JOURNAL OF MATERIALS SCIENCE **39** (2004) 1727–1733

Tensile tests on plain and fibre reinforced geothermal cements

M. L. BERNDT
Department of Energy Sciences and Technology, Brookhaven National Laboratory, Upton, NY 11973, USA
E-mail: allan@bnl.gov

C. E. MANCINI
Department of Materials Science and Engineering, Stony Brook University, Stony Brook, NY 11794, USA

The tensile behaviour of different geothermal well cement formulations was investigated in order to determine how these materials perform under such loading and how tensile capacity can be improved. The influence of latex, perlite and fibres on the load-displacement relationship of the cements was measured on notched cylinders. The fracture surfaces were examined to further elucidate failure mechanisms. Unreinforced cements exhibited linear elastic behaviour to different degrees and failed in a brittle manner. Cements reinforced with either carbon (150 μm) or steel (1–3 mm) microfibres required greater loads for failure. However, the microfibres did not provide any major improvement in ductility. Addition of 13 mm steel fibres to the cements resulted in both strengthening and transition to ductile behaviour. Inclusion of the types of fibres studied in this work in cements offers potential benefits in maintaining the integrity of geothermal wells when tensile stresses are involved.

1. Introduction

Geothermal well cements are used to (a) seal the annulus between steel casing and surrounding formation and (b) provide structural support and corrosion protection to the casing. Once a well is constructed these cements are subjected to chemically aggressive geothermal fluids in addition to stresses induced by operating and environmental conditions. The type and magnitude of stresses are complex and depend on a number of factors [1]. Compressive, shear and tensile stresses are involved and these are controlled by well operating parameters such as temperature and pressure, far-field stresses and elastic properties of the surrounding formation. Of particular concern is the development of significant tensile stresses [1, 2] and the ability of geothermal well cements to withstand such stresses. Traditional well cements are typically weak in tension and brittle. Therefore, potential improvement in the tensile strength and ductility of well cements is being sought to reduce failures and ultimately increase well life. While stress regimes in operating geothermal wells are more complex than uniaxial tension, study of tensile properties is fundamental to understanding how improvements in cement structural performance can be achieved.

In order to investigate the tensile behaviour of well cements and the effect of additives on this behaviour, it was necessary to select a suitable test method. Previous research used splitting tensile tests to indirectly measure tensile strength [3]. However, a direct method was desired to enable study of the uniaxial tensile load-displacement characteristics of the cements. Direct tensile tests on cementitious materials are difficult to perform owing to problems associated with ensuring purely tensile stresses and avoiding inadvertent introduction of localized stresses at grips [4]. Tensile tests on concrete have been performed using different end bonding methods and loading arrangements [e.g., 5–12]. Different control methods during loading are also possible and uniaxial tensile tests are often conducted at constant crack mouth opening displacement rate from gauge feedback. The objectives of the work described in this paper were to apply uniaxial tensile tests to plain and fibre reinforced well cements and to compare the resultant behaviour of these materials. Other mechanical and thermal properties of the cements investigated have been reported previously [2, 13–15].

The geothermal cement formulations studied were based on a commonly used mix comprising of API Class G cement, silica flour and water. Low density cements were also investigated as these are used for cementing weak formations. Such cements may incorporate lightweight additives or use nitrogen and a foaming agent to create a porous structure. Latex-modified cements were also included in the study. Latex is used in geothermal cement mixes to improve durability through reduction in permeability and enhanced resistance to chemical attack. Furthermore, improvement in the

0022–2461

adhesion between well cement and steel casing is expected. The mechanisms by which latex improves bonding, and creates films within the hydrated cement phase is described by Chandra and Ohama [16]. With regard to mechanical properties, addition of latex tends to increase the tensile and flexural strengths [16] and decrease the elastic modulus of cementitious materials [17]. A further benefit of latex for the cement slurries of interest is the improvement in flow properties and this enables reduction of water/cement ratio.

Several different fibre types were investigated in this work for potential improvement in tensile load and strain capacity. These included carbon and steel microfibres and 13 mm long steel fibres. The resultant composite materials consisted of stiff, strong fibres embedded in a brittle cement matrix. Owing to the need to maintain pumpability of the geothermal well cements, the length and volume fraction of fibres that could be added were limited. Therefore, the fibre reinforced cements studied either contained relatively short fibres at high volume fractions or longer fibres at low volume fractions.

