



Crack-Resistance of 25 Cements Determined by the Ring Shrinkage Test

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Abstract. Cement-based materials are known for shrinkage strain on drying, causing easily crack initiation under internal or external restraint. Higher crack resistance can be achieved by slowing down the strength gain with slower chemical shrinkage evolution. The ring shrinkage test provides an accelerated method how to quantify cracking resistance on drying, supported with long-term experiments for concrete surface cracking. The experiments presented in this paper use ring shrinkage tests for 25 mortars, which cover 10 commercial cements and 15 on-demand cements, spanning Portland cements and blended cements for 30% slag substitution at maximum. The results proved that the ring cracking time increases while lowering the Blaine fineness or increasing slag substitution. A crack-resistant cements have upper limits for 2-day compressive strength and Blaine fineness which are for Portland cements as 27.7 MPa and 312 m²/kg, respectively. The results show that only two out of ten commercial cements can be considered as crack-resistant, while cements on-demand increase this ratio to 10 out of 15.

Keywords: ring shrinkage test · drying shrinkage · autogenous shrinkage · chemical shrinkage · crack-resistant cements

1 Introduction

Volumetric shrinkage of concrete may result in crack initiation due to internal or external restraints. The first group of driving force is chemical shrinkage, where the C₃S hydration yields approximately 9% volume decrease, manifested partially as autogenous shrinkage. The second, diffusion-driven phenomenon, stands for drying shrinkage. Cement paste, mortar and concrete prisms led to linear shrinkage strains over 3000×10^{-6} , 1400×10^{-6} and 500×10^{-6} at 500 days, on $4 \times 8 \times 32$ mm and $50 \times 50 \times 400$ mm prisms. The mixes had $w/c = 0.50$ and ambient relative humidity was 48% [1]. The magnitude of drying shrinkage was found to be controlled mainly by w/c ratio and relative humidity.

Studies on durability in the USA after 1930s stated that concrete cracking is the most relevant factor, stemming from the production of high early strength cements [2]. Demands for fast construction schedules led to higher C₃S contents, increased the Blaine fineness of cement and lowered the w/c ratio. These changes transformed crack-resistant cementitious materials to crack-prone counterparts [3]. Several recommendations were

issued for crack-resistant cements on drying conditions; German concrete pavements in 1930s preferred slow-hardening cements [4], in 1940 P. H. Bates coined Type II cement in ASTM C 150, restraining 7-day strength to approximately 15 MPa (2200 psi), see more examples in [5].

Visible surface cracks on concrete pavements in the Czech Republic present quantified example of high early strength gain cements [6]. A vehicle equipped with laser crack measurement system (LCMS) scans annually 2 m long sections. The LCMS scanned 887 lane-km out of ~ 1650 current lane-km, covering approximately a half of all concrete pavements on the Czech highways. The scans are automatically segmented into four stages, see Fig. 1 (left):

- 0 – section without visible cracks.
- 1 – visible hairline cracks.
- 2 – crack up to 0.4 mm wide without the presence of concrete disintegration.
- 3 – visible disintegration of concrete, namely around joints.

The crack evolution statistics is showed in Fig. 1 (right) with regards to pavement age. Visible cracking at 10–15 years exists on 75% of the pavements, decreasing the service life from 40–50 to approximately 25–30 years. Many sections had to be removed even after a few years with unacceptable surface cracking. Such situation is in contrast with old concrete pavements which were built in the 1970s and 1980s with concrete lasting well over 40 years free of serious surface cracks. The major shift lies by our opinion in higher cement reactivity. The old cement attained flexural strength 4.8 MPa at 3 days which current road cement reaches already at 1.5 day.

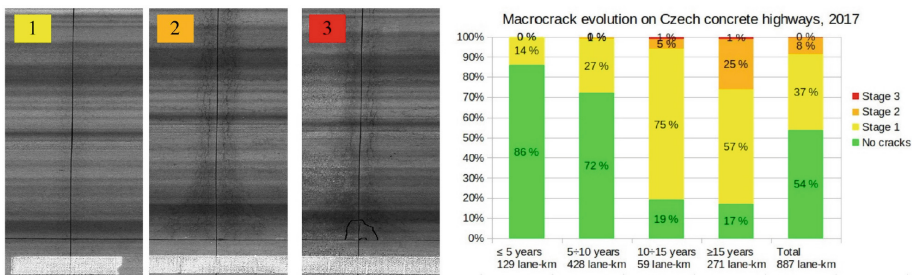


Fig. 1. Stages of concrete surface cracking (left) and statistics of cracking stages with regards to pavement age (right).

