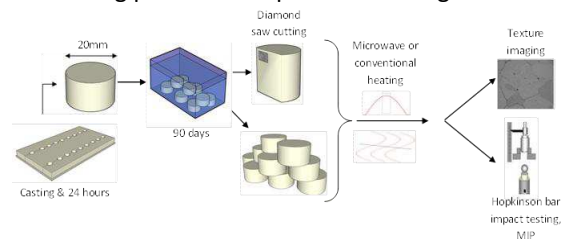


FIGURE 1. Concrete sample preparation and testing protocols.

Samples were cast in 20mm ($\approx 10g$) cylinders and were tested in two ways.

- The first protocol consisted in heating whole cylindrical particles, and then subjecting them to Hopkinson bar impact testing. The Hopkinson bar apparatus is recognized as a sound technique for measurement of important mechanical properties that relate to the level of embrittlement [2, 3].
- The second protocol, dedicated to texture analysis, consisted of diamond saw cutting samples to expose a flat surface in the cylindrical samples prior to heating them, and then observing the flat surface by SEM. The primary concern of this protocol was avoiding tampering with the heated sample surface before observation.

TEXTURE AND EMBRITTLEMENT

On a macroscopic scale, porosity changes in concrete, due to exposure to heat for example, reduces the mechanical strength of the material. This is a key problem associated with fire resistance of concrete. Changes in mechanical strength with total porosity, as plotted in Fig. 2, show an inverse relationship for all concrete types. Such a measurement however does not reveal the actual nature of the changes that take place inside the material, so that it is of no use for process design.

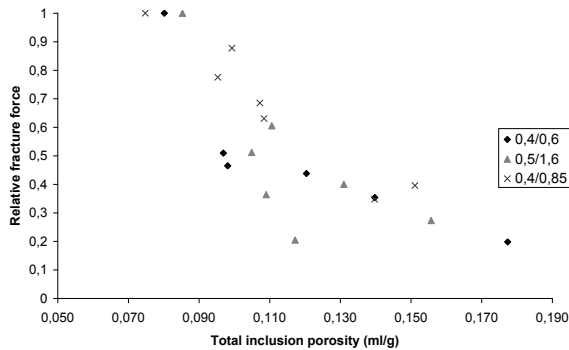


FIGURE 2. Example of correlation between macroscopic textural measurements (here: total porosity measured by mercury porosimetry) and embrittlement.

The source of changes in mechanical properties however, can be found at the local scale, which macroscopic textural measurements cannot reveal. By quantifying the texture of concrete at the local scale through SEM imaging and digital analysis of the porosity, the authors have shown [1] that distinct changes in fracture porosity occur, albeit at different scales inside the concrete, during heating. As seen in Fig. 3, two distinct fracture networks have been identified that develop sequentially, whose properties appear to correlate with important processing performance criteria.

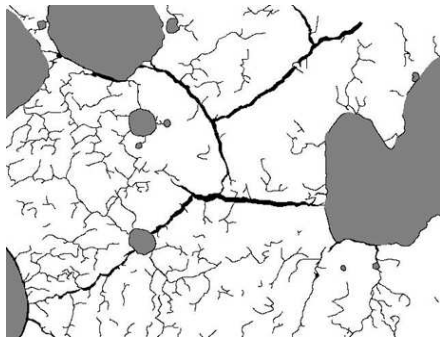


FIGURE 3. SEM photomicrograph exemplifying the formation of primary and secondary fracture networks (grey: aggregate particles; white: cement paste)

The first fracture network is constituted of large fractures that run throughout the concrete sample. The formation of this network is associated with the aggregate-cement interfacial transition zone or ITZ [4], which is a few tens of micrometers thick. It is known for its high relative water content, steep moisture gradient, high porosity and high portlandite content. The growth of the primary network is also found to be associated with pore holes, which play a role in dissipating heat-induced stresses inside the cement matrix. Fig. 3 is a clear example of the primary network growth principles.