2. Experimental procedure

A range of different cements was tested. The baseline mix was API Class G cement/40% silica flour with a density of 1.92 g/cm^3 that is frequently used to complete geothermal wells. The water/cement ratio of this mix was 0.55. Latex-modified and lightweight cements were also tested. The latex used was styrene butadiene rubber supplied by BJ Services (BA-86L) with a polymer solids content of 46%. A polymer solids/cement ratio of 0.10 by mass was used, thus giving a water/cement ratio of 0.46. Lightweight mixes were produced by inclusion of perlite (Sproule WR-1200). A higher water/cement ratio of 0.89 was required in this mix to maintain pumpability. Table I gives the mix proportions of the plain (unreinforced) cements.

One type of steel fibres tested was straight, round, 13 mm long brass coated drawn wire with a diameter of 0.16 mm (OL 13/0.16, Bekaert Corporation). These were added to the standard, and latex-modified cements at volume fractions of 0.5 and 1%. The second type of steel fibres was fine microfibres 1–2 mm long (E-281, American Metal Fibers Inc.) and these were used at a volume fraction of 5% in the standard cement. The mild steel microfibres were ribbon shaped and had a cross section of approximately 10 by 25 μm. Milled polyacrylonitrile-derived carbon microfibres with a length of 150 μm and diameter of 7.2 μm (Panex 33, Zoltek Corporation) were added to the standard cement at a volume fraction of 2%. The volume fractions were determined from previous work [3] to optimize indirect tensile strength while maintaining pumpability.

The procedure used to prepare the test specimens first involved premixing of bentonite with water in a high shear blender. The remaining mixing was performed in a planetary mixer and the order of addition to the water-bentonite fluid was as follows: dispersant, latex (if used), perlite (if used), silica flour, cement and fibres. Six specimens for each mix were cast in cylindrical moulds 52 mm diameter and 104 mm high. The specimens were removed from the moulds after 24 h and then cured in water for 28 days at 52°C.

The ends of the specimens were cut with a diamond saw to remove laitance and to prepare surfaces perpendicular to the cylinder axis. Cylindrical aluminium caps were then glued to the specimens using epoxy adhesive. Grit blasting the specimen ends was found to improve adhesion. Roller bearing chains were used to link the caps to the testing machine following ASTM D 2936. A displacement measuring device was attached to the specimens. This consisted of two independent aluminium yokes placed around the specimen circumference that were each fastened with three mounting points screwed into the specimen surface. The yokes were positioned equidistant from the specimen midheight to give a gauge length of 70 mm. LVDTs were mounted at diametrically opposite locations. The tests were performed in an Instron Model 1321 universal testing machine at 2 mm/min.

Initial tests on cylinders without notches were unsuccessful since failure generally occurred at the bonded end caps. Subsequently, notches were cut around the circumference of the specimens at the midheight using a diamond cut-off saw. These notches were saddle shaped, 3.2 mm wide and 8.0 to 9.0 mm deep. The greater depths were required on the fibre reinforced specimens to cause failure at the notch. The test specimen configuration is shown in Fig. 1.

TABLE I Mix proportions of plain cements by mass

Mix type	Cement	Silica flour	Water	Latex	Perlite	Bentonite	Dispersant
Standard	1	0.4	0.55	–	–	0.034	0.012
Latex	1	0.4	0.343	0.217	–	0.01	0.006
Perlite	1	0.4	0.89	–	0.085	0.02	0.012

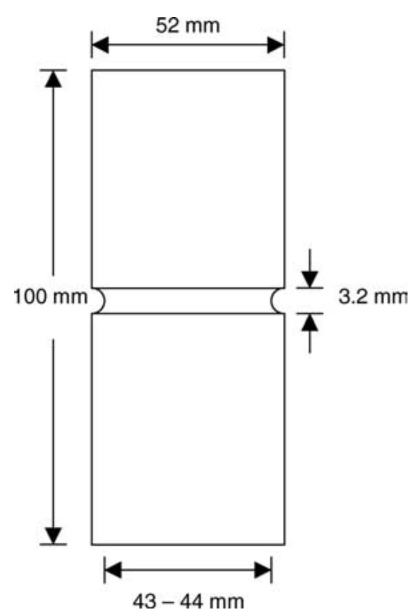

Figure 1 Schematic diagram of tensile test specimen.

