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FATIGUE PROPERTIES OF A SELECTED ALKALI-ACTIVATED ALUMINOSILICATE COMPOSITE

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Abstract: *New materials used for the construction of structures or building components must meet strict construction requirements. Knowledge regarding the fatigue resistance of materials plays a key role during the design and maintenance of civil engineering structures. Previous work by the research team behind this paper deals with selected alkali-activated aluminosilicate (AAAS) composites based on ceramic precursors in terms of their characterization by mechanical fracture parameters, the approximation of their strength evolution over time, and the identification of selected mechanical fracture parameters using artificial neural networks and the comparison of measured load–displacement diagrams with those from simulations in ATENA FEM software. The presented contribution complements a set of mechanical fracture properties of a selected AAAS material via the determination of fatigue properties.*

Keywords: Alkali-activated aluminosilicates, Ceramic waste, Fracture, Fatigue.

1. Introduction

The basic raw material for the production of alkali-activated composite is a precursor with a suitable chemical and mineralogical composition. Amorphous aluminosilicates such as metakaolin or metashale are suitable, as they are materials containing a mixture of aluminosilicates and silica in their reactive form. A source of precursors is being sought in the field of waste materials so that the final product can be designated as a low-carbon product. In terms of their "carbon footprint", alkali-activated aluminosilicates (AAAS) are more environmentally friendly than Portland cement, as they use waste produced in construction, metallurgy, energy and elsewhere (Gavali et al., 2019). In recent years, attention has turned to waste ceramic materials, which are known for having been used as admixtures in mortars and concretes in the ancient past. Waste bricks obtained from the demolition of brick buildings and brick dust obtained from the grinding of insulating elements both appear to be suitable ceramic materials (Reig et al., 2013).

Previous papers deal with selected AAAS composites based on ceramic precursors in terms of their characterization by mechanical fracture parameters (Rozsypalová et al., 2021), the approximation of their strength evolution over time (Frantík et al., 2021), and the identification of selected mechanical fracture parameters using artificial neural networks and the comparison of measured load–displacement diagrams with those obtained from simulations in ATENA FEM software (Lipowczan et al., 2021). The presented contribution aims to complement the set of mechanical fracture properties which are known for a selected AAAS material via the determination of its fatigue properties. For illustrative purposes, these pilot AAAS fatigue results are compared with selected quasi-brittle civil engineering materials – fine-grained alkali-activated composite (Šimonová et al., 2021) and cement composite (Seitl et al., 2019).

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2. Materials and specimens

The paragraphs below introduce the materials which have been selected for testing and comparison – an alkali-activated aluminosilicate composite and a special fine-grained alkali-activated composite, as well as a cement-based composite.

2.1. Alkali-activated aluminosilicate (AAAS) composite

The test specimens were used of waste brick powder that was obtained from the grinding of brick insulating elements as a precursor. The particle size of the waste brick powder was 0–0.3 mm. Potassium water glass with a silicate modulus of M_s = 3.0 modified by potassium hydroxide to M_s = 1.0 was used for alkaline activation of the precursor. Quartz sand composed of three fractions (PG1 0.063–1.00 mm, PG2 0.25–4.00 mm and PG3 1.00–4.00 mm) in the ratio 1:1:1 was used as the aggregate. The mass ratio of the precursor, alkaline activator and aggregate was 1.6: 1: 1.7. The specimens, which had dimensions of 40 × 40 × 160 mm, were prepared for mechanical and fracture testing. After removal from the moulds, the test specimens were wrapped in PE foil and stored until they were tested.

2.2. Alkali-activated concrete/mortar (AAC)

The composition of alkali-activated concrete (AAC-1) is shown in (Hsu, 1981). The dry mass of the activator was 8 % and the water to slag ratio was 0.45. Sodium water glass and potassium hydroxide were combined to reduce efflorescence and an appropriate silicate modulus of the activator (M_s = 0.67 or mass ratio (K_2O + Na_2O) / SiO_2 is 60/40). This activator composition is suitable in terms of both setting and strengths. A naphthalene-based plasticizer was also used to achieve better workability of the mixture. All of the specimens were carefully wrapped in PE foil (to prevent moisture exchange with the environment) and stored outside the laboratory (temperature ≈ 5 ÷ 25°C for 28 days). The fatigue properties of AAC are mentioned in (Zhang and Wu, 1997).