The restrained ring test presents a well-established experimental method for assessing the crack resistance of cementitious materials, firstly documented in 1939 by R. Carlson [3]. The test modifications exist on ≥ 13 different ring dimensions and have been adopted by several standards, for example AASHTO T334 or ASTM C1581 [5]. The test captures tensile stresses evolution induced by restrained drying and autogenous shrinkage. Stress relaxation due to aging creep acts positively for crack mitigation. Therefore, the test combines evolution of at least four phenomena occurring during hardening; autogenous and drying shrinkage, creep and tensile strength. The ring shrinkage test yields cracking time of a cementitious material on a given geometry, being useful for comparison and

material optimization [7]. Classical setup uses sealed curing for one day with the drying afterwards, which reached different degrees of hydration for various cements.

Long-term cracking of concrete surface was found to correlate well with the cracking time in the restrained ring test [3]. The experiment was carried out on 27 different cements used to cast 104 panels, $2.74 \times 1.22 \times 0.41$ m in size. They were placed on the shore of the Green Mountain Dam, Colorado, during 1943. The cracking time of the mortar ring showed strong correlation with the surface cracking after 53 years; the smaller the cracking time the more severe long-term cracking. The best performance was found for low alkali cements, lower C_3A content and coarse cements.

This paper focuses at crack resistance assessment of 25 cements, demonstrating the current situation and possible ways for crack-resistant cements. For the campaign, 10 existing commercial cements and 15 on-demand cements were gathered and tested. The ring test used dominantly mortars 3:1 (sand:cement) and $w/c = 0.45$, which yielded mortars with 510–530 kg/m^3 of the cement..

2 Materials and Methods

The experimental program covered 25 cements from eight cement plants, produced between 2017 and 2021. Ten cements are standard products from the market, and fifteen cements were produced on demand, mostly decreasing the grinding time which led to a lower fineness. The experimental program covered Portland cements and Portland-slag blended cement up to 30% slag substitution. Blaine fineness spanned 250 to 433 m^2/kg , see Table 1.

The graphical workflow of experiments is summarized in Fig. 2. Isothermal calorimetry tested cement pastes, while ring shrinkage tests and compressive strength were tested on mortars. Isothermal calorimetry used TamAIR calorimeter at 20 °C (Thermometric AB, Stochholm, Sweden). Only one test per paste was conducted due to excellent reproducibility. Compressive strength followed EN 196–1, carried out on three specimens. Dimensions of the ring test originated from R. W. Carlson’s design, here, it had a thinner steel ring in order to increase the deformation [8], see Fig. 3. Mortars were made from cements mainly at $w/c = 0.45$ m using a sand:cement ratio of 3:1. After 24 h of sealed curing, the rings were exposed to a relative humidity of 45–55% and 19–22 °C. Two to four rings were prepared from each mortar to quantify scatter of cracking time. Further details are summarized in [7].

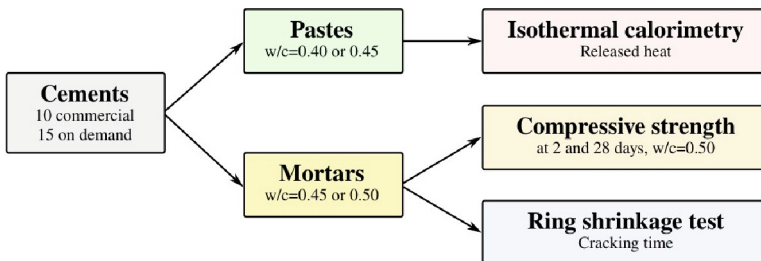


Fig. 2. Graphical workflow of experiments.