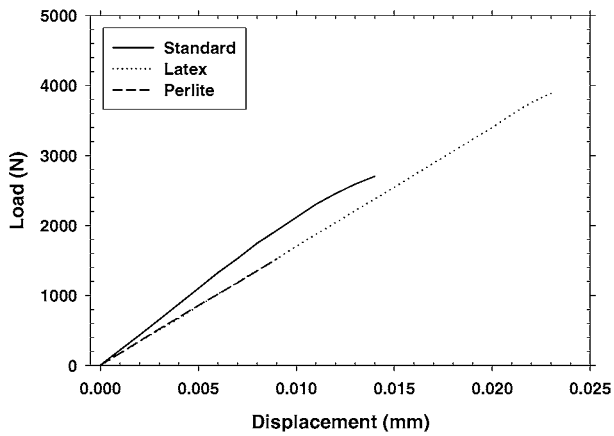

Figure 2 Load-displacement curves for plain cements.

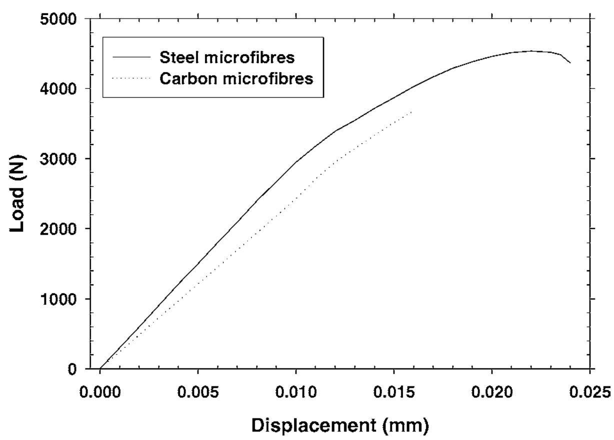

Figure 3 Load-displacement curves for microfibre reinforced cements.

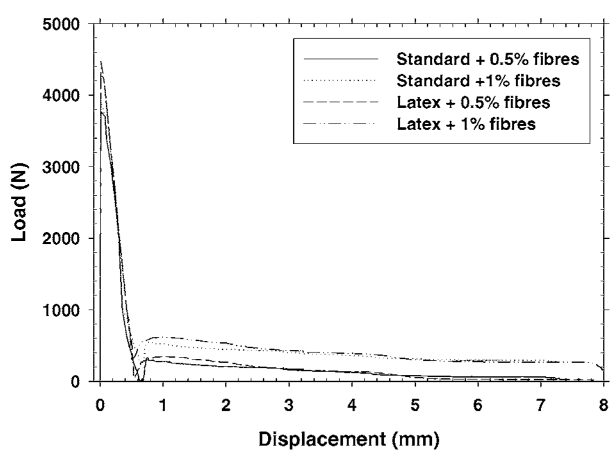

Figure 4 Load-displacement curves for 13 mm steel fibre reinforced cements.

3. Experimental results

The results of the tensile tests are presented as load-displacement (or elongation) curves. Fig. 2 shows examples of such curves for the unreinforced standard, latex-modified and lightweight cements. Examples of the results for the cements reinforced with microfibres and 13 mm steel fibres are given in Figs 3 and 4, respectively. In the curves shown the displacement was calculated as the average between the two LVDTs. It is noted, however, that some of the specimens exhibited bending behaviour and one LVDT often showed much greater displacement than the other. In such cases, cracks tended to initiate on one side of the notch rather than uniformly through the cross section. Table II compares the peak load and elastic modulus values obtained for the different cements. The modulus was calculated from nominal stress at the minimum cross-section divided by the strain as measured over a gauge length of 70 mm in the linear elastic region.

The unreinforced cements failed in a brittle manner. Cements containing carbon and steel microfibres were also brittle. This behaviour may also be influenced by the test procedure of using constant crosshead speed rather than closed loop control. Ductility was most influenced by inclusion of 13 mm steel fibres. In the latter case, following initial crack formation the steel fibre reinforced cements continued to bear load as multiple matrix cracking, fibre alignment, disbondment and pullout occurred. Particles of cement matrix surrounding the fibres popped out as the tests progressed. Visual observations of the steel fibre reinforced specimens during testing and after failure indicated elastic deformation of fibres as they aligned with the axis of load application and re-aligned to the original orientation after failure.

