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Long-Term Mechanical and Durability Behaviour of Two Alkali-Activated Types of Concrete

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Abstract. A promising solution for reducing the carbon footprint of concrete is the use of alkali-activated concretes (AAC). Before this material can be widely applied, its long-term behaviour needs to be understood, especially since some studies reported a decrease of mechanical properties over time. Similarly, Prinsse et al. reported decreasing mechanical properties, especially elastic modulus and flexural and splitting tensile strength for the studied slag-based AAC (S100) and the blended slag- and fly-ash-based AAC (S50) up to the tested age of 2 years. They hypothesized that these decreases could be only temporarily. To test that hypothesis, this study continued to monitor the mechanical properties of both AACs up to the age of 5 years. As a reference, two OPC-based concretes (OPCC), with different strength classes, are monitored up to the age of 3.5 years. In addition, the internal structures of the concretes are assessed for carbonation and internal micro cracking. S100 shows stabilization of the elastic modulus and the compressive strength, whereas the tensile splitting strength continued to decrease up to 5 years. This is attributed to a combination of carbonation and drying, since the microscopic analysis showed increased porosity around the ITZ and in the carbonated region. In addition, S50 shows an ongoing decrease of all tested mechanical properties, which is attributed to carbonation. No decreases in mechanical properties are found for OPCC.

Keywords: Alkali Activated Concrete · Time-dependent Behaviour · Alkali Activated Slag · Drying · Durability · Carbonation

1 Introduction

The second most widely used material in the world is concrete [1]. Although concrete enabled us large prosperity, due to its widespread usage, the concrete industry is responsible for 5–8% of the anthropogenic carbon footprint [2–4]. With the current rise in climate awareness, reducing this carbon footprint of concrete has become an important priority of the concrete industry. One way to significantly reduce the CO₂-emissions is to replace the conventionally used OPC-binder, with alternative binders. Especially alternative binders made upon alkali activation of precursors (e.g. Ground Granulated Blast Furnace Slag) are promising [5, 6]. Using these binders in concrete creates so-called Alkali Activated Concretes (AACs).

In order to apply AACs it is vital to understand the structural behaviour over a long period of time, because we commonly build concrete structures to last for over 50 years. However, there is limited understanding of the long-term behaviour of AACs [7–11]. As a consequence, there is lack of norms and guidelines for their structural application. This limits the application of AACs. The understanding of this long-term behaviour is limited, since studies reported contradicting results, with some reporting a decrease in mechanical properties over time [12–18]. These contradicting results are hard to compare, since different mix designs and curing methods were used among the studies.

To overcome this, a study was performed by Prinsse et al. [10]. They compared two mix designs consistently, in an attempt to see if there is a decrease in mechanical properties over time and if so, what the effect is on the flexural behaviour of reinforced concrete beams. They found a significant reduction in the tensile splitting strength, flexural strength and elastic modulus, monitored up to the age of 2 years, for both tested mix designs [10]. They hypothesized that these decreases could be caused by drying. Especially since drying at 40% relative humidity of OPC-based concrete could reduce the elastic modulus to 70% of the elastic modulus when continuously cured. Maruyama et al. [19] attributed this decrease to micro cracking of the interfacial transition zone (ITZ). However, Prinsse et al. did not find a clear microstructural change with Environmental Scanning Electron Microscopy (ESEM). Even more, the effect on the structural behaviour of the AACs beams under four point bending was, at least up to the tested ages, limited [10]. This discrepancy between the structural behaviour and the material properties could be a size effect of drying, but it also leads to the hypothesis of temporarily decreasing material properties due to Eigen stresses. However, no recovery of the mechanical properties could be observed within the first 2 years of age.

In continuation of the aforementioned study, the aim of the current research is to investigate if the previously observed decrease in mechanical properties is temporarily. If not, what is causing the decrease in mechanical properties over time? And how does this relate to the time dependent behaviour of conventional concrete?

To answer these questions, the earlier mentioned study of Prinsse et al. [10] is continued by monitoring and testing specimens exceeding the age of 5 years. In addition, a normal and a high strength OPC-based concrete are studied up to 3.5 years. Lastly, an attempt is made to explain the decrease of long-term mechanical properties, by evaluating the internal structure and potential damage of the concretes in three different ways.

