THE EFFECT OF WOLLASTONITE MICRO-FIBRE ASPECT RATIO ON REINFORCEMENT OF PORTLAND CEMENT-BASED BINDERS

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ABSTRACT

Reinforcement of Portland cement-based binders with natural wollastonite micro-fibres has been further investigated with emphasis on the effect of fibre geometry on property modification. Wollastonite micro-fibres were separated into five different size groups before addition to cement and cement-silica fume matrices. The aspect ratio (length/width) of the wollastonite micro-fibres was determined by scanning electron microscopy technique. Systematic experimentation including flexural tests, mercury intrusion porosimetry, helium gas pycnometry, and isopropyl alcohol saturation measurement showed that, at constant micro-fibre content, the flexural strength and total porosity of the composites remained essentially unchanged and independent of the aspect ratio of the wollastonite micro-fibres. However, the volume of coarse pores, the pore size distribution, the flexural toughness and overall ductility characteristics of the hydrated cement and cement-silica fume matrices were observed to change systematically as the aspect ratio of wollastonite micro-fibres was increased. A discussion of the differences in the observed properties is presented.

Introduction

Previous investigations have demonstrated that natural wollastonite micro-fibres are effective in strengthening and toughening hydrated cement and cement-silica fume matrices. It was shown that wollastonite micro-fibres varying from 2% to 15% by volume can be readily mixed with the cement matrix and improve both flexural strength and overall ductility characteristics of the composite matrices [1]. The composite system can be further optimized by combining a maximum amount of wollastonite micro-fibres (about 11.5% by volume) and a suitable amount of silica fume (about 5.2%) in the base matrix [2]. It was also shown that flexural toughness and ductility of Portland cement based-matrices are systematically modified as the amount of wollastonite micro-fibres in the composite matrices increases [3]. Fibre aspect ratio and geometry of steel and carbon micro-fibres are known to have significant influence in the strengthening and toughening of cement-based composite mixtures [4-8]. There appears that little or no information is available in the literature with regard to the effect of fibre geometry and fibre aspect ratio of wollastonite micro-fibres on reinforcement of Portland cement-based composite systems. For this reason, a systematic study was carried out. The wollastonite micro-fibres available for industrial applications were first separated into five different size groups by a screening process. The aspect ratio (length/width) of the individual micro-fibres in each size group was determined by scanning electron microscopy technique. The separated wollastonite micro-fibres in each size group were then mixed with cement and cement-silica fume matrices. The property modifications in each series of composite systems were systematically evaluated. The results of the study are presented in this paper.
Experimental

Materials

Type 10 Portland cement* was used as the base component. Natural wollastonite micro-fibres which appeared in the shape of acicular particles were obtained from an American supplier**. The silica fume*** used in the study was similar to that used in the previous investigations. The superplasticizer solution**** is made up of about 48% solids and 52% water.

Micro-Fibre Separation

Natural wollastonite micro-fibres, calcium meta-silicate (β – CaO·SiO₂) mineral, available for industrial applications are generally in the form of loose acicular particles of mixed particle size. The transverse dimension (width) of the individual particle can vary from about 10 to 100 µm and the length can vary from about 0.05 mm to 2.0 mm. The wollastonite micro-fibres are therefore very non-uniform. In the present study the wollastonite acicular particles were manually separated by passing the particles through a combination of four different mechanical sieves in the following sequence:

![Sieve Sequence Diagram]

The collected fibres were categorized in the following five different size groups. Those fibres remaining on the No. 50 sieve were identified as >295 µm; those fibres passing through the No. 50 sieve and remaining on the No. 80 sieve were identified as >175 mm; those fibres passing through the No. 80 sieve and remaining on the No. 100 sieve were identified as >149 mm; those fibres passing through the No. 100 sieve and remaining on the No. 200 sieve were identified as >75 mm; those fibres passing through the No. 200 sieve were identified as <75 mm. The length and the transverse dimension of the individual wollastonite micro-fibres in each size group were accurately measured from photomicrographs obtained by a scanning electron microscope. #

Specimen Preparation

Two series of specimens were prepared with one series using plain cement as the base matrix and the other series using the cement-silica fume mixture (95 parts cement and 5 parts silica fume) as the base matrix. In each series of specimens, the amount of wollastonite micro-fibres was kept constant at 11.5% by volume. The appropriate amount of wollastonite micro-fibres selected from each size group was separately mixed with the base matrix.