Scanning electron microscope (SEM) images of the fracture surfaces for the fibre reinforced cements are depicted in Figs 5 to 13. The material with 2% volume fraction carbon microfibres in Fig. 5 shows remaining fibres and channels where fibres have been pulled out of the matrix. The fibre surfaces are relatively smooth. Fig. 6 is a higher magnification view of the carbon microfibre reinforced cement and shows a fibre that has fractured (arrowed) and partially pulled out while leaving the remaining portion embedded in the matrix. Cracking in the matrix under the pulled out fibre is also evident. Fibre fracture was observed to be a relatively frequent feature in this material. Thus, failure of the carbon microfibre reinforced cements includes both fibre fracture and pullout mechanisms. Figs 7 and 8 illustrate the fracture surface of the material containing

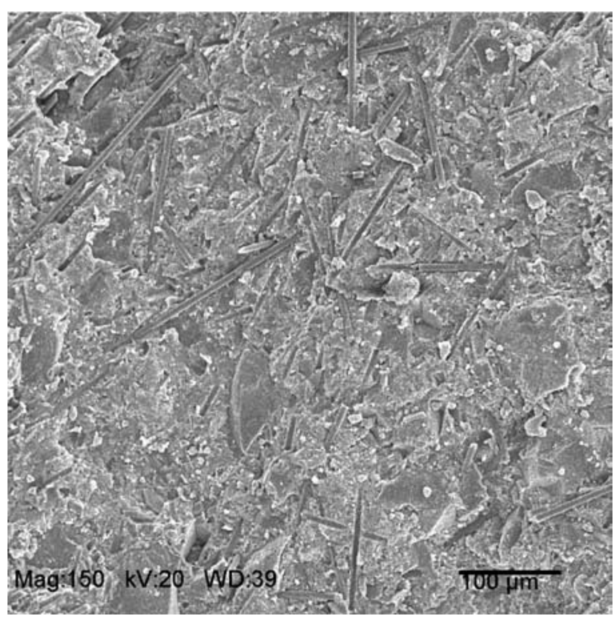

Figure 5 Fracture surface of carbon microfibre reinforced cement.

TABLE II Peak load and elastic modulus results

Mix type	Fibres	Notch depth (mm)	Peak load (N)	Elastic modulus (GPa)
Standard	None	8	2751 ± 258	9.72 ± 0.88
Latex	None	8	3785 ± 243	7.80 ± 0.85
Perlite	None	8	1140 ± 112	7.78 ± 0.67
Standard	2% carbon microfibres	9	3499 ± 392	11.8 ± 1.30
Standard	5% steel microfibres	9	4762 ± 218	14.3 ± 0.92
Standard	0.5% 13 mm steel fibres	9	3368 ± 305	10.5 ± 0.86
Standard	1% 13 mm steel fibres	9	3855 ± 298	10.7 ± 0.82
Latex	0.5% 13 mm steel fibres	9	3920 ± 334	8.79 ± 0.78
Latex	1% 13 mm steel fibres	9	4468 ± 312	8.91 ± 0.79

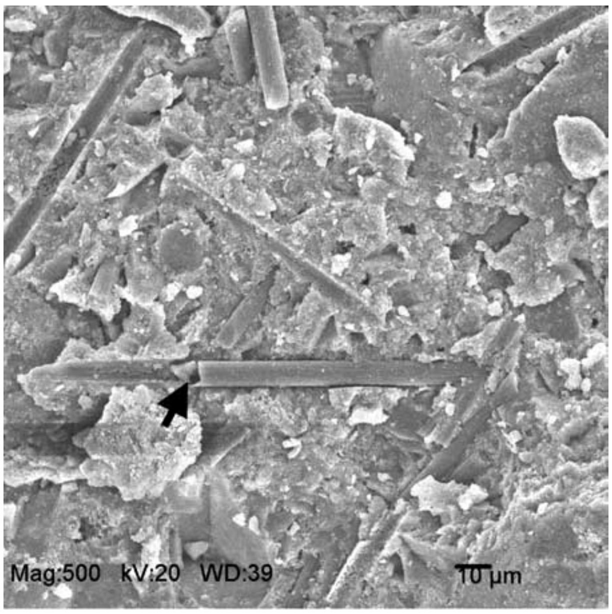

Figure 6 Fracture surface of carbon microfibre reinforced cement showing fractured fibre and partial pullout (arrowed).

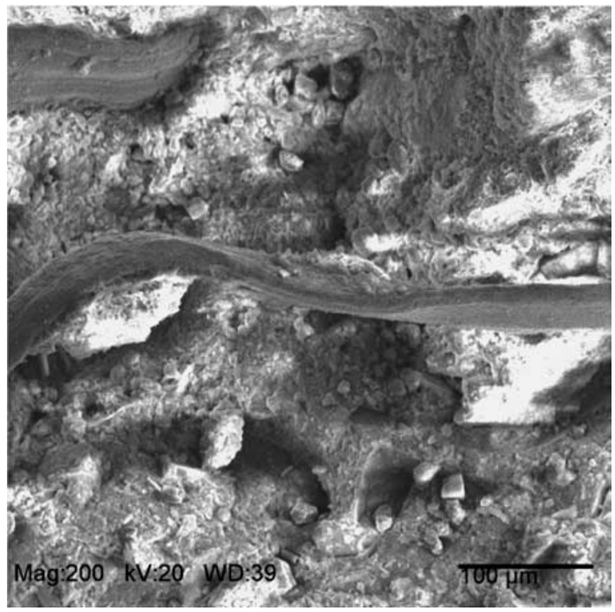

Figure 8 Steel microfibres in cement matrix.

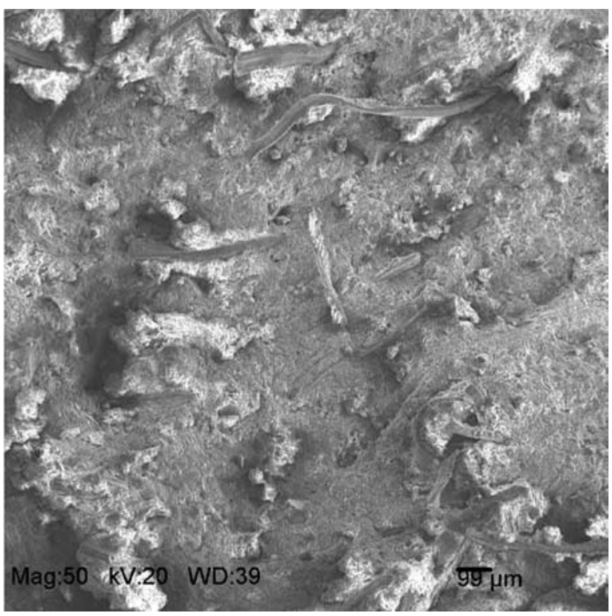

Figure 7 Fracture surface of steel microfibre reinforced cement.

5% volume fraction steel microfibres. The ribbon shaped microfibres are twisted and give a more three dimensional reinforcement than the other fibres tested. The fracture surface was very rough compared with the other tested materials and this is probably due to the extensive crack path deviation incurred by the microfibres. Fig. 9 shows twisted fibres with calcium hydroxide deposits on the surface.

The fracture surface of the cement reinforced with 0.5% volume fraction 13 mm steel fibres is shown in Fig. 10. Pullout of fibres at different orientations is evident. Areas of matrix pop out surrounding the base of the projecting fibres and interfacial disbondment are also observed. Fig. 11 gives a view of reaction products on the fibre surface and a channel left from fibre pullout. The fibres in the latex-modified cement had smoother surfaces than those in the material without latex as indicated in Fig. 12. This figure also shows adhesion of the matrix to the base of the fibre as opposed to pop out observed with longer projecting fibres.

4. Discussion

The load-displacement curves for the unreinforced cements in Fig. 2 indicate different degrees of linear elastic behaviour. In the case of the standard Class G/40% silica flour cement, this behaviour was maintained up to approximately 70% of the peak load. The lightweight cement containing perlite exhibited linear elastic behaviour until failure. Similarly, the response of the latex-modified cement was also predominately linear elastic. The elastic modulus was highest for the

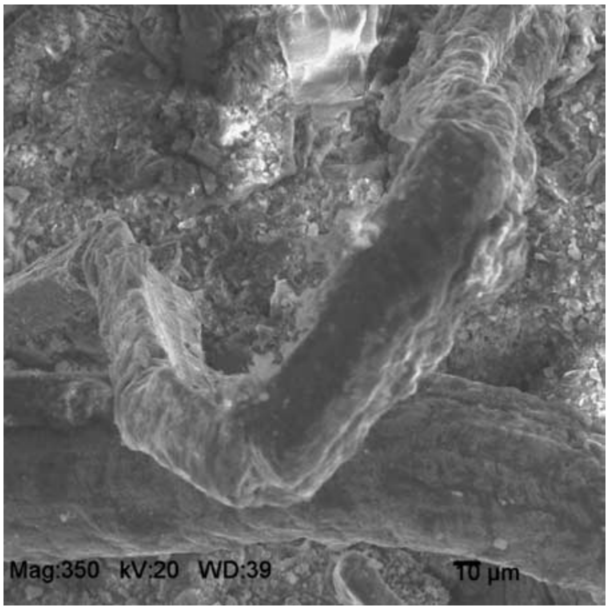

Figure 9 Surface features of steel microfibres.

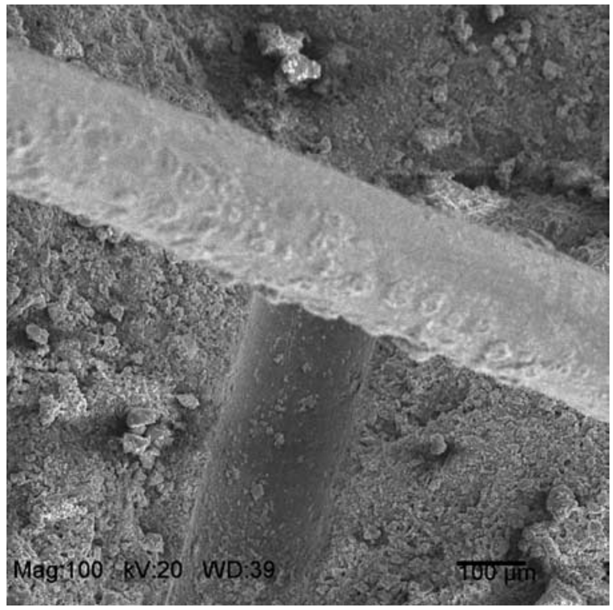

Figure 11 Surface features of 13 mm steel fibre and pullout zone.

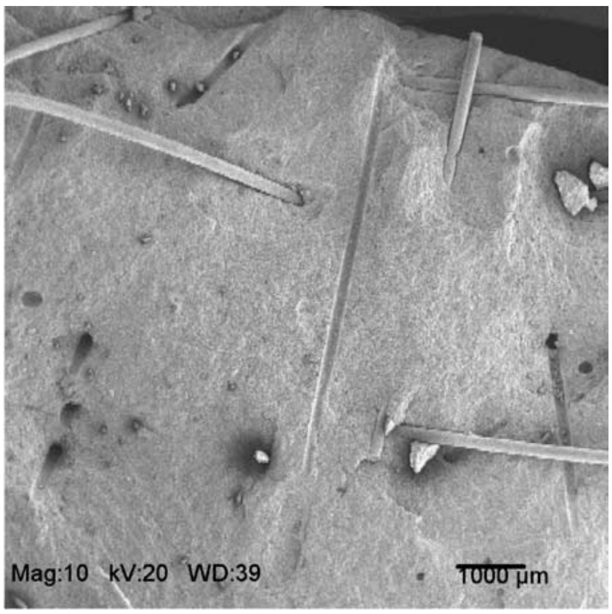

Figure 10 Fracture surface of cement reinforced with 13 mm steel fibres.

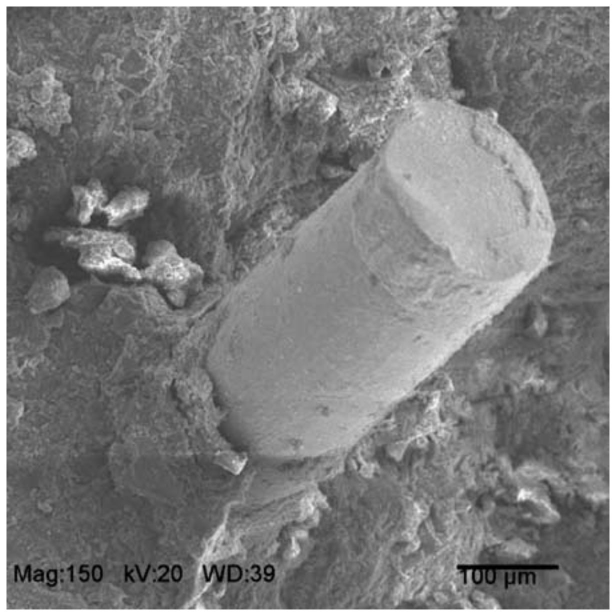

Figure 12 Fracture surface of latex-modified cement reinforced with 13 mm round steel fibres showing anchored matrix and surface features of fibre.

standard cement and was similar for the perlite- and latex-modified cements. The greatest displacements and peak loads were measured for the latex-modified cement indicating some advantages of using this additive when tensile capacity is desirable. In addition, the lower water/cement ratio of the latex-modified cement contributed to the better performance of this material. The corresponding low values for the perlite-modified cement are due to the higher water/cement ratio as well as the perlite itself.

Addition of all types of fibres to the standard plain cement increased the peak load. The steel microfibres imparted the greatest increase and this is probably due, in part, to the high volume fraction used. This finding further correlates with the observed rough fracture surface and tortuous crack path. The SEM images indicated interfacial calcium hydroxide on the steel microfibre surfaces (Figs 7–9) whereas the carbon microfibres lacked this (Figs 5 and 6). Observation of fibre fracture in the cements containing carbon microfibres suggested utilization of the fiber strength. Some strain hardening type behaviour was associated with the steel microfibres in addition to a minor amount strain softening as shown in Fig. 3. The load-displacement curves of the microfibre reinforced cements showed linear elastic behaviour up to approximately 70% of peak load, which was the same as that for the unreinforced cement. Elastic modulus was increased by the addition of both types of microfibres. The increase in modulus of the microfibre reinforced cements may impact the nature of stresses developed in the cement annulus of geothermal wells. Such stresses are influenced strongly by the ratio of the cement modulus to that of the surrounding rock or formation [1, 15].

Fig. 4 indicates that addition of 0.5% volume fraction 13 mm steel fibres markedly changed the post-peak tensile behaviour of the cements. Examination of the load-displacement curves shows that after peak load was achieved there was a significant decrease in load. A rebound (or "snap back") and an extensive tail region followed this decrease and were associated with elastic deformation of disbonded fibres and progressive fibre pullout. Pullout also involved failure within the anchoring matrix and its extent depended on the fibre orientation. From Fig. 4 it can be seen that the load-displacement curves were similar in form for the standard and latex-modified cements containing 13 mm fibres. Furthermore, the addition of latex had some benefit in terms of peak load. Assuming a normal distribution and equal variances, a t-test at the 5% level of significance indicated that the mean peak load of the latex-modified mix containing 0.5% steel fibres was higher than the mean for the same material without latex. However, ductility and post-peak residual strength were not significantly affected by latex. Examination of the fibre surfaces in the fractured specimens revealed that latex altered the interfacial microstructure. It was apparent that latex improved fibre to matrix bonding and, consequently, the load required for failure was increased. Hence, there is some synergy between latex and the steel fibres. Interestingly, latex did not enhance the splitting tensile strength of either plain or fibre reinforced cements in previous tests [3].

Increasing the volume fraction of 13 mm steel fibres from 0.5 to 1% resulted in statistically significant increases in mean peak load for both the standard and latex-modified cements as determined from a t-test at the 5% level of significance. The general characteristics of the load-displacement curves were similar for the materials containing 0.5 and 1% steel fibres. However, an increase in the loads carried in the post-peak region was evident when fibre volume fraction was increased to 1%. With regard to the effect of fibres on elastic modulus, t-tests at the 5% level indicated that the mean elastic moduli for the cements with 0, 0.5 and 1% fibre volume fractions were not significantly different for either the standard or latex-modified mixes. Thus, the fibres did not result in a significant increase in elastic modulus. The presence of latex in the 13 mm steel fibre reinforced mixes did cause a significant decrease in mean elastic modulus at both fibre volume fractions studied.

Some improvements in the tensile test method used in this work are worthy of consideration. For example, the use of fixed end platens may be preferable in order to reduce non-uniform displacement and cracking. More information on the post-peak behaviour of the fibre reinforced cements may be obtained by closed loop control of the tests at constant displacement rate as measured directly from LVDTs or at constant crack mouth opening displacement rate measured from clip gauges at the notch. Such arrangements have been used in studies on circumferentially notched cylinders and double edge notched prisms of fibre reinforced concrete [6–12]. These modifications would enhance the understanding of the response of different geothermal well cement formulations to purely uniaxial tensile loads.

5. Conclusions

Load-displacement curves obtained in tension revealed the influence of additives and fibres on behaviour of geothermal well cements under this type of loading. Plain (unreinforced) cements showed expected brittle behaviour. Addition of latex improved the peak load required for failure and the ultimate displacement at failure. Fibres were found to have the greatest affect on tensile properties. Despite the short length of carbon and steel microfibres, benefits in terms of load bearing capacity were achieved. However, tensile strain capacity was only improved to a minor degree by the microfibres. The mode of failure for the carbon microfibre reinforced cements included both fibre fracture and pullout. Inclusion of 13 mm steel fibres at 0.5 and 1% volume fractions imparted significant ductility to the cements as well as increasing peak load. Residual load bearing capacity was provided by the steel fibres and increased with fibre volume fraction. Incorporation of latex in the steel fibre reinforced cements improved peak load due to enhanced bonding with the cement matrix. Addition of steel or carbon microfibres resulted in increased elastic modulus of the cements, with 5% steel microfibres giving the stiffest material. The 13 mm fibres did not significantly change the mean elastic modulus at the volume fractions studied. The results indicate how the tensile properties of well cements can be modified through the use of latex and different types of fibres and the respective effectiveness of such materials in improving load and strain capacity.

References

1. A.J. PHILIPPACOPOULOS and M. L. BERNDT, *Geothermics* **31** (2002a) 657.
2. *Idem.*, *Soc. Petrol. Engng. SPE* 77755 (2002b).
3. M. L. BERNDT and A. J. PHILIPPACOPOULOS, *Geothermics* **31** (2002) 643.
4. A. M. NEVILLE, "Properties of Concrete," 4th ed. (John Wiley and Sons, New York, 1996) p. 595.
5. B. BARRAGÁN, R. GETTU, R. F. ZALOCHI, M. A. MARTÍN and L. AGULLÓ, in Proceedings of the Fifth International RILEM Symposium, Lyon, September 2000, edited by P. Rossi and G. Chanvillard (RILEM, Cachan, 2000) p. 441.
6. L. BIOLOZI, A. MEDA, G. ROSATI and G. L. GUERRINI, in Proceedings of the Fifth International RILEM Symposium, Lyon, September 2000, edited by P. Rossi and G. Chanvillard (RILEM, Cachan, 2000) p. 557.
7. S. CANGIANO, G. A. PLIZZARI and P. COLOSIO, in Proceedings of the Fifth International RILEM Symposium, Lyon, September 2000, edited by P. Rossi and G. Chanvillard (RILEM, Cachan, 2000) p. 399.
8. M. DI PRISCO, R. FELICETTI and F. IORIO, in Proceedings of the Fifth International RILEM Symposium, Lyon, September 2000, edited by P. Rossi and G. Chanvillard (RILEM, Cachan, 2000) p. 233.
9. C. FAILLA, G. TONIOLO and L. FERRARA, in Proceedings of the Fifth International RILEM Symposium, Lyon, September 2000, edited by P. Rossi and G. Chanvillard (RILEM, Cachan, 2000) p. 253.
10. Q. LI and F. ANARSI, *ACI Mater. Journal* **97** (2000) 49.
11. M. MARAZZINI and G. ROSATI, in "Structural Applications of Fiber Reinforced Concrete," edited by N. Banthia, C. MacDonald

and P. Tatnall (ACI SP-182, American Concrete Institute, Farmington Hills, 1999) p. 29.
12. K. NOGHABAI, in "Structural Applications of Fiber Reinforced Concrete," edited by N. Banthia, C. MacDonald and P. Tatnall (ACI SP-182, American Concrete Institute, Farmington Hills, 1999) p. 109.
13. A. J. PHILIPPACOPOULOS and M. L. BERNDT, *Geoth. Res. Council Trans.* **24** (2000) 81.
14. *Idem.*, Brookhaven National Laboratory Report 68266 (2001a).
15. *Idem.*, *Geoth. Res. Council Trans.* **25** (2001b) 119.
16. S. CHANDRA and Y. OHAMA, "Polymers in Concrete" (CRC Press, Boca Raton, 1994) p. 83, 113.
17. G. MANTEGAZZA, A. M. PENNA and S. TATTONI, in "Fourth CANMET/ACI International Conference on Superplasticizers and Other Chemical Admixtures in Concrete," edited by V. M. Malhotra (ACI SP-148, American Concrete Institute, Detroit, 1994) p. 415.

Received 9 January
and accepted 28 October 2003