2 Experimental Part

2.1 Mix Proportions and Specimen Preparations

To determine if the decrease in mechanical properties is temporarily, the AACs from Prinsse et al. are tested up to 5 years of age. The tested AACs are a slag-based concrete (S100) and a blended precursor made from 50% GGBFS and 50% FA (S50). The details of these mix designs and the chemical compositions of the precursors can be found in Prinsse et al. [10]. Two OPC-based mix designs are made as reference. A normal strength concrete (NSC) is designed to have a similar elastic modulus as the AACs and a high

strength concrete (HSC) is designed to have a similar compressive strength as the AACs (Table 1). The NSC and HSC are mixed in batches of 100L. After casting the samples are vibrated for approximately 20 s, followed by sealed curing. After 24 h of sealed curing, the samples are demoulded and placed inside a fog room (95% RH, 20 °C) up to an age of 28 days. After the 28-day curing period, all the samples are exposed to controlled laboratory conditions (55% RH, 20 °C). The OPC-based concretes are tested from 28 days up to an age of 3.5 years.

Table 1. Mix proportions for NSC and HSC.

	NSC	HSC
Ingredient	(kg/m ³)	(kg/m ³)
CEM I 42.5 N	260	0
CEM I 52.5 R	0	366.7
Sand (0–4 mm)	847.4	841.7
Gravel (4–8 mm)	394.2	373.3
Gravel (8–16 mm)	729.2	653.3
Water	156	166.7
w/c-ratio	0.6	0.45

2.2 Testing and Methods

The following three mechanical properties are studied: compressive strength, tensile splitting strength and the elastic modulus.

Firstly, the compressive strength is determined on 100 mm cubes. These tests used a loading rate of 6.5 kN/s and are performed in accordance with EN 12390–3. Secondly, the tensile splitting strength is determined on 100 mm cubes. A loading rate of 1.1 kN/s is used. These tests are performed in accordance with EN 12390–6. Lastly, to determine the elastic modulus, 100 × 100 × 400 mm³ prisms are tested in compression. A loading rate of 6.5 kN/s is used. Following method B of EN 12390–13, the elastic modulus is determined from the last loading cycle from 0.1 $f_{cm,prism}$ up to 0.33 $f_{cm,prism}$. The compressive strength of the prisms ($f_{cm,prism}$) is determined as 85% of the 100 mm cubic compressive strength (f_{cm}). The aforementioned tests are performed in threefold. For both the compressive strength test and the tensile splitting strength test the CYBERTONIC machine is used, whereas for the elastic modulus test, the TONI-BANK machine is used. The previously published results of Prinsse et al. [10] are used to complement the development of material properties over time. Before using their results, they are reanalysed to be consistent with the newly obtained results.

In addition to determining these material properties over time, the internal structure and potential damage of the materials are assessed. Three indicators are used for this: carbonation depth, non-linearity of elastic stress-strain curves in compression and

microscopic images of epoxy impregnated samples. Firstly, the carbonation depth is determined with phenolphthalein following the procedure of EN 13295. That testing consists of at least four sections from at least two different samples per mix design at an age of 3.5 and 5 years. Secondly, the non-linearity of the stress-strain curves of the elastic modulus test is used as an indicator for internal damage [20]. In the current study this non-linearity is assessed by determining the coefficient of variation for the elastic moduli, determined from 10–20% of $f_{cm,prism}$ and 10–33% $f_{cm,prism}$ of the third loading cycle. Lastly, microscopic images from epoxy impregnated samples with a size of $45 \times 45 \times 10 \text{ mm}^3$ are analysed. These samples are sawn out of 100 mm cubes, using a wet sawing method. After sawing, the samples are impregnated with an epoxy. Next, the top layer is removed by grinding. This grinding allows for making a smooth top surface, but also removes suddenly formed drying cracks during the preparation of the samples. Finally, an optical UV-light microscope is used to analyse the samples.

2.3 Results

Following these six testing procedures the results are presented as follows. Firstly, the development of the compressive strength is shown, followed by the tensile splitting strength and elastic modulus. Next, the non-linearity of the stress-strain curves is presented. Subsequently, the carbonation depths are reported. Lastly, the results of the microscopic analysis are presented.

The development of the compressive strength over time is presented in Fig. 1a. The strength of S100 seems to be constant up to the age of around 5 years (1981 days) with the slight reduction of the mean compressive strength after the age of 91 days, which stays within the standard deviation of earlier measurements. Whereas for S50 the compressive strength reduces after the age of 695 days, with an average value decrease of 22% from the age of 695 days to 1983 days. This is different from the OPC-based concretes, which both show an increase in strength over time. Especially NSC shows a significant increase over time, as the mean compressive strength increases with 73% from 28 days to 1304 days.