All sample preparations were processed in a similar manner. When no silica fume was used, the wollastonite micro-fibres were gradually blended with the cement matrix until a uniform solid solution was obtained by visual inspection. When silica fume was used, the silica fume was first manually blended with cement in a steel mixing bowl until a uniform mixture was obtained by visual inspection. The wollastonite micro-fibres were then gradually blended with the cement-silica fume mixture until a uniform solid solution was obtained. The amount of superplasticizer solution equivalent to about 1% by weight of the solid mixture was added to the liquid water gauged to provide a water/solid ratio of 0.35. The liquid water containing the superplasticizer solution was first placed in the mechanical blender and mixed for about 1 minute. The dry mix solids were then added to the water solution and blended for a period of about 4 minutes. The blended cement composite mixture was cast into a plastic mould which measured about 40 x 130 x 4 mm. The cement mixture was allowed to harden in a moisture controlled room at saturation for a period of 24 hours. The hardened cement specimen was then de-moulded. After the de-moulding process, the specimen blocks were kept in a calcium hydroxide solution to continue the normal hydration treatment. The hydration treatment was carried out for a period of 28 days. After every 7 days of hydration, a composite block was removed from the calcium hydroxide solution and sliced into small beams measuring about 4 x 65 x 4 mm. These small beam specimens were used for flexural strength determination and microstructural characterization.

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* Type 10 Portland cement containing 19.8% SiO₂, 4.2% Al₂O₃, 3.2% Fe₂O₃, and 61.2% CaO was supplied by the St. Mary's Cement Co., Montreal, Quebec, Canada.
** The natural wollastonite micro-fibres were of the NYAD G grade and obtained from NYCO Minerals, Inc., Willsboro, N. Y., U.S.A.
*** The silica fume containing about 95.2% SiO₂, 1.6% carbon, 0.27% K₂O, and 0.10% Na₂O was obtained from the SKW Co., Montreal, Quebec, Canada.
**** The superplasticizer solution was the Atlas Mighty 150 RD2 grade.
# The scanning electron microscope is the Cambridge Steroscan Model S250 manufactured by Cambridge Instrument, England.
Test Measurements

The flexural strength of each composite beam specimen was determined by a three-point loading test method using a computer controlled Material Testing System\(^{##}\). Prior to the bending test, each beam specimen was maintained in the calcium hydroxide solution after the slicing operation. During test, the excess water on the surface of the specimen was first removed using a water absorbent type paper and the specimen was then installed on the specimen support. A total of 5 or 6 specimens was tested for each test condition and the average result was used.

Subsequent to the bending test, two representative fractured beam specimens from each series of composite mixture were selected for porosity measurement using the helium gas pycnometry method. After the pycnometry measurement, each beam specimen was broken into two portions of approximately equal size and weight. One portion was prepared for the mercury intrusion porosimetry measurement and one portion was used for the isopropyl alcohol saturation measurement. All specimens selected for porosity evaluations employing the three different methods were oven-dried at 105 °C for a period of at least 72 hours.

