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BASALT FIBER REINFORCED OIL WELL CEMENT SLURRIES

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Abstract

In the continuing search for hydrocarbons, oil wells are being drilled up to depths of 10.000 m, dealing with high temperatures and pressures that are quite uncommon in conventional concrete construction. Cementing such wells requires material satisfying strict specifications in terms of both rheological and mechanical properties. This paper reports some experimental results regarding the benefits of randomly distributed short basalt fibers on fracture toughness of oil well cement slurries. The mechanical response of a single fiber with diameter as small as 10 μm was studied, using a specially designed setup. The information obtained allowed to study the sensitivity of basalt to the alkali-silica reaction and to assess the optimal range for the fiber aspect ratio. Also, the influence of fiber content and length on both the slurry flowability and fracture toughness of the hardened composite was investigated. Although the fibers proved to be quite brittle, test results showed that an appropriate choice of fiber volume and length can noticeably increase fracture toughness of the cement matrix, without appreciably impairing the slurry's rheological properties. However, the long-term loss of ductility should be carefully studied, especially because of accelerated aging induced by the high temperature levels encountered in deep boreholes.

1. Introduction

Cement slurries are currently used for oil well completion and therefore they can be subjected to extraordinary elevated pressures and temperatures as high as 200°C. Nevertheless they have to fulfill several performance requirements, that is primarily, set control and excellent flow for pumpability [1]. Moreover, modern well construction techniques allow the producing formation to be selected by perforating the well casing through the cement sheath by means of bullets or shaped explosive charges. Hence,

growing attention is being paid also to mechanical properties such as tensile strength, impact resistance and fracture toughness which are usually unsatisfactory in plain cement paste. These properties are known to benefit from the addition of fiber reinforcement [2]. However, the severe performance requirements eliminate a priori most types of fiber that are commonly used in concrete construction. Most of the commonly used synthetic materials such as polypropylene or nylon cannot sustain elevated temperatures. Steel fibers markedly affect the slurry flowability. Materials that are too light or dense should also be discarded to prevent mix segregation. For these reasons, mineral fibers appear to be the most promising for this kind of application and, among these, basalt fibers are expected to gain popularity for their high strength, excellent resistance to high temperature and low cost. Basalt fiber is obtained by drawing the molten naturally occurring basalt rock into thin continuous glassy filaments (diameter = 9-15 μm). In spite of the very brittle nature of the material, the small size of the fibers allows to limit the dimension of inherently existing flaws so that tensile strengths as high as 1500-3000 N/mm can be obtained. In this respect, basalt glass is similar to common E-glass, but considerably more resistant to alkali attack, to the point that it could compete with the more expensive alkali-resistant glass [3].

The feasibility of using basalt fiber has been investigated in a recent study of the rheological and mechanical properties of oil well cement slurries at elevated temperatures [4]. The results showed that the fibers did not significantly affect the mix viscosity, even though no evident correlation was found with fiber content up to 1% by weight. Unexpectedly, no recognizable improvement of tensile strength and fracture toughness was observed either, presumably because of the pronounced fiber brittleness. As a matter of fact, the beneficial effect of basalt fiber on the toughness of cement composites is still questionable. As reported in the literature [5], basalt fiber can actually be corroded by an alkaline solution, yielding weak insoluble hydroxides that are deposited on the surface without contributing to strength. Hence, a sizeable strength reduction has been observed after a 2 month immersion in a 0.1M NaOH solution (pH = 13.1) at room temperature, or after only 2 hours at 100°C. On the other hand, the corrosion process could also be offset by a beneficial side effect, ascribable to blunting of the superficial microcracks. Regardless of the sensitivity to alkali exposure, cementitious composites containing either basalt or AR glass fiber usually exhibit a remarkable embrittlement in the long term [5, 6]. As the bond strength increases with the maturity of the cement matrix, the maximum load carried by each filament and the stress concentration on the bonded surface tend to increase, leading to brittle failure of fibers instead of the more ductile fiber pull-out. This phenomenon is likely to be accelerated under the hygro-thermal conditions encountered in deep oil wells and should be considered in material testing, as recommended by API for testing oil well cements [7].