Analysis of concrete texture for low microwave energy inputs and low furnace temperature, points towards fractures being initiated near aggregate grain boundaries, hence inside the ITZ and forming a more or less connected network of large fractures that percolates through the concrete sample. X-ray tomographic images (Fig. 4) confirm this finding, originally derived from analysis of two-dimensional images.

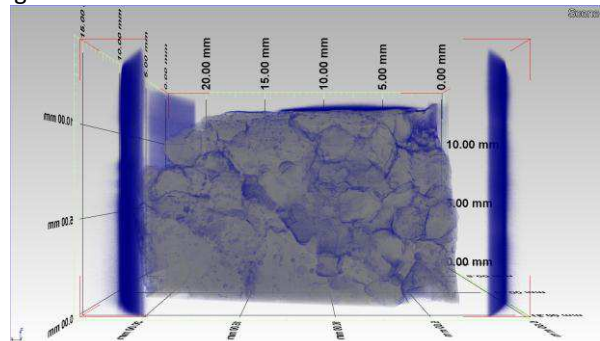


FIGURE 4. X-ray tomographic images of the sample-wide primary network of fractures formed by concrete heating.

As shown in fig. 5, the length of the primary network only changes marginally with additional energy input into the concrete, whereas the width of primary fractures widens monotonically during prolonged heating due to drying shrinkage of the cement matrix.

In terms of processability of concrete for recycling, the primary network leads to aggregate liberation inside the concrete bulk, which the authors refer to as *textural liberation*. In contrast, experimental results show that single-particle impact breakage testing of heated concrete samples, which we refer to as

physical liberation, leads to a significantly lower degree of actual aggregate liberation [1] unless heating has created a dense secondary network of fractures through the continuous cement phase.

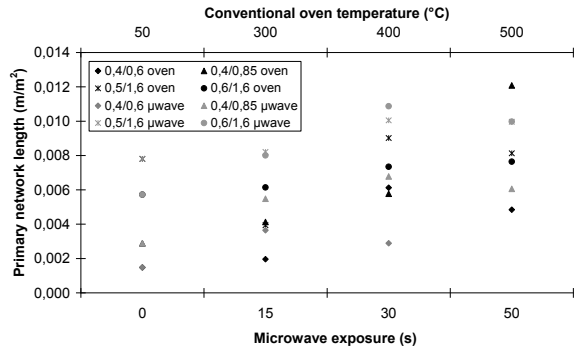


FIGURE 5. Changes in primary network length with increased heat energy.

The secondary fracture network however, which consists of a large number of small fractures that seem to appear randomly as opposed to the primary fractures, are distributed throughout the cement phase. The density of these fractures increases with heat energy input. Upon single-particle impact breakage of heated concrete samples, the authors [5] have shown that fineness of product fragments increases. The source of this lies with the density of the secondary network fractures, which controls the fineness of the product produced by impact breakage.

As shown in Fig. 6 by the convergence between physical and textural liberation at high heat energy input, it is the secondary network which controls the physical liberation of aggregates. Indeed, the formation of the secondary network by heating is necessary for impact breakage to harvest the textural liberation caused by the primary network formation.

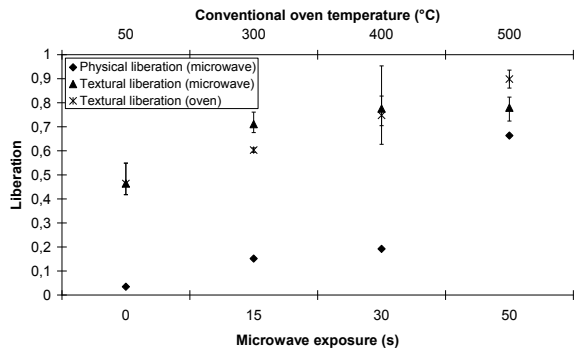


FIGURE 6. Convergence between physical and textural liberation with increased heat energy input.

The ITZ is known to have a significant role in the mechanical performance of concrete, stiffness in particular [4]. Formation of the primary network, as it occurs largely in the ITZ, is therefore expected to modify the mechanical strength of concrete, as measured by Hopkinson bar tests, as this will separate the aggregate and cement making the mechanical performance of the concrete more strongly dependent on the properties of the cement. HPB measurements do not permit however a definite assessment as to which one of the two fracture networks is most detrimental to concrete strength. Fig. 7 shows that the secondary network formation correlates strongly with the loss in mechanical strength of concrete.

