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# FLEXURAL BEHAVIOR OF CEMENT SYSTEMS REINFORCED WITH HIGH ASPECT RATIO ARAGONITE MICRO-FIBRES

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#### Introduction

Micro-fibres in the context of cement science have been distinguished from larger macrofibres by an arbitrary limit in specific surface area (e.g. 200 cm<sup>2</sup>/g) or fibre length (<6 mm) [1]. Cement composites with high volume fractions (>10%) of these fibres can be produced with significant increases in strength and toughness relative to the unreinforced matrix.

The use of micro-fibre reinforced cement systems for concrete repair has led to several improvements in these technologies that should have an impact on service-life of structures. Micro-fibres function by arresting and stabilizing matrix cracks before unstable growth occurs. There is evidence of pseudo-strain-hardening after the first matrix crack occurs [1].

Several types of micro-fibres have been investigated as candidates for reinforcing cement matrices. These include carbon, steel, polypropylene and wollastonite fibres. Recent work suggests that calcium carbonate micro-fibres with aspect ratios varying from 20-80 can be synthesized [2]. Solution-precipitation processes involving a variety of precursor salt solutions and blowing of CO<sub>2</sub> gas into suspensions are used. This paper describes the synthesis of high aspect ratio aragonite micro-fibres using CaCl<sub>2</sub> and Na<sub>2</sub>CO<sub>3</sub> starting solutions and crystal growth using a seeding technique. The performance of these microfibres as reinforcement in portland cement paste matrices is verified.

## Experimental

Micro-Fibre Synthesis. Reagent grade CaCl<sub>2</sub> and Na<sub>2</sub>CO<sub>3</sub> powders were used. Solutions, 0.1M CaCl<sub>2</sub> and 0.1 M Na<sub>2</sub>CO<sub>3</sub>, were prepared at 23°C. A litre of the Na<sub>2</sub>CO<sub>3</sub> solution was placed in a separation funnel. An equal amount of CaCl<sub>2</sub> solution was placed in a reaction vessel. The reaction vessel was placed in a temperature controlled water bath at 90°C. The Na<sub>2</sub>CO<sub>3</sub> solution was allowed to flow slowly into the reaction vessel for a period of 3-4 hours. The solution was filtered, the filtrate was washed and the crystals were oven-dried for 8 hours.

The precipitate was identified as aragonite using XRD analysis. The aragonite needles were referred to as the first generation micro-fibres. The procedure was repeated using a small amount of precipitate as a 'seed' crystal source. The seed crystal amount was about 1 mol/100 mol CaCl<sub>2</sub>. The micro-fibres produced were referred to as second generation. A

third and fourth generation micro-fibre were also produced. The average length of the aragonite crystals was 30, 50 70 and 100  $\mu$ m for the first to fourth generation products respectively. An SEM photomicrograph of fourth generation micro-fibres is shown in FIG. 1.

The following parameters were found to have some effect on the growth of the needle-like CaCO<sub>3</sub> crystals: reaction temperature, solution concentration, reaction time, mechanical stirring. For example reaction at 2°C produced no needle-like crystals. The needles produced at 90°C were more regular and longer than those at 80°C.

The conditions of formation at 90°C described above appeared to provide the most suitable fibres for inclusion in cement matrices. It was therefore decided to use aragonite micro-fibres synthesized at 90°C (0.1M solution concentration) as reinforcement for cement paste binders.

#### Micro-Fibre Reinforced Cement Paste

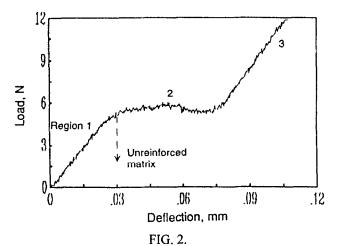
The performance of high aspect ratio aragonite fibres in a high performance cement-silica fume paste was evaluated in a few preliminary experiments. Silica fume (SF) pastes (water/cementitious solids = 0.35, 15% SF) containing 3, 6, 9 and 20 volume % aragonite fibres (synthesized at  $90^{\circ}$ C) were cast in the form of wafers and hydrated in lime saturated water. Beam specimens,  $42 \text{ mm} \times 3.5 \text{ mm} \times 3.5 \text{ mm}$ , cut from the wafer specimens, were tested in flexure at 14 days (3 point bending, MTS loading system) using displacement control. A classical reinforcing action was observed in many specimens at the 9% volume level. A typical load deflection curve is given in FIG. 2. Three regions are observed: region 1 where the load is carried by both fibres and matrix; region 2 where multiple fracture of the matrix occurs; region 3 where the load is carried by the fibres bridging cracks.

Not all results gave this idealized behavior due to non-uniform fibre dispersion in some instances. The presence of aragonite micro-fibres significantly increased the peak-load (by a factor of 2) in most cases.



FIG. 1.

SEM photomicrograph of high aspect-ratio aragonite microfibres aynthesized through reaction of 0.1M solutions of CaCl<sub>2</sub> and Na<sub>2</sub>CO<sub>3</sub>.



Typical load deflection curves for silica fume cement binders reinforced with aragonite micro-fibres and the unreinforced matrix. Fibre volume fraction, 9%.

## **Concluding Remarks**

High aspect ratio aragonite micro-fibres can be easily synthesized through reaction of calcium chloride and sodium carbonate. These micro-fibres can function as reinforcement in hardened cement paste matrices.

#### References

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- 2. Ota Y., Inui S., Iwashita T., Kasuga T. and Abé Y., J. Amer. Ceram. Soc., 78 (7), 1983 (1995).