Granulated blast furnace slag with a predominant amount of glassy phase ground to a Blaine fineness of 400 $m^2 \cdot kg^{-1}$ was used for the production of the fine-grained composite (AAC-2). It was alkali-activated using sodium hydroxide solution in a dose corresponding to 6 % Na_2O of the slag weight. The water to slag ratio, including water from the activator as well as extra added water, was 0.45. In addition, 1 % of lignosulfonate-based plasticizer was used. CEN siliceous sand with a maximum grain size of 2 mm was used as aggregate in a dose three times higher than the slag weight. The fresh mortar was cast into moulds and sealed with stretch PE foil to prevent moisture loss. After 24 hours, the specimens were demoulded and stored under laboratory conditions at a temperature of (21 ± 2) °C in a closed box with RH > 95% until they were tested. More details about the mechanical fracture and fatigue characteristics of this fine-grained composite are shown in (Šimonová et al., 2021).

2.3. Fine-grained cement composite (FGCC)

The fine-grained cement-based composite was selected for comparison because the used aggregates were smaller than 2 mm ($d \leq 2$ mm) and mirrored the grading characteristics of standardized sand according to EN 191-1, see (Seitl et al., 2019).

3. Methods of fatigue measurement

The fatigue properties of quasi-brittle materials have been studied for many years – see e.g. (Hsu, 1981, Zhang and Wu, 1997, Lee and Barr, 2004). For the purposes of this study, these properties were measured on three-point bend test (3PBT) configuration with a notch at mid-span (see Fig. 1). The depth of the notches was 4 mm (relative notch depth a/W = 4/40 = 0.1). The fatigue tests were carried out using the experimental set-up shown in Fig. 1 (left). A computer-controlled servo-hydraulic testing machine under load control was used. The ratio of minimum stress to maximum stress in one cycle of loading in a fatigue test $R=P_{max}/P_{min}$ was equal to 0.1. The controlled load frequency for the fatigue tests was equal to 10 Hz. The fatigue endurance limit was determined based on an $S-N$ (Wöhler) curve. The limit of 2×10^6 cycles to fracture was used to consider the safe applied stress amplitude (σ_a) for loading during the whole component lifetime. The experiments were performed in a laboratory with controlled temperature and humidity. The temperature was set to 23 ± 2 °C and the absolute humidity was kept at 10 $g \cdot m^{-3}$ (the corresponding relative humidity is 50 ± 2 %).

3.1. *S–N* curve fitted using Basquin's relationship

Often, *S–N* curves are represented as a straight line in a semi-logarithmic plot as the solution provided by Basquin's relationship (Basquin, 1910, Seitl et al., 2019). Accordingly, the relation between stress range $\Delta\sigma$ and lifetime (number of cycles *N*) is given by the following equation

$$\Delta\sigma = AN^B \tag{1}$$

where the parameters A and B represent the independent term and the slope of the resulting straight line, respectively, in double logarithmic scale.

Basquin's relationship is not valid in the low cycle fatigue region, so caution should be used if the calculated lifetime is low. In our study, the interval of the number of cycles for the application of Basquin's relationship was from 1×10^3 up to 2×10^6 cycles.

Than application for a given completely applied stress $\Delta\sigma_a$ greater than the endurance limit, the number of stress reversals (*N*) after which fatigue failure will occur could be determined