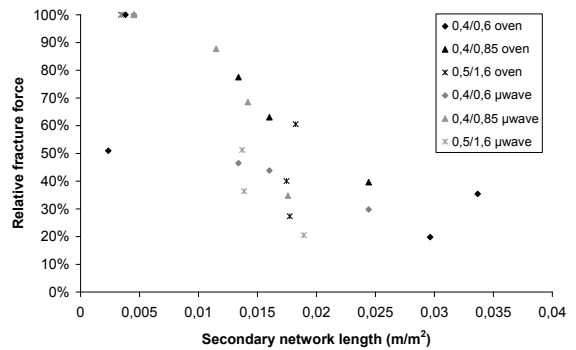


FIGURE 7. Changes in mechanical strength as a function of the secondary fracture network growth.

Having highlighted direct links between changes in texture properties and processability criteria of relevance to developing a concrete recycling process, namely aggregate liberation, product fineness and concrete strength, the following section elaborates on the implications this understanding has for process design.

FROM MATERIAL TEXTURE TO PROCESS DESIGN

The key concept which this work endorses is that efficient process design should be based on understanding, and then exploiting, known links between textural properties and process performance criteria. Having gained some degree of understanding of the relationship between texture and processability criteria for concrete, the purpose of this section is to elaborate on the implications for designing a concrete recycling process that embeds a pretreatment heating step.

From analysis of changes in concrete texture, it was found that:

- Low heat energy input yields a significant degree of textural liberation of aggregates.
- Low heat energy input yields a significant reduction in concrete strength, which reduces further with increased heat input.
- High heat energy input is required for single-particle impact breakage to physically liberate texturally liberated aggregates.
- High heat energy input is required for single-particle impact breakage to produce cement fines (hence liberated cement particles).

Designing an acceptable concrete recycling process must provide satisfactory answers to the following issues:

- Aggregate and cement liberation
- Separation of liberated phases
- Low overall energy consumption

Matching present knowledge about textural behaviour of concrete during heating with processing objectives is not a clear-cut issue. Nevertheless, it narrows down the possibilities.

The first one would be to use low energy heating to form the primary network for high textural liberation of aggregate particles, and then subject the material to a comminution step capable of harvesting the textural liberation. From single-particle impact breakage tests, it can be inferred that repeated impacts would be necessary to physically liberate the aggregate particles. However, repeated impacts would run the risk of comminuting the aggregates, thereby decreasing their recyclability. This seems to call for a shear-inducing comminution process, such as with high-pressure grinding rolls. Mechanical shear would likely yield a high physical liberation of texturally liberated aggregates. However, cement fragments may end up in size classes similar to that of aggregates, possibly leading to subsequent difficulties for separating aggregates from cement.

The second option would consist in using high energy input, for which microwave heating is possibly most suited and effective, to generate a dense secondary network of fractures in the cement matrix. The subsequent comminution step would then require minimal energy input, and would yield cement particles that fall in a size range finer than the aggregate particles, thereby making physical dry

classification possible. Considering our current understanding of heat-induced textural changes in concrete, this processing route seems to be a promising option. As the energy input necessary for this processing choice is high, recovery of heat energy may be necessary for economic viability. This could occur on the form of feeding hot and finely distributed liberated cement particles into the clinker mix, implying that the recycling process should take place at the clinker making site.

CONCLUSIONS

The concept supported by this work is that efficient comminution and separation process design should be based on understanding, and then exploiting, known links between textural and process performance criteria, as materials are being subjected to given physical stresses.

As an illustrative example of this concept, material properties relevant to recycling concrete, a model material for complex multiphase materials, were investigated in relation with local analysis of fracture porosity, one key textural property of concrete. The understanding of texture variations in concrete subjected to heating and impact breakage was used to elaborate on possible concrete waste recycling strategies.

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