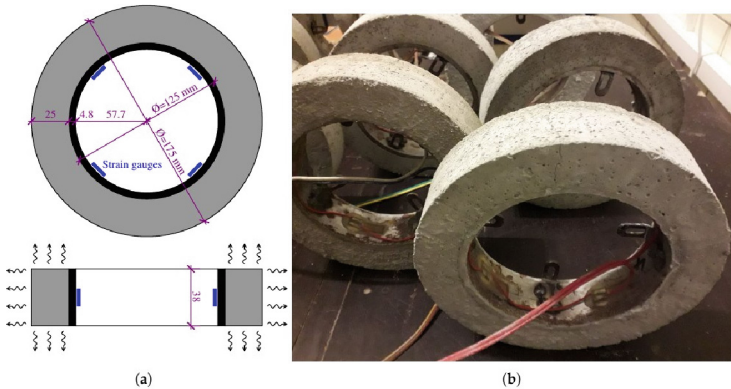


Fig. 3. Used ring geometry with four strain gauges (a) and the ring with a crack (b).

3 Results and Discussion

3.1 Isothermal Calorimetry

Released heat from isothermal calorimetry is summarized in Fig. 4 [7]. An asterisk denotes a crack-resistant cement, which satisfied 40-day cracking criterion as described further. The released heat shows the differences mainly up to 24 h of hydration. The analysis shows that crack-resistant cements liberate less heat at 24 h than 156, 142 and 154 J/g when considering cement groups CEM I, CEM II/A-S and CEM II/B-S.

3.2 Ring Shrinkage Test

Most rings broke in a brittle manner, showing a single crack across the ring cross-section. Figure 5 shows a characteristic evolution of hoop strain for five selected cements. Brittle failure is apparent in the CEM I 42.5 R(sc) cement at 30 days, and CEM II/A-S 42.5 N at 16 days. Cements CEM I 42.5 N with 256 m²/kg, slag-blended cement and CEM II/B-S 32.5 R showed no cracking within the measured period. The strain fluctuations originate from fluctuating ambient relative humidity in the range of 45–55%.

The results from 25 tested cements can be summarized in Fig. 5. The description around a data point stands for the Blaine fineness and for the average cracking time from at least two ring. The symbol \geq indicate termination of experiment with at least one uncracked ring, with the duration of minimum average cracking time. The red cross is used to mark a crack-prone cement that cracks sooner than 40 days. The criterion was deduced from behavior of CEM I 42.5R(sc) Mokra from 03/2018, which is a standard cement used for concrete pavements in the Czech Republic and which takes part in 75% visible pavement cracking up to 15 years, see Fig. 1. The green dots mark crack-resistant cements which show cracking time over 40 days. Two beneficial factors are evident for crack-resistant cements. Low Blaine fineness slowing down hydration and higher clinker substitution with a less reactive slag. The violet line marks off a hypothetical threshold which can be used for classifying crack-resistant cements surpassing 40 days of the cracking time.

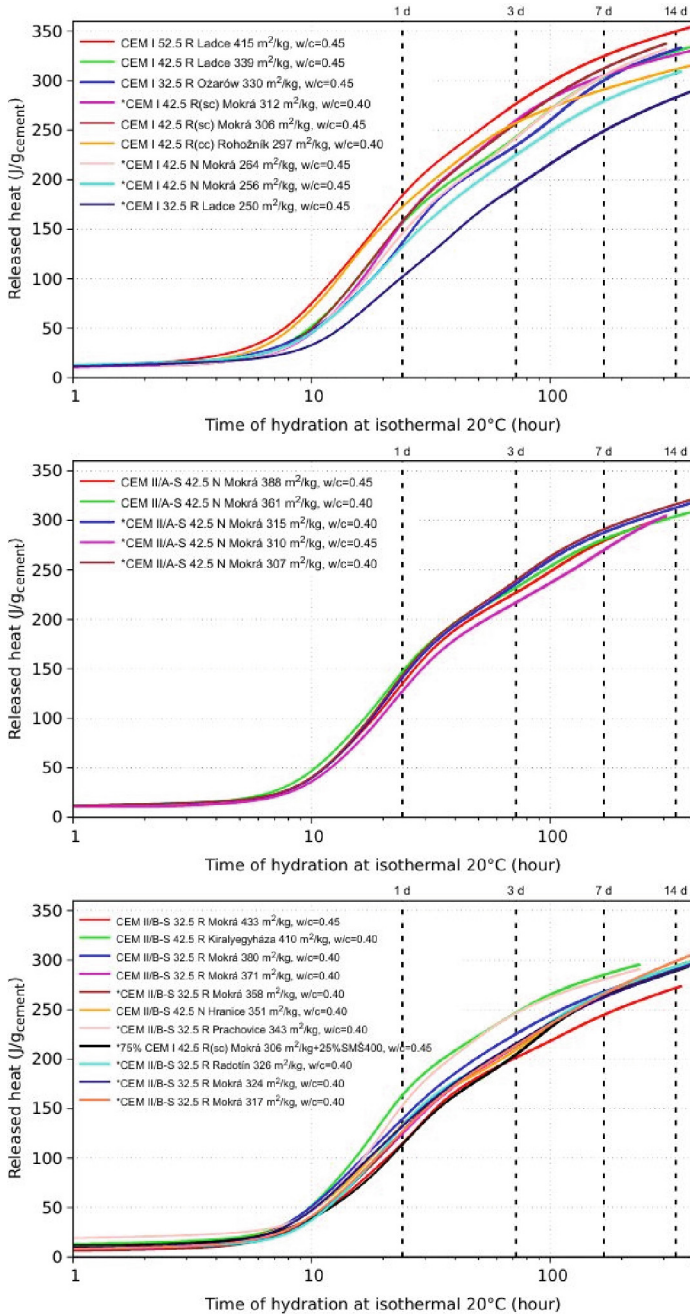


Fig. 4. Released heat for 25 cements. An asterisk denotes a crack-resistant cement.

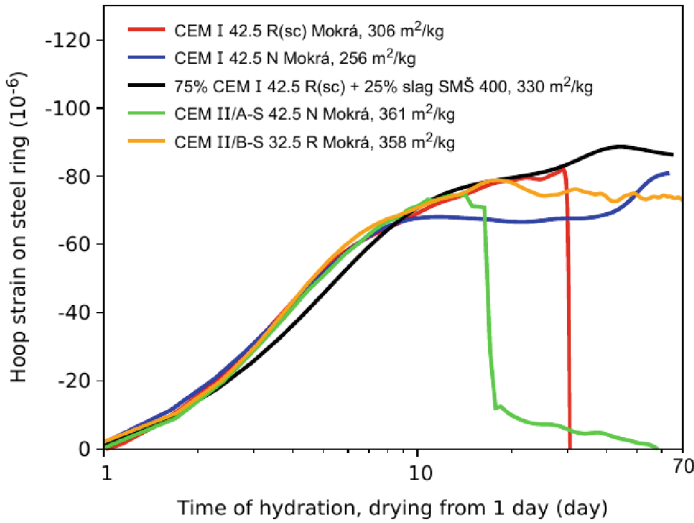


Fig. 5. Hoop strain on the steel ring and behavior of five selected cements.

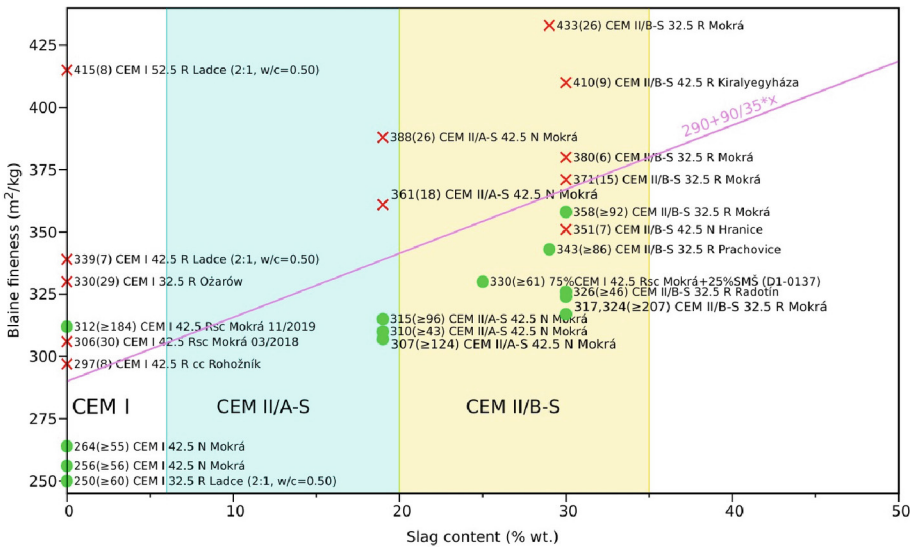


Fig. 6. Results from 25 cements tested in the ring test. The labels code Fineness (Average_cracking_time) Cement_description. Crack-resistant cements are marked with green dots while the red crosses denote cements cracking under 40 days in the ring test.