Similarly, the evolution of the tensile splitting strength is shown in Fig. 1b. For both AACs the splitting strengths shows a decreasing trend up to the testing age (around 5 years, i.e. 1981 days and 1983 days). Especially, S100 shows a significant decrease in the mean strength of 21% from 695 days to 1981 days. Whereas, the splitting tensile strength of S100 was relatively constant up to 695 days. On the contrary, S50 shows a continuous decrease in the mean splitting tensile strength for all the tested ages. Opposite trends are found for NSC and HSC, which both show an increase in splitting strength until the tested age (3.5 years). The larger splitting tensile strength increase is found for NSC, since the splitting strength increase with 66% from 28 days to 1304 days of age.

Furthermore, the development of the elastic modulus is shown in Fig. 1c. From this figure it becomes clear that S50 has an ongoing decrease up to 30% in the mean elastic modulus from 28 days to 1983 days. Whereas, the elastic modulus of S100 seems to decrease with 20% from 28 days to 193 days, followed by a stabilized trend up to 1981 days. On the contrary, both NSC and HSC show an increasing mean elastic modulus from 28 days up to 3.5 years of age, even though some of the consecutively measured increases are within the standard deviations.

Next the internal structure and potential damage are assessed. The first used indicator is the non-linearity, expressed as coefficient of variation, of the stress-strain curve from the third loading cycle of the elastic modulus test. The results are presented in Fig. 1d. For both AACs the non-linearity is larger, as the coefficient of variation is larger, compared to the OPC-based concretes. Even more, for both AACs the non-linearity increases from 28 days up to an age of 695 days, whereas the OPC-based concretes show relative stable developments of the non-linearity in their stress-strain relationships. The non-linearity for S50 ranges 3.9%-8.4%, whereas this is between 2.9%-6.7% for S100. For NSC and HSC the non-linearity ranges between 0.1%-0.8% and 1.4%-2.4%, respectively. Note that for both the AACs and the OPC-based concretes the coefficient of variations of the mechanical properties are randomly distributed between 1–10% over time. This is within the allowed statistical spread defined by EN 12390.