$$N = \left(\frac{\Delta\sigma_a}{A}\right)^{\frac{1}{B}} \tag{2}$$

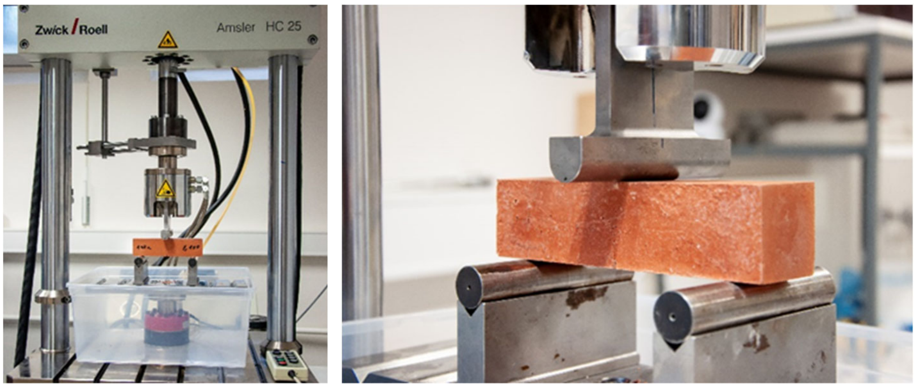

Fig. 1: The experimental set up for fatigue measurements (left); detail of a specimen with dimensions of 40 × 40 × 160 mm (120 mm span) with a relative notch depth of 0.1 (right).

4. Results and discussion

The experimentally obtained pilot fatigue results (*S–N* curve) for the selected AAAS composite are shown in Fig. 2, where the bending stress amplitude applied during the fatigue experiments is plotted against the logarithm of the number of cycles to failure or at 2×10^6 cycles, a limited number of cycles, for run-outs, i.e. for unbroken specimens.

The values of *A*, *B* describing the fatigue behaviour of all materials considered with the coefficient of determination are shown in Tab. 1. The resultant *S–N* curves for all composites are shown in Fig. 2.

The AAAS composite exhibits the lowest bending strength values of all of the mentioned materials. The data also shows the quite low fatigue resistance of the AAAS composite – it is lower than most of the selected materials, as shown in Tab. 1. by materials index *B*. The compared and tested data show similar coefficients of determination. All of the materials based on alkali-activated matrix have a similar fatigue response in terms of lower fatigue resistance compared to the selected cement-based composite.

5. Conclusion

From the presented pilot experimental fatigue research work in the field of alkali-activated aluminosilicate composites based on ceramic precursors the following conclusions can be drawn:

- The tested material showed relatively low fatigue resistance.
- Even at the lowest flexural strength, the material demonstrated a similar fatigue response to the compared selected alkali-activated materials.
- It is obvious that fatigue behaviour cannot be predicted from statically determined strength values.

Tab. 1: Values of Basquin's relationship constants A, B describing the fatigue behaviour of civil engineering materials with the coefficient of determination R².

Materials	Source	*A*	*B*	R^2
AAAS		1.526	–0.058	0.584
ACC-1	(Hsu, 1981)	2.005	–0.027	0.537
ACC-2	(Šimonová et al., 2021)	4.052	–0.082	0.822
FGCC	(Seitl et al., 2019)	3.075	–0.020	0.675

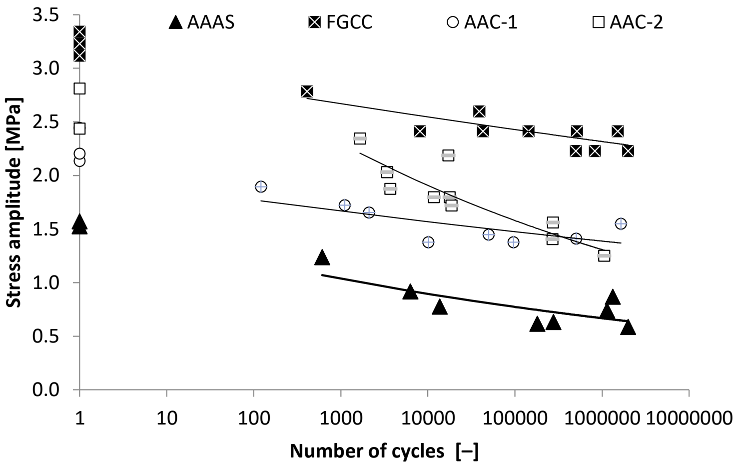

Fig. 2: Comparison of S−N diagrams of the tested AAAS composite and the FGCC, AAC-1 and AAC-2 materials.

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