Maximum fineness for CEM I, CEM II/A-S and CEM II/B-S groups were found as 290, 340 and 380 m²/kg for crack-resistant cements. There are only two crack-resistant cements from commercial production on the Czech market: CEM II/B-S 32.5 R Radotín with Blaine 326 m²/kg and CEM II/B-S 32.5 R Prachovice with Blaine 343 m²/kg.

Green-colored cements can be produced by modifying manufacturing process, especially grinding time. Unfortunately, those crack-resistant cements slowly vanished from the market. The reason stems from demands on fast construction schedules, supported by general unawareness of early strength gain with the consequences for long-term cracking.

Table 1. Summary of main properties of tested cements. The cements are sorted according to class and the Blaine fineness.

Cement	Blaine fineness (m ² /kg)	Compressive strength, 2d (MPa)	Compressive strength, 28d (MPa)	Released heat, 24 h (J/g)	Ring crack time \pm st.dev. (d)
*CEM I 32.5 R Ladce	250	11.9	37.9	102	$\geq 60 \pm 0$
*CEM I 42.5 N Mokrá	256	21.1	47.7	133	$\geq 56 \pm 0$
*CEM I 42.5 N Mokrá	264	21.2	53.3	146	$\geq 55 \pm 0$
CEM I 42.5 R(cc) Rohožník ^b	297	28	56	172	8 ± 4
CEM I 42.5 R(sc) Mokrá, March 2018 ^b	306	27.5	59.5	157	30 ± 0
*CEM I 42.5 R(sc) Mokrá, 11/2019	312	27.7	59.7	156	$\geq 184 \pm 35$
CEM I 32.5 R Ožarów ^b	330	21	45	137	29 ± 5
CEM I 42.5 R Ladce	339	27.1	52.2	156	7 ± 1
CEM I 52.5 R Ladce	415	32.6	58	184	8 ± 2
*CEM II/A-S 42.5 N Mokrá	307	22.5	53.1	142	$\geq 124 \pm 0$
*CEM II/A-S 42.5 N Mokrá	310	18.2	50.4	127	$\geq 43 \pm 19$
*CEM II/A-S 42.5 N Mokrá	315	21.2	52.4	140	$\geq 96 \pm 0$
CEM II/A-S 42.5 N Mokrá	361	22.6	52.5	147	18 ± 2

(continued)

Table 1. (continued)

Cement	Blaine fineness (m ² /kg)	Compressive strength, 2d (MPa)	Compressive strength, 28d (MPa)	Released heat, 24 h (J/g)	Ring crack time ± st.dev. (d)
CEM II/A-S 42.5 N Mokrá ^b	388	21	54	135	26 ± 1
*CEM II/B-S 32.5 R Mokrá	317	14.9	46.6	126	≥ 207 ± 0
*CEM II/B-S 32.5 R Mokrá	324	16.4	48.5	133	≥ 207 ± 0
*CEM II/B-S 32.5 R Radotín ^b	326	18	48	133	≥ 46 ^a
*75% CEM I 42.5 R(sc) Mokrá + 25% SMŠ 400	330	18	48.5	113	≥ 61 ± 5
*CEM II/B-S 32.5 R Prachovice ^b	343	20.1	50.8	154	≥ 86 ± 6
CEM II/B-S 42.5 N Hranice ^b	351	~21	~52	134	7 ± 2
*CEM II/B-S 32.5 R Mokrá	358	19.2	52.3	126	≥ 92 ± 16
CEM II/B-S 32.5 R Mokrá	371	18.1	53	125	15 ± 3
CEM II/B-S 32.5 R Mokrá	380	20	53.9	139	6 ± 2
CEM II/B-S 42.5 R Királyegyháza ^b	410	18	50	164	9 ± 7
CEM II/B-S 32.5 R Mokrá ^b	433	17	51	115	26 ± 7

^a One ring did not crack for 77 days, the second one exhibited strange early cracking at 15 days, which was interpreted as a manufacturing defect. * stands for a crack-resistant cement, ^b denotes a commercially produced cement

3.3 Discussion

Mix design for ring mortars used the sand:cement ratio of 3:1 in the most of cases. One may truly argue that decreasing cement content will lead to higher crack resistance since drying shrinkage would be reduced. Such approach is true for mortar/concrete optimization [3], however, the goal was to reveal susceptibility for cracking of cements. The reason is that under drying conditions, drying shrinkage creates several invisible

microcracks which later coalesce into visible cracks [3]. This was already confirmed by micromography during sealed conditions [9]. In order to reveal microcrack formation, resulting into brittle ring failure, one has to increase cement content to a certain level. Otherwise, invisible microcracks remain hidden in mortar's microstructure and there will be very little projection to macroscopic behavior. They are dangerous for long-term performance since, during any driving force, they start coalescing into visible cracking.