For the helium gas pycnometry measurement, the true volume of the whole beam specimen was accurately measured and repeated 5 or 6 times until a consistent value was obtained. For mercury intrusion measurement, the portion of each specimen was first broken into 7 to 8 small pieces and the small pieces were then inserted into the specimen container of the porosimeter. The mercury porosimeter\(^{###}\) was operated at pressures up to a maximum of 414 MPa (60,000 psi) and the helium gas pycnometer\(^{####}\) was operated at a gas pressure of about 0.21 MPa (30 psi). For isopropyl alcohol saturation measurement, the portion of the specimen was first weighed accurately to obtain a dry weight of the specimen. All specimens selected for evaluation were dried by a vacuum method at ambient temperature for a period of about three hours inside a closed desiccator. After the vacuum drying process, isopropyl alcohol was allowed to flow into the desiccator to saturate the specimens. The specimens were immersed in the isopropyl alcohol liquid for a period of three hours. The saturated weight of the specimen was obtained and the amount of isopropyl alcohol entered into the specimen was determined. The volume of the isopropyl alcohol entered into the specimen with respect to the geometric volume of the specimen was considered to be the porosity of the specimen.

Results and Discussion

Composite systems prepared with wollastonite micro-fibres having different aspect ratio were observed to show different properties. The results are separately discussed in terms of flexural strength, total porosity, pore size distribution, threshold pore diameter, flexural toughness and ductility characteristics.

[A] Aspect-Ratio of Wollastonite Micro-Fibres

Different length and width values of individual wollastonite micro-fibres in each size group are clearly visible in the SEM photomicrographs. Those fibres remaining on Sieve No. 50 (295 µm) identified as >295 are larger in both transverse dimension and length; fibres passing through Sieve No. 200 (74 µm) identified as <75 are considerably smaller in both transverse dimension and length. In order to be able to measure the transverse dimension and length of the latter group of fibres more accurately, the SEM photomicrograph was recorded at a higher magnification.

<table>
<thead>
<tr>
<th></th>
<th>A. Width</th>
<th>A. Length</th>
<th>Asp. Ratio</th>
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<tr>
<td>&gt;295</td>
<td>86.45</td>
<td>1668.75</td>
<td>19.49</td>
</tr>
<tr>
<td>&gt;175</td>
<td>86.67</td>
<td>947.91</td>
<td>11.01</td>
</tr>
<tr>
<td>&gt;149</td>
<td>45.00</td>
<td>505.41</td>
<td>10.65</td>
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<tr>
<td>&gt;75</td>
<td>27.47</td>
<td>168.98</td>
<td>3.78</td>
</tr>
<tr>
<td>&lt;75</td>
<td>9.33</td>
<td>44.17</td>
<td>4.49</td>
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The above table is the summarized values of the transverse dimension (width) and the length of each individual wollastonite micro-fibre in each size group as measured in (mm) from the magnification scale. The value is the average of about 10-12 representative individual fibres. The SEM photomicrographs are shown in Figure 1.

\(^{##}\) The Material Testing Systems is the MTS Model 810 apparatus manufactured by MTS Systems Corp., Mpls, MN, U.S.A.

\(^{###}\) The mercury porosimeter is the Model 60,000 psi Porosimeter manufactured by American Instrument Co., Maryland, U.S.A.

\(^{####}\) The helium gas pycnometer is the Beckman 930 Comparison Pycnometer manufactured by Beckman Instrument, Inc., California, U.S.A.
The flexural strength of type 10 Portland cement paste prepared with a water/cement ratio of 0.35 after 28 days of hydration in calcium hydroxide solution was about 12.3 MPa (1790 psi). When 11.5% by volume of wollastonite micro-fibres were added to the cement matrix, the flexural strength of the cement-wollastonite composite system was increased to the range of 21.1 - 22.6 MPa (3050 - 3280 psi). This strength level was observed in all specimens prepared with micro-fibres in five different size groups. There was no evidence showing a dependence of flexural strength on the aspect ratio of wollastonite micro-fibres. Composite mixtures prepared with wollastonite micro-fibres having the largest (19.49) and smaller (3.78-4.49) aspect ratio appeared to show similar flexural strength. The observed results indicate that the fibre geometry of wollastonite micro-fibres was not a determining factor on the strengthening process in the cement-wollastonite composite system. It is evident that the bond developed between the wollastonite micro-fibres and hydrated cement product is essentially similar for both large and small fibres. The intrinsic material properties of the wollastonite micro-fibres, such as modulus of elasticity and strength, rather than the fibre geometry, appear to be the most important factors in the strengthening process of these cement composites.
The flexural strength of the cement-silica fume composite mixture was about 9.8 MPa (1420 psi). The flexural strength was observed to increase to the range of 17.4 - 22.8 MPa (2540 - 3310 psi) when a similar amount of wollastonite micro-fibres for each size group was mixed with the cement-silica fume matrix. There was also no evidence indicating dependence of flexural strength on aspect ratio of wollastonite micro-fibres in this three-component composite system. The correlation between flexural strength and aspect ratio of wollastonite micro-fibres in the cement-wollastonite and the cement-silica fume-wollastonite composite systems is shown in Figure 2.

![Figure 2. Flexural strength versus aspect ratio of wollastonite micro-fibres in the cement-wollastonite (CW) and cement-silica fume-wollastonite (CSFW) composite systems.](image)

[C] Total Porosity

The total porosity in hydrated cement and cement-silica fume matrices reinforced with wollastonite micro-fibres having different aspect ratio was observed to show similar values within the data set for each method used in the porosity determination process. All specimens used for porosity measurements were hydrated for a period of 28 days and have similar flexural strength for each series of composite mixtures. As shown in previous investigations [1,2], the wollastonite micro-fibres are assumed to be non-porous and the porosity is concentrated only in the hydrated cement paste matrix. The results for each series of composite specimens are discussed in terms of the method of measurement.

(1) Mercury Intrusion Porosimetry

The total pore volume in the hydrated cement matrix was about 24.8% and increased to the range of 27.5% - 29.9% in the cement-wollastonite composite mixtures after addition of 11.5% by volume of wollastonite micro-fibres. These results are consistent with previous investigations [1,2]. The total pore volume in the cement-silica fume base matrix without the presence of wollastonite micro-fibres was about 21.7%. The total pore volume in the cement-silica fume-wollastonite system was observed to increase to the range of 24.9% - 27.8% with addition of similar amount of wollastonite micro-fibres. There was no systematic change of total pore volume with increase of aspect ratio in both composite systems. However, a small decrease of the total pore volume is noted in the cement-silica fume-wollastonite composite system when wollastonite micro-fibres having smaller aspect ratios (3.78-4.49) were used. This observation was not noted in the cement-wollastonite system. The variation of total pore volume versus aspect ratio (length/width) of wollastonite micro-fibres for the cement-wollastonite composite system and the cement-silica fume-wollastonite composite system is shown in Figures 3 and 4.

(2) Helium Gas Pycnometry

The total pore volume determined by the helium gas pycnometry was observed to be significantly lower than corresponding values determined by the mercury intrusion porosimetry. This observation was noted in both composite systems studied. Total pore volume values were in the range of 4.5% - 9.5% for the cement-wollastonite composite system and in the range of 2.8% - 7.1% for the cement-silica fume-wollastonite composite system. Variation of aspect ratio of wollastonite micro-fibres did not alter the total pore volume in the composite specimens determined by the helium gas infiltration process. The addition of a small amount of silica fume was observed to result in a slightly lower pore volume in the cement-silica fume-wollastonite composite system as observed in previous investigation [2]. Total pore volume determined by the helium gas pycnometry method versus aspect ratio of wollastonite micro-fibres is also plotted in Figures 3 and 4 respectively for the cement-wollastonite system and the cement-silica frame-wollastonite composite system.
The total pore volume determined by the isopropyl alcohol saturation measurement was observed to be in the range of 23.7% - 25.7% for the cement-wollastonite composite system and in the range of 18.9% - 21.6% for the cement-silica fume-wollastonite composite system. It is evident that these porosity values are significantly higher than those values determined by the helium gas pycnometry measurement and lower than those values determined by the mercury intrusion porosimetry method for both composite systems. There is also no evidence showing systematic variation of total pore volume in the composite mixtures with change of aspect ratio of the wollastonite micro-fibres. It appears that variation in the fibre geometry of the wollastonite micro-fibres did not prevent liquid infiltration into most of pore space in both composite systems.

The total pore volume determined by the isopropyl alcohol saturation method was observed to be slightly lower when compared with that determined by the mercury intrusion porosimetry method for both cement-wollastonite and cement-silica fume-wollastonite composite systems. In a previous investigation, the total porosity in the composite specimens determined by the isopropyl alcohol saturation measurement was observed to be slightly higher than the results obtained by the mercury intrusion measurement [2]. This difference could have been attributed to different sources of wollastonite micro-fibres used in the early and the present investigations. It is possible that the surface wetting process and the extent of interaction between the surface of the wollastonite micro-fibres and the hydrated calcium silicate in the composite matrices in the two investigations were slightly different [8-11].

[D] Pore Size Distribution

It was indicated from the analysis of the pore size distribution curves for the cement-wollastonite composite system that the pore volume with coarse pores in the range of 1.00 - 0.10 µm was increased by the addition of wollastonite micro-fibres in the cement paste. The magnitude of the increase became larger as the aspect ratio of wollastonite micro-fibres was increased from 3.89 to 19.49. Pore volume in the smaller size range, 0.01 - 0.1 µm, was observed to be very similar and unaffected by the change of aspect ratio of the wollastonite micro-fibres. However, the pore volume in the very small pore region (< 0.01 µm) was observed to become larger when composite mixtures were prepared with wollastonite micro-fibres having larger aspect ratio, as shown in Figure 5.
The pore size distribution curves for the cement-silica fume-wollastonite composite system varied in a slightly different manner when compared with that observed in the cement-wollastonite composite system. The volume of coarse pores in the range of 1.00 - 0.10 µm was observed to be relatively small in the base cement-silica fume matrix. The volume of coarse pores in the same size range was observed to increase significantly when 11.5% by volume of wollastonite micro-fibres were added to the cement-silica fume matrix. The amount was dependent on the aspect ratio of the wollastonite micro-fibres in the composite mixture. The change of pore volume at 0.10 µm was particularly pronounced. The change of pore volume in the region of 0.10-0.01 µm occurred in two stages, a rapid rise and then leveling off. The pore size distribution curves for the cement-silica fume-wollastonite composite system prepared with wollastonite micro-fibres having different aspect ratio are shown in Figure 6.

The observation of a large volume of coarse pores in the region of 0.5-0.1 µm resulting from the addition of wollastonite micro-fibres in the cement and cement-silica fume matrices in the present study was found to be consistent with previous results [2]. This trend was also found to be very similar to that obtained in a study of cement mortars [12]. In the study of cement mortars, it was suggested that large pores are formed at the interface between the sand particle and the cement paste. These generally increased with cement-sand ratio in mixes with and without the presence of silica fume. Addition of silica fume to mortars was also observed to result in an increase of large pores in this range at the sand-paste interface. It therefore had the general effect of increasing the volume of coarse pores.

The change of pore size distribution observed in the cement-wollastonite and cement-silica fume-wollastonite composite systems was similar to that for the cement-sand system. However, the volume of coarse pores in the region of 0.5-0.1 µm in the cement-silica fume-wollastonite composite system was observed to be more pronounced than in the cement-wollastonite composite system. In the present study, when long wollastonite micro-fibres with an aspect ratio of 19.49 were used to reinforce the cement-silica fume base matrix, the volume of coarse pores in this region was found to be almost double that obtained with short wollastonite micro-fibres with an aspect ratio of 4.49. Since the amount of wollastonite micro-fibres in each specimen remained constant at 11.5% by volume, the change of aspect ratio of wollastonite micro-fibres would affect the total number of individual micro-fibres present in the matrix. Wollastonite micro-fibres with larger aspect ratio would have smaller number of individual micro-fibres present in the base matrix. It is suggested that more coarse pores are developed along the interface between the surface of long acicular wollastonite micro-fibres and the hydrated cement paste.
[E] Threshold Pore Diameter

The threshold pore diameter is the specific pore diameter at which the rapid increase of pore volume during the mercury intrusion process starts. The threshold pore diameter in the plain cement paste was about 0.116 µm. The threshold pore diameter in the cement-wollastonite composite system containing 11.5% by volume of small wollastonite micro-fibres with an aspect ratio of 4.49 was also about 0.116 µm. However, as the aspect ratio of wollastonite micro-fibres increased the threshold pore diameter in the cement-wollastonite composite system also gradually increased. The threshold pore diameter in the cement-wollastonite composite system containing 11.5% by volume of the largest wollastonite micro-fibres with an aspect ratio of about 19.49 was increased to 0.175 µm. The threshold pore diameter in the cement-silica fume base matrix was about 0.07 µm. The threshold pore diameter in the cement-silica fume-wollastonite composite system containing 11.5% by volume was also observed to increase when the aspect ratio of the wollastonite micro-fibres was changed. The threshold pore diameter in the cement-wollastonite composite system containing 11.5% by volume of the smallest wollastonite micro-fibres with an aspect ratio of 4.49 was also about 0.07 µm but it gradually increased to 0.116 µm when large wollastonite micro-fibres with an aspect ratio of about 19.49 were used. These results indicate that the fibre geometry of the wollastonite micro-fibres in the composite matrices has an effect on the mercury intrusion process.

[F] Toughness and Ductility Characteristics

Load-deflection curves obtained from flexural tests provide information about the flexural toughness and ductility characteristics in hydrated cement paste reinforced with wollastonite micro-fibres [1]. A previous investigation has shown that flexural toughness and ductility of Portland cement based-binders change as the amount of wollastonite micro-fibres added to the cement and cement-silica fume base matrices increases from 2% to 15% by volume. In the present investigation, the flexural toughness and ductility of the Portland cement-based binders reinforced with wollastonite micro-fibres were also observed to change systematically when aspect ratio of the wollastonite fibres in the composite matrices was changed. When short wollastonite micro-fibres with an aspect ratio of 4.49 were used for reinforcement, fracture and total failure in the hydrated composite matrix occurred abruptly with a straight drop in the load-deflection plot. There appears to be no additional contribution to toughness from post cracking events by the addition of the short wollastonite fibres. This observation is not unexpected because the size of the wollastonite micro-fibres and the size of the cement grains are in the same range. When wollastonite micro-fibres identified as >175 (aspect ratio = 11.01) were used for reinforcement, fracture and total failure in the hydrated composite matrix occurred very gradually and the descending branch covered an extensive region in the load-deflection plot The improvement of the ductility in the composite matrices resulting from reinforcement with medium size wollastonite micro-fibres identified as >175 was more pronounced than that with larger micro-fibres identified as >295. This is attributed to greater uniformity of the medium size micro-fibres, as shown in the SEM photomicrographs in Figure 1. The load-deflection curves for the cement-wollastonite composite system and the cement-silica fume-wollastonite composite system are shown in Figures 7 and 8 respectively.

A correlation between the post-peak deflection and the aspect ratio of wollastonite micro-fibres was established and is shown in Figure 9. It is evident that cement binders reinforced with wollastonite micro-fibres having larger aspect ratio have a much larger post-peak deflection indicating that the ductility of composite matrices is much larger than similar binders prepared with smaller fibres and smaller aspect ratio.

![Figure 7. Load-deflection curves of the cement-wollastonite composite mixtures prepared with wollastonite micro-fibres having different aspect ratio (length/width).](image-url)
A measure of flexural toughness can be determined by calculating the area under the load-deflection curve as shown in a previous study [3]. A correlation between the relative toughness (toughness of composite/toughness of base matrix) and the aspect ratio of wollastonite micro-fibres was also established and is shown in Figure 10. It is also evident that cement binders reinforced with more uniform medium size wollastonite micro-fibres are tougher than similar binders prepared with either smaller fibre size or fibres with larger aspect ratio.

Conclusions

The experimental results obtained in this investigation appear to support the following conclusions.

1. The flexural strength of cement and cement-silica fume matrices reinforced with 11.5% by volume of wollastonite micro-fