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MICROSTRUCTURE OF GLASS FIBRE REINFORCED WATER RESISTANT GYPSUM BINDER COMPOSITES

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ABSTRACT

The microstructure at the interface was studied in case of glass fibre reinforced gypsum binder composites exposed to water, accelerated ageing, i.e. alternate wetting and drying cycles at 27° to 60°C and to natural weathering. After 50 wetting and drying cycles at 60°C, deposition of gypsum binder products was observed in an irregular manner at the surface and in between glass fibre strands. Durability of E-type glass (borosilicate glass) fibres used as reinforcement in the water resistant gypsum binder composites has been assessed after exposure to aqueous extract of gypsum binder at temperature 27° to 60°C. The extracted glass fibres were characterized by scanning electron microscope and weight loss. Up to 40°C, no damage to glass fibres was noticed but at 50°C and 60°C, the glass fibres are attacked and erosion of glass surface takes place leading to the weakening of the interfacial bond at the matrix and fibre interface. A correlation between the durability of glass fibre and its weight loss was established. The loss in durability of glass fibre governs the flexural strength of gypsum binder composites tested under accelerated ageing.

Introduction

The gypsum plaster and plaster products are essentially used for interior decoration. These products are not suitable for outdoor construction owing to their solubility in water. A water resistant gypsum binder has been developed by blending ground granulated slag/fly ash, cement/hydrated lime and additives with 70-80 % calcined gypsum (β-hemihydrate/anhydrite) produced by calcining phosphogypsum. The hydraulic properties i.e. hydration and durability of the binder have been reported elsewhere (1-2). The glass fibre reinforced composites were produced using water

resistant gypsum binder and E-type (low-alkali borosilicate) glass fibres by spray-suction technique developed at the Central Building Research Institute, Roorkee. The strength and durability properties of glass fibre reinforced water resistant gypsum binder composites have shown that 4 per cent (by weight) 50 mm long glass fibres, the composite developed much higher strength and better water resistance than the plain plaster composites. It was noticed that gypsum binder had higher rate of strength development at 27 ± 2°C, 90 per cent RH than cured in water, air and natural weathering. When subjected to alternate wetting and drying cycles, the strength of gypsum binder was reduced and the weight loss was increased with the increase in temperature. Maximum fall in strength was noticed at 60°C of exposure.

In the present study, attempts were made to obtain microstructural information relevant to the glass reinforced gypsum binder composites under different environmental conditions. The results on durability of E-type glass fibre in the gypsum binder-water extract at 27°C to 60°C supplemented by determination of weight loss and microstructure of glass fibre were considered to establish a correlation between the fall in strength of gypsum composite with the durability of glass fibre. The results of findings are described and discussed in the paper.

Experimental

The water resistant gypsum binder (WRGB) was produced by blending the ground granulated slag (4200 cm^2/g Blaine's), cement (3200 cm^2/g Blaine's) and small quantity of retarder with the β-hemihydrate plaster produced by heating processed phosphogypsum.

E-type of glass fibre used in the preparation of gypsum binder composites was procured from the Fibre Glass (India) Ltd. The fibre rovings containing 204 filaments of 8-10 μm diameter were cut to 5 cm length and 4 per cent (by weight) of it was used to reinforce WRGB by spray suction technique to form composite specimens. The gypsum composites after 28 days of curing in over 90 per cent RH followed by drying were exposed to water, alternate wetting and drying cyles at 27°C and 60°C and natural weathering as per details given in reference (3).

To study the effect of temperature on E-type glass fibre, the uncoated glass filaments were reacted with the aqueous extracts of WRGB. The extract was prepared by shaking polythene bottle containing mixtures of gypsum binder and distilled water in the proportion of 1:5 (by volume) for a period of 24 hours. The reaction of glass fibre with aqueous extract of gypsum binder was carried out at 27°C, 40°C, 50°C and 60°C upto a period of 28 days.

After digestion and removal of the bulk of the extract, the fibres were treated with 2 N acetic acid, washed with water and dried at room temperature and stored in screw capped bottles prior to testing.

The fibres were removed from the composite by splitting it cen-

trally in the plane of the board to expose the fibre strands. These segments were placed in cold HCl solution (Conc. HCl : H_2O = 1 : 2) for 10 minutes, rinsed in distilled water for further 10 minutes and then dried on paper tissue for 20 minutes. The strands from the split faces after peeling away the matrix, were then mounted on polythene frames, rinsed in acetone and dried (4).

The microstructure of the fibre was studied with a scanning electron microscope (SEM) model 501 Philps (Holland). The weight of dried glass fibres was also noted to calculate the weight loss of the fibres.

Results and Discussion

Properties of Gypsum Binder

The physical properties of gypsum binder were setting time, initial 70 and final 145 minutes, soundness 1.60 mm, compressive strength and bulk density at 1, 3, 7 and 28 days : 10.1, 23.1, 28.6, 35.0 MPa; 1.54, 1.68, 1.85, 1.95 g/cc respectively. Hydration products of the binder monitored by Differential Thermal Analysis and X-ray diffraction were identified as ettringite and C-S-H. Microscopic examination revealed formation of well defined radiating needles of ettringite and dense calcium silicate hydrate. The clusters of needles are increased interspersed with lath and prismatic crystals with increase in curing period. The formation of ettringite and C-S-H are responsible for the increase in the strength of binder.

Performance of Gypsum Binder Composites

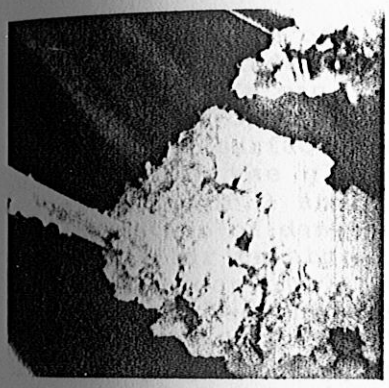
The gypsum binder composites (flexural strength 22 MPa) on immersion in water upto 28 days showed lower water absorption than the plain plaster composites. The gypsum binder and plain plaster composites retained 42.0 per cent and 7.0 per cent strength of original value respectively.

The gypsum binder composites when subjected to alternate wetting and drying cycles at 27°C to 60°C, the weight loss and fall in flexural strength of composites took place (3). It was noticed that maximum fall in strength occurred at 50°C. The fall in strength and increase in weight-loss of the composites was attributed to the decomposition of ettringite ($C_3A.3CaSO_4.32H_2O$) beyond 27°C. It is due to increase in the solubility of sulphate. The solubility of sulphate is increased with increase in temperature to 60°C, while the aluminium concentration does not differ markedly than obtained at 27°C. These results are well in agreement with the finding of Ghorab and Kishar (5). The gypsum composites exposed to natural weathering up to a period of three months showed fall in strength than the composites cured at 27°C and 90 per cent RH. Moreover the decrease in strength was much lower than the strength attained on subjecting the gypsum composites to 50 alternate wetting and drying cycles at 50°C.

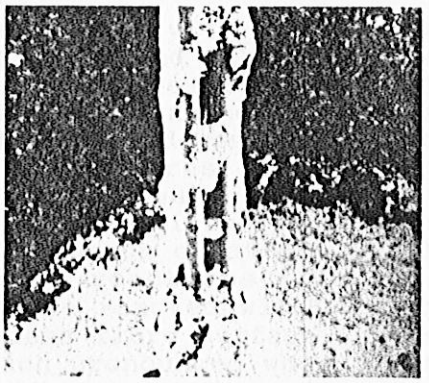
Microstructure at the Interface

Micrographs of the fibre and the fibre/matrix interface are shown in figure 1(a-d). These are typical of all the samples stored in water, exposed to wetting and drying cycles and natural weathering. Indeed it is significant that few differences between wet and dry stored 28 days old samples have been observed with gypsum binder composites.

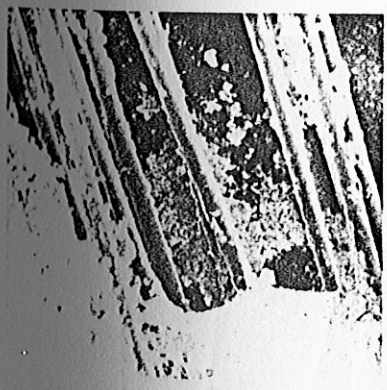
Figure 1(a) shows the filling of spaces between the filaments of a glass fibre strand with a dense crystallized gypsum binder products cured in water. A close examination of fibre shows that no visible deterioration of the glass fibre occurred.



(a) x 640



(b) x 320



(c) x 160

(d) x 320

Fig. 1

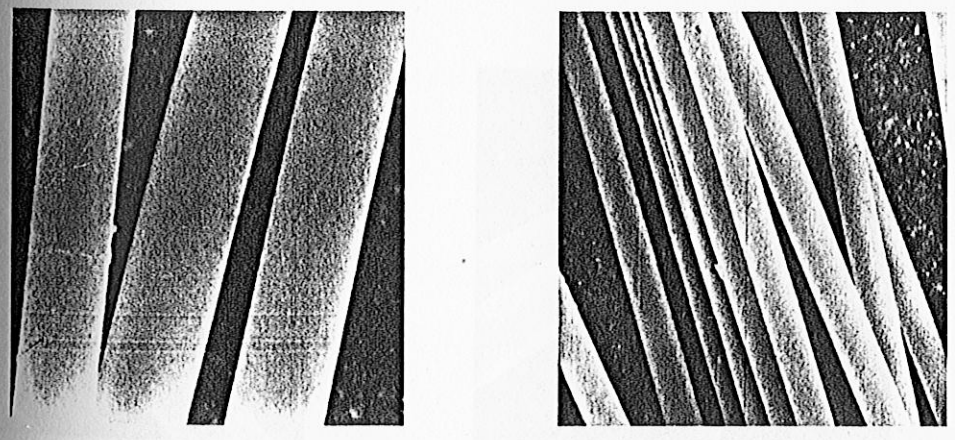
Micrographs of Glass Fibre/Matrix Interface of Gypsum Binder Composites : (a) Cured in Water (28 Days); (b) Exposed to 50 Alternate Wetting and Drying Cycles at 27°C; (c) Exposed to 50 Alternate Wetting and Drying Cycles at 60°C; (d) Exposed to Natural Weathering (28 Days).

Figures 1(b) and 1(c) show microstructure of glass fibre exposed to 50 alternate wetting and drying cycles at 27°C and 60°C. It can be seen from Figure 1(b) (27°C) the crystallized gypsum binder is tenaciously bonded to the glass fibre at the interface. While Figure 1(c) (60°C) clearly shows deposition of matter at the surface and in between glass fibre in an irregular manner showing cracking of the matrix leading to reduction in the mechanical properties due to weakening of the fibre/matrix bond.

Figure 1(d) shows microstructure of composites exposed to natural weathering at 28 days showing an intermittent point contact between glass fibre and the hardened gypsum binder phases in a haphazard manner resulting in a discontinuous and irregular interfacial bond.