An attempt was made to replace results of ring shrinkage test by any readily available tests. There are available released heat at 24 h and 2-day compressive strength. The coefficient of correlation between these two sets is 0.84 since it is generally known that increasing reactivity leads to a higher strength, see Fig. 7. However, there is no simple way how to replace the ring shrinkage test with released heat, Blaine fineness or compressive strength, other phenomena come into play, such as drying shrinkage, creep, alkali content etc.

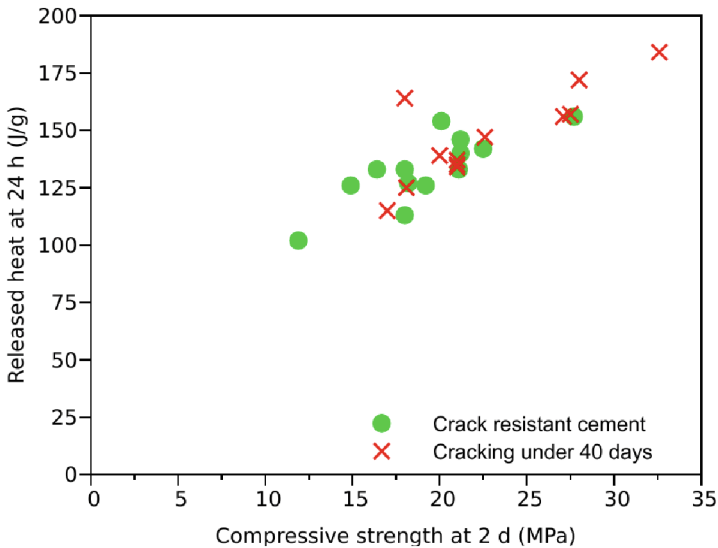


Fig. 7. Relationships among compressive strength, released heat and crack resistance.

For crack resistant cements, it is possible to find the limits in terms of Blaine fineness, 24 h released heat and 2-day compressive strength for three groups of cements, yielding:

- **CEM I** 156 J/g, 312 m²/kg, 27.7 MPa
- **CEM II/A-S** 142 J/g, 315 m²/kg, 22.5 MPa
- **CEM II/B-S** 154 J/g, 358 m²/kg, 20.1 MPa

Systematic testing of cements began in 2017 under the project SPROFIN 500–115-001 issued by Road and Motorway Directorate. Elimination of surface cracking and prolonging service life of concrete pavements were the main objectives. Based on those results, a pilot highway concrete pavement was cast in the length of almost 9 km, using 25% clinker substitution with blast-furnace slag [10]. Visual inspection after 4 years

showed that surface cracking is substantially decreased compared to reference sections made from standard Portland cement.

4 Conclusion

This paper assessed crack resistivity of 25 cements with the help of ring shrinkage test exposed to drying at 24 h. The 40-day criterion for a crack-resistant cement was derived from surface cracking of monitored Czech highways. Cements with slower hydration, i.e., with higher slag substitution or lower Blaine fineness, generally exhibit higher crack-resistivity over cements with higher early strength. Such results are consistent with existing knowledge [2, 3]. Only two out of ten commercial cements fulfilled requirements for crack-resistant cements, both belonging to the CEM II/B-S 32.5 R class. On the other hand, 10 out of 15 cements on-demand testified crack-resistivity, belonging to 32.5 R and 42.5 N classes.

Approximate threshold for crack-resistant cements was proposed, limiting the Blaine fineness as a function of the slag-substitution level. Isothermal calorimetry and early compressive strength provide another estimation for the cracking resistivity of a cement. The ring shrinkage test presents an indispensable method how to access crack resistivity. Further research is needed for more detailed experimental identification of material properties, such as free shrinkage, aging creep, μ CT, to calibrate multiphysical models for crack formation and propagation.

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