To better understand the feasibility and effectiveness of basalt fiber reinforcement in improving the tensile behavior of oil well slurries, a research program has been undertaken at Columbia University's Carleton Laboratory. The principal results regarding the fiber response and composite performance are presented herein.

2. Experimental program and test methods

The test program was aimed to examine the possible fracture toughness enhancement of typical oil well cement slurries by adding different amounts of randomly distributed basalt fibers (0-2% by weight). The mechanical response of single pristine basalt filaments was studied and the effect of exposure to an alkaline solution analyzed. Based on the preliminary results, an optimal range for the aspect ratio was determined and several cement slurries were prepared, containing variable amounts of fibers of different lengths. The flowability of the mixes was compared and the toughness of the hardened composite studied, in order to find a good compromise between these two opposing demands. Finally, the effect of accelerated curing was checked to ascertain the ability of the composite to retain its properties at elevated temperatures.

2.1 Tests on single basalt fibers

In a preliminary stage of the study, the basalt fibers produced by two different companies (type A and B in the following) were examined and compared for mechanical strength and alkali corrosion resistance. The fibers were immersed in 0.1M NaOH solution (pH = 13.1) at room temperature (24°C) for 30 or 60 days, then removed, washed in running distilled water and allowed to dry in air. Both virgin and corroded fibers were observed through the optical microscope and their diameter measured by means of a digital camera and a pixel count algorithm (sensitivity = 0.15 μm/pixel).

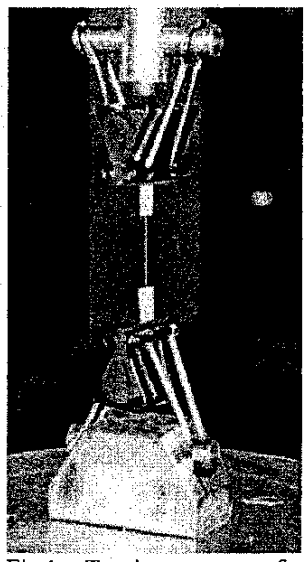

Fig.1 – Tension test setup for single basalt fibers.

To accurately determine the maximum tensile load carried by a thin basalt filament (about 0.2 N only), a simple but reliable device was used (Fig. 1). A weight greater than the fiber strength is placed on the platen of a laboratory scale and the fiber is clamped to it. After zeroing the scale indicator, the fiber is progressively pulled up to failure and the maximum recorded negative weight is taken as the fiber strength. Batches of 12 fibers for each case have been analyzed to compensate for the scatter of the results.

Other experiments, not reported herein, were performed to measure the average bond strength between a single fiber and the cement matrix and to assess the maximum fiber length that guarantees fiber pullout instead of brittle tensile failure (i.e. the critical length - $l_{cr} \cong 1$ mm). It has to be noted that the optimal fiber length in terms of energy dissipation is generally greater than the critical length, because of randomness of fiber distribution and uneven compaction of the fiber reinforced matrix.

Table 1: Plain cement slurry mix design and fiber dosages.

Component	Parts (by weight)
Type H Cement	100
Proprietary Admixture	0.5
Water	38

Fiber Length (mm)		1.0	1.9	3.1	5.5
Aspect Ratio		90	170	275	490
Fiber Content (by weight)	0.5%	x		x	
	1.0%	x	x	x	x
	2.0%	x		x	

2.2 Mix design and tests on fresh cement slurries

On the basis of the aforementioned results, "type A" fiber was selected and cut to strictly controlled lengths in the range of 1.0-5.5mm. These correspond to aspect ratios of 90-490 (av. diam. = 11.2 μm). Different amounts of chopped fibers were then added to the cement slurry, as summarized in Table 1. The type H cement (API designation) is a basic portland cement that can be used for a wide range of well depths and temperatures, in conjunction with accelerators, retarders or viscosity control agents. To prevent any damage to the fibers during the slurry preparation, only the plain slurry constituents were mixed with a high-speed blade-type mixer, whereas the composite was stirred manually after fiber addition. To assess the effect of fiber addition on slurry rheology, a plastic mould resting on a smooth glass plate was filled with the fresh mix and then lifted. The average radius of the flattened cement paste disk that forms on the glass plate was then taken as a measure of the slurry's flow capability (see insert in Fig. 4).

2.3 Tests on hardened cement slurries

The fiber reinforced slurries were used to prepare groups of 4 small beams (25.4x25.4x140 mm) to be tested for toughness in three-point bending over a 101.6mm span (Fig. 2a). The curing procedure called for keeping the samples in the moist room (24°C – 100% U.R.) for 2 weeks and allowing them to settle in the laboratory environment for 1 day before testing. To check the effects of accelerated aging, a second batch of samples was cast and the molds were covered with a sealed steel plate and dipped in hot water (80°C) for 24 hours. After cooling, the samples were kept at room temperature for 1 week prior to testing. The beams were tested using a 10kN MTS closed-loop hydraulic press driven by the control signal provided by an extensometer at the bottom string (strain rate = 0.2μm/s).

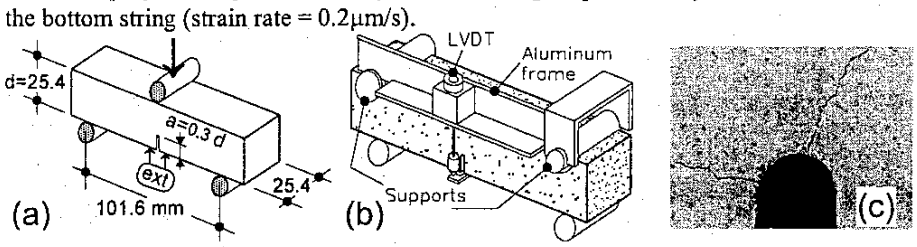

Fig. 2 – Toughness test setup: (a) specimen geometry, (b) special frame for net deflection measurement and (c) drying shrinkage cracks at the notch tip.

A special frame was attached to measure the net mid-span deflection of the specimen, from which the fracture energy would be determined (Fig. 2b).

To improve both test stability and repeatability, a small notch (a/d = 0.3) was cut into the mid-span section. How long prior to testing the notch was cut had a considerable effect on the test results. As a matter of fact, cement slurries are prone to early-age cracking due to drying shrinkage. Direct observation through the microscope showed that a network of thin cracks formed on the sample surface. Moreover, 1-2 inclined cracks developed at the notch tip within one hour after cutting the notch, caused by the induced hygral gradients (Fig. 2c). Hence, the first-crack load, needed to compute the toughness indexes according to ASTM C1018 [8], could be considerably affected by this phenomenon. For this reason, an alternative approach, based on fixed reference thresholds of the specimen deflection, has been adopted to quantify the toughness of the composite, as suggested in the recent RILEM Recommendations [9]. The deflection thresholds shall correspond to average crack openings that are relevant for the serviceability and ultimate limit states. However, smaller crack openings have been considered herein, since more attention is usually paied to water and gas tightness of these fine-grained composites.

3. Experimental results and discussion

3.1 Strength and alkali resistance of basalt fiber

The mechanical behavior of basalt fiber in tension can be regarded as perfectly elastic-brittle and is characterized by a remarkably high strength (Fig 3a). This strength is greatly affected by exposure to NaOH solution (Fig. 3a), but the reduction is comparable to that of commonly used AR glass fiber [3]. It is interesting to note that the variance of strength data was less for the fiber that proved to be less sensitive to alkali attack (Fig. 3b). This evidence seems to confirm the beneficial side effect of corrosion in blunting the random inherent defects on the fiber surface. Because of its better chemical resistance, only fiber type A was considered in the remaining part of the research.

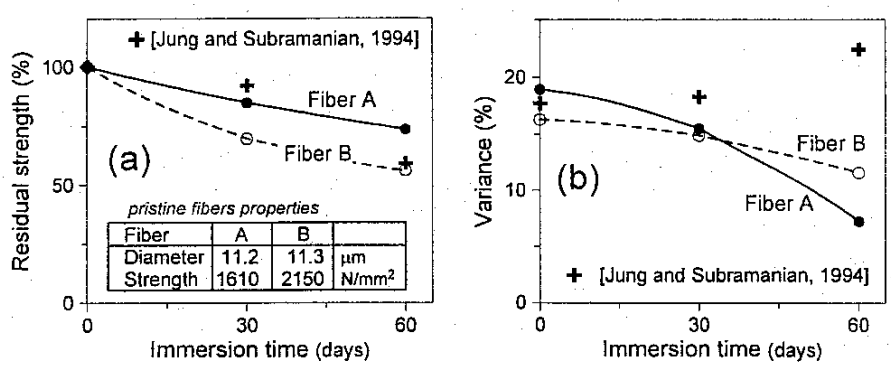

Fig. 3 – Effect of exposure to the alkaline solution on (a) residual basalt fiber strength and (b) strength variance.

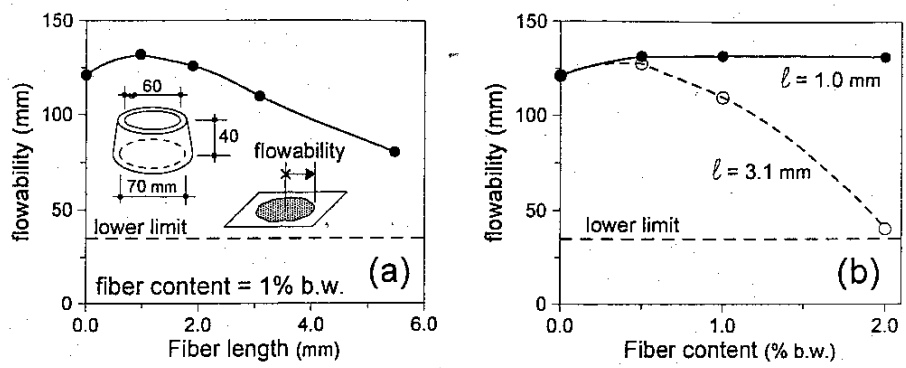

Fig. 4 – Effect of (a) fiber length and (b) fiber content on slurry flowability.

3.2 Slurry flowability

The effect of short basalt fibers on slurry flowability turned out to be very variable, depending upon both fiber length and content (Fig. 4). On the one hand, basalt fibers are smoother than cement grains and are expected to flow better. On the other hand, increasing amounts of fibers make them more likely to collide and restrain the motion of particles. The fiber length strongly influenced the rheology of the composite. The flowability was slightly improved by adding relatively short fibers in any dosage within the considered range (0.5-2% by weight), whereas it was dramatically reduced in the case of increasing amounts of longer fibers.

3.3 Fracture toughness of hardened cement pastes

An effective representation of fiber benefits on the tensile behavior of cement pastes is the load-deflection curve (Fig. 5). Provided the notch is cut shortly before running the test, the curves are quite similar up to the peak load, with no recognizable stable crack propagation. In the softening branch, fibers cause a pronounced improvement of the composite toughness, especially fibers of greater length.

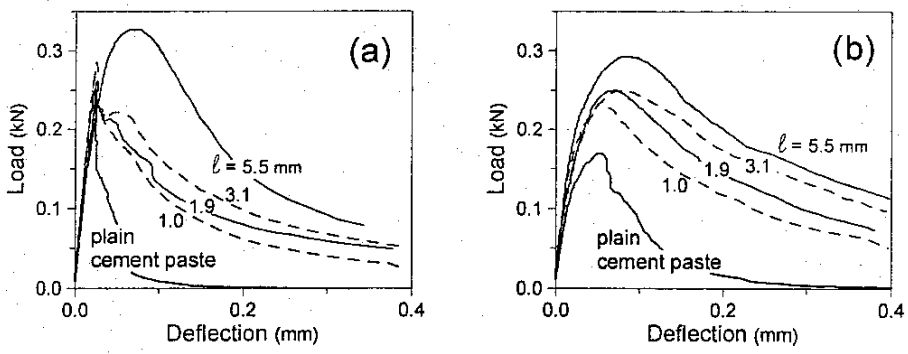

Fig. 5 – Load deflection curves: (a) samples tested immediately after cutting the notch and (b) samples tested 24h after cutting the notch (fiber content = 1% b.w.).

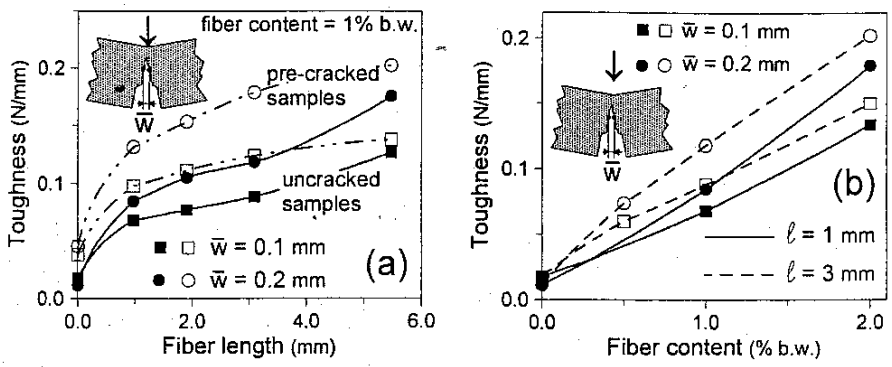

Fig. 6 – Specific fracture energy (toughness) up to prescribed average crack openings: (a) effect of fiber length and (b) effect of fiber content.

Actually, 5.5mm long fibers exhibit a strain-hardening behavior, which offsets the sharp instability at the peak. In the case of tests run 24 hours after cutting the notch, allowing drying cracks to develop, a markedly non-linear loading branch can be recognized, accompanied by a considerable increase in dissipated energy (Fig. 5b). In fact, the micro-cracks start propagating at quite low load levels and branch into a more tortuous and extended crack surface. Similar effects were observed in the case of different fiber contents. These were comparable to the effect of increasing fiber length. To better summarize the toughening effect provided by different basalt fiber reinforcements, the areas under the load-deflection curves up to assigned deflection levels were computed. The deflection thresholds correspond to average crack openings of 0.1 mm and 0.2 mm (Fig. 6). As for the effect of fiber length, it can be noted that the steepest toughness increase occurred in a range up to the critical fiber length ($l_{cr} \cong 1$ mm), whereas a more gradual improvement was recorded for longer fibers. As expected, the effect of fiber content is substantially linear. Summing up, even if longer fibers prove to be more efficient, short fibers can lead to similar results if added in larger amounts.

3.4 Effect of hot curing

A totally different behavior was observed in the case of hot-cured slurries (80°C for 24h in sealed conditions) (Fig. 7). The cement pastes got so strong and brittle that in most cases it was not possible to follow a stable descending branch. Direct observation of the crack faces showed that the majority of fibers were broken instead of pulled out. This is probably due to improved bond strength (i.e. shorter critical length) and higher alkaline corrosion rate at elevated temperature.

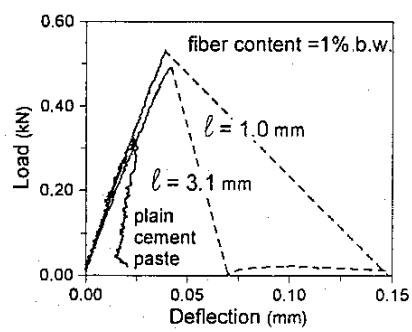

Fig. 7 - Brittle failures in bending of the hot-cured samples.

4. Concluding remarks

The objective of this paper was to ascertain the feasibility of randomly distributed basalt fiber reinforcement to improve the fracture toughness of oil well cement slurries. Nine types of slurries reinforced with fibers of different lengths and contents were tested for rheological and mechanical performance. Results indicate that basalt fiber performance in terms of mechanical strength and alkali resistance is comparable to that of common AR glass fiber. The composite flowability, which is a crucial factor for slurries to be pumped into deep bore holes, is even improved by short fiber (l = 1mm) within the examined dosage range (0-2% by weight), whereas it is markedly reduced in the case of longer fiber. As for fracture toughness, increasing fiber amounts and lengths improved the composite response considerably. Hence, it appears that the optimum choice for the considered application is represented by a relatively high dosage of short fiber, given also the relatively low cost of basalt fiber. However, the composites proved to be extremely sensitive to a hot curing procedure. This means that the improvement of mechanical properties in tension is not likely to be retained in the long term or in the case of deep well cementing jobs. This phenomenon can be ascribed to the stronger bond and the accelerated alkaline corrosion that develop at elevated temperature. Nevertheless, further studies are needed to assess the relative importance of the mechanical and chemical causes and to prevent their negative effects.

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