Durability of Glass Fibre

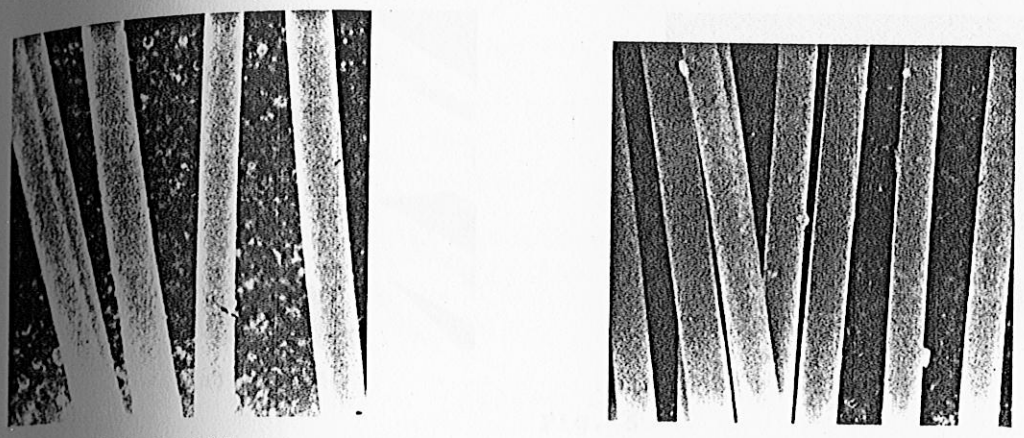
Microstructure of glass fibres reacted with the aqueous extract of gypsum binder at 27°C, 40°C, 50°C and 60°C are shown in Figures 2-5. It can be seen from the Figures 2(a) and 2(b) (3 and 7 days at 27°C) that euhedral fibres showing no pitting or deterioration of surface are observed, little deposition of material is formed on the glass surface when fibres reacted with binder extract for 3 and 7 days at 40°C [Figures 3(a) and 3(b)]. No indication of damage is visible. When glass fibres are subjected to similar treatment at 50°C, evidence of attack of alkalies is easily seen at 7 and 28 days of reactions [Figures 4(a) and 4(b)]. When the glass fibres are reacted with the aqueous extract at 60°C, the attack on glass fibre surface is visible at 7 days [Figure 5(a)] which is much pronounced when the fibres are exposed for 28 days [Figure 5(b)]. It is quite clear that a reaction film had formed on the surface of the fibre which subse-



(a) x 1250 (b) x 640

Fig. 2

Microstructure of Glass Fibres at 27°C : (a) 3 Days and (b) 7 Days.

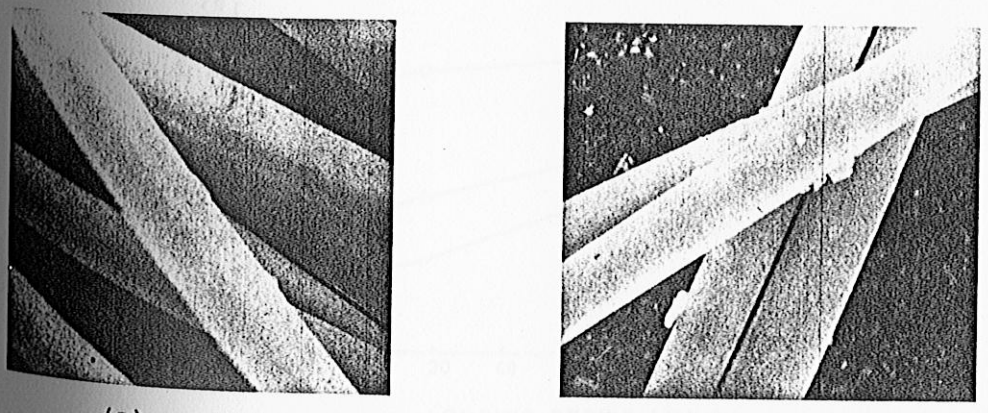


(a) x 640 (b) x 640

Fig. 3

Microstructure of Glass Fibres at 40°C : (a) 3 Days and (b) 7 Days.

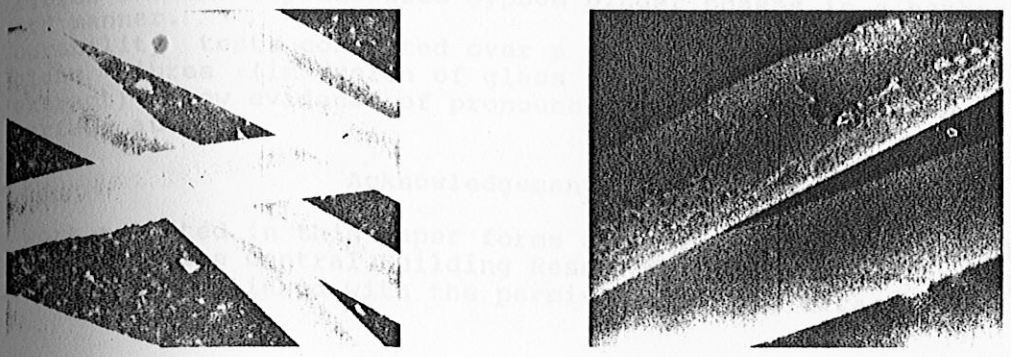
quently detatched itself from the bulk. As a consequence of surface degradation, glass fibres become weaker with time when placed in binder environment and the possibilities exist that the submicroscopic etchpits provide locations for nucleation and growth of crystalline phases in the interfacial zone is thus modified as a result of attack on the fibres. A degree of stability in the interfacial zone is important, however, the improved properties of glass reinforced gypsum binder composites are to be retained. One way of achieving this objective is by using the composite in temperature not exceeding 50°C.



(a) x 1250 (b) x 1250

Fig. 4

Microstructure of Glass Fibres at 50°C : (a) 7 Days and (b) 28 Days



(a) x 1250 (b) x 1250

Fig. 5

Microstructure of Glass Fibres at 60°C : (a) 7 Days and (b) 28 Days.

The weight loss of glass fibres reacted with binder extract upto a period of 3 months at 27°C to 60°C are plotted in Figure 6. It can be seen that with the increase in temperature, the weight loss of glass fibre is increased and maximum weight loss in fibre weight is visible at 60°C. The etching of the glass fibre and weight loss at 50°C & 60°C is directly responsible for the fall in the strength of gypsum composites when subjected to alternate wetting and drying cycles.

Conclusions

1. As compared to plain plaster composites, gypsum binder composites show better resistance when stored in water.

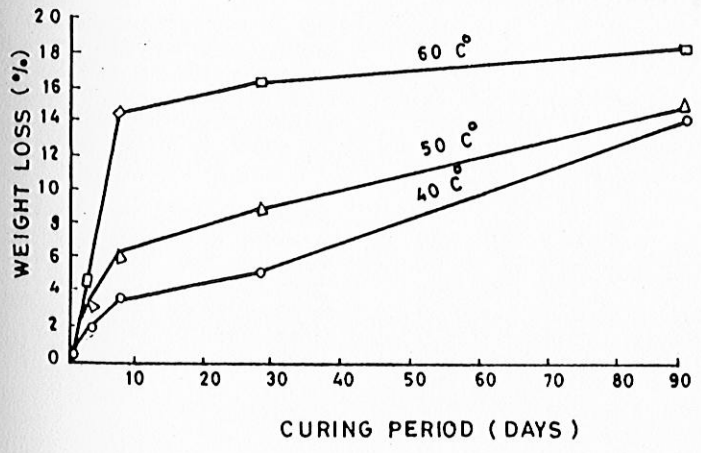


Fig. 6

Effect of immersion on the weight loss of glass fibres in aqueous gypsum binder extract at different temperatures

2. Microstructure of the hardened gypsum binder composites studied show an intermittent point contact between glass fibres and the crystallized gypsum binder phases in a haphazard manner.
3. Durability tests conducted over a period of 28 days on the glass fibres (immersion of glass fibres in aqueous binder extract) show evidence of pronounced deterioration of glass surface at 60°C.

Acknowledgement

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