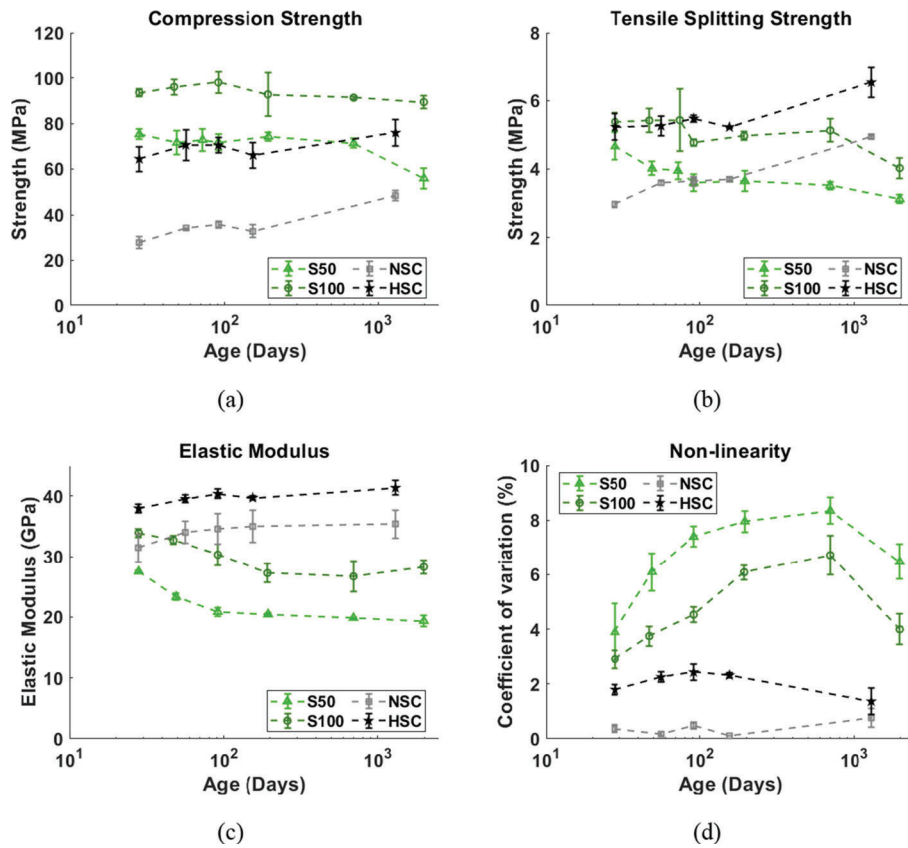


Fig. 1. Development of (a) compressive strength, (b) tensile splitting strength, (c) elastic modulus and (d) non-linearity of third loading cycle of elastic modulus over time for the alkali-activated concretes (S50, S100) and OPC-based concretes (NSC, HSC).

The second indicator is the carbonation depth. The results are presented in Table 2. These results show that there is carbonation in the AACs, whereas there is no measurable carbonation depth in the OPC-based concretes. Especially, S50 shows a significant carbonation depth of 23.87 mm, which is significantly larger compared to the 4.24 mm depth of S100.

Table 2. Carbonation depth for the investigated materials at an age of 3.5 and 5 years. The standard deviation is reported in brackets. All mentioned values are in millimetres, unless specified differently.

Label	Age (days)	Sample 1	Sample 2	Sample 3	Mean
S50	1989	25.22	23.59	22.81	23.87 (± 1.23)
S100	1987	4.73	4.87	3.11	4.24 (± 0.98)
NSC	1304	0	0	0	0
HSC	1299	0	0	0	0

Lastly, the microscopic results are presented in Fig. 2. Since these samples are cut from larger samples ($100 \times 100 \text{ mm}^2$), the middle dashed lines indicate the center of the original samples (i.e. areas which were not directly exposed to drying and carbonation). These samples are impregnated with epoxy which under UV light shows regions with higher porosity and cracks. Accordingly, the results show higher porosity in the paste for NSC, compared to HSC. This was expected for a lower-strength concrete with a higher water/binder-ratio. In addition, both OPC-based concretes show porous ITZs, whereas the ITZs of NSC are more porous, compared to HSC. For both OPC-based concretes the porosity is more or less uniform over the analyzed section. This is different for the AACs, where more porous surface layers are found. These high porosity layers are thicker for S50. This is in line with the found carbonation depths of these AACs and the lack of carbonation of the OPC-based concretes. Interestingly, the carbonated outer layer of S100 is brighter, indicating a higher porosity, compared to S50. In the porous outer layer, porous ITZ's are found for both AACs, whereas no distinguishable ITZ's are found in the noncarbonated regions of the AACs. Even more, there is very limited epoxy penetration in the non-carbonated region of the AACs, indicating a dense microstructure. Lastly, none of the materials show internal cracks in the paste at the current level of detail, whereas all the materials show to contain some micro cracks in aggregates.

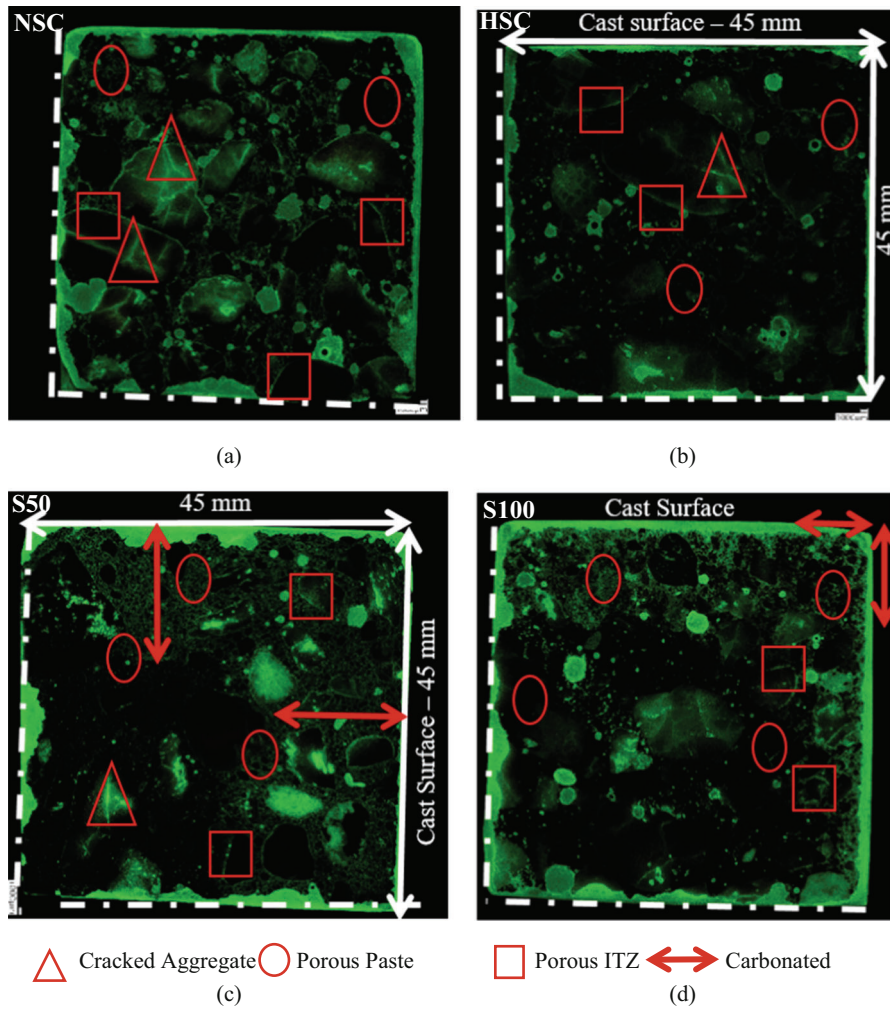


Fig. 2. Microscopic analysis of 45 mm x 45 mm epoxy impregnated samples of (a) NSC, (b) HSC, (c) S50 and (d) S100.

3 Discussion

The mechanical properties for two types of AACs have been tested up to the age of 5 with the aim to answer the following question: to what extent are the previously observed decreases in the mechanical properties of the AACs, S50 and S100, of temporarily nature? It was not expected to find recovery of the mechanical properties, as similar studies reported deteriorating mechanisms, such as efflorescence, carbonation and drying, for their AACs over time [12–17].

The current study shows that there is no recovery of the mechanical properties of both tested AACs after 5 years. In fact, the tensile splitting strength continued to decrease

for both AACs. Whereas, for S100 the compressive strength and elastic modulus show a stable trend over time. On the contrary, S50 showed a continuation of the decrease in the elastic modulus. Even more the compressive strength showed a significant decrease of 26% from 28 days up to the age of 5 years. These decreases in mechanical properties for AACs are not observed for OPC-based concretes. On the contrary, both NSC and HSC showed increasing mechanical properties up to the tested age of 3.5 years compared to 28 days values. Especially the decrease in compressive strength for S50 is remarkable, since it was not observed in the previous study. This decrease can be attributed to carbonation, since a carbonation depth of 23.87 mm was found for S50 at an age of 5 years. This is in line with the previously reported carbonation in AACs by Nedeljkovic [9]. Upon carbonation in AACs, the microstructure is decalcified due to the reaction with penetrated CO₂. The decalcification leads to so-called carbonation shrinkage, which could lead to micro cracking and a decrease in strength [9, 21]. This effect has also been reported for slag-rich cements [22]. On the contrary, cement-rich OPCCs show in general no decalcification of the matrix, which is attributed to free CaO in the pores. Upon reaction of free CaO and penetrated CO₂, solids are formed, which decreases the porosity. Consequently, a slight increase in the mechanical properties is usually the effect of carbonation for OPC-based concretes [9, 22]. The carbonation rate of alkali activated ground granulated blast furnace slag (GGBFS) has been reported to be small, due to the commonly obtained dense pore structure, high Ca/Si ratio and slower consumption of Na⁺ ions from the pore solution [9], which is in line with the 4.24 mm carbonation depth found for S100.

However, S100 also has decreasing mechanical properties over time. Especially the tensile splitting strength for S100 continued to decrease until the tested age of 5 years. One explanation might be accelerated drying in the carbonated region, since the microscopic analysis showed a higher porosity in the carbonated region for S100, compared to S50. Even more, the ITZ showed to be more porous in the outer layer, since more epoxy could penetrate in those areas. This could explain the ongoing decrease of the tensile splitting strength, as mainly the aggregate-paste interface is stressed during the tensile splitting strength test. This is in line with the findings from Ismail et al. [23], since they reported a change in the microstructure for slag-based pastes exposed to drying. Upon drying of slag-based pastes, the C-A-S-H gel is prone to desiccation and chemical changes, as the chemically bound water is easily removed at ambient temperatures. Whereas for a blended precursor system (fly ash and slag combined), there was no notable chemical change found upon drying, because most of the water is physically bound [23].

In addition, the non-linearity indicator in elastic modulus measurements is significantly larger for the AACs and shows to increase over time for both AACs. This supports the aforementioned findings of carbonation for S50 and chemical changes upon drying for S100. Even though this indicator showed a decreasing non-linearity from 2 to 5 years of age. This decrease could be attributed to the higher starting load used for the elastic modulus test at an age of 5 years, as a higher starting load avoids reopening of closed cracks. Figure 3 shows the effect of the load level on the elastic modulus for both AACs at an age of 2 and 5 years. The figure shows that higher loads lead to higher elastic

moduli. However, this means that the stress range over which the elastic modulus is determined, becomes more important compared to OPC-based concretes.

Importantly to note, the current study focuses on two specific AACs and compares it with two OPC-based concretes under specific exposure conditions. Thereby, the results presented in this study may not be found for different AACs or for the same AACs under different exposure conditions. AAC is a broad class of materials and even the two studied AACs showed different sensitivity to the deterioration mechanisms carbonation and drying. Both of these mechanisms start on the surface and progress inwards. Consequently, the effect of these mechanisms on the mechanical properties does not only depend on the exposure conditions, but also on the size of the element and might therefore be less prominent in the structural tests, at least at early testing age. However, it would only be a matter of time, before significant effects can also be expected in larger elements. Therefore, it is vital to recognize the effects of decreasing mechanical properties in structural elements.

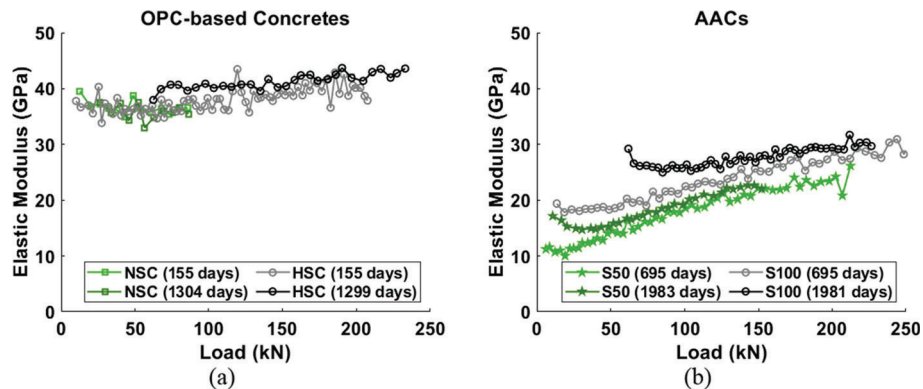


Fig. 3. Elastic modulus determined over every load step from the elastic modulus test for the studied (a) OPC-based concretes and (b) AACs.

4 Conclusions

The aim of this study was two-folded. Firstly, it needed to be investigated why these particular AACs showed decreasing mechanical properties in the previous study by Prinsse et al. [10] and if these decreases were only temporarily. The findings of this study can be summarized as follows:

- After monitoring the mechanical properties of both AACs until the age of 5 years, it is shown that the decreasing properties are not of temporarily nature. Even more, the properties continued to decrease in some cases and even the compressive strength started to decrease for S50. This development of mechanical properties is fundamentally different from OPC-based concretes, which show increasing mechanical properties over time under the same conditions.

- A first reason for this fundamentally different behaviour is found in the higher sensitivity to carbonation of S50, as after 5 years a carbonation depth of 23.87 mm is found.
- Whereas for S100, the combination of drying and carbonation are believed to be the main reasons for the decreasing trend of mechanical properties over time. Especially the carbonated region and the ITZ are subjected to chemical changes, which is most profoundly affecting the tensile splitting strength.
- Based on the microscopic observations, the ITZ zones in AACs seems not to be porous, unlike the one in OPC based system. However, in drying and carbonation affected zones the AACs show clear ITZ zones.
- Independent of the reason for the development of the mechanical properties of the studied AACs, the non-linearity of the stress-strain behaviour in compression (in Elastic modulus test) shows to be an effective indicator for internal damage. Since both AACs showed to have a higher non-linearity, compared to the studied OPC-based concretes. Even more, the non-linearity showed to increase over time for the AACs, which is in line with the ongoing deterioration of carbonation and drying in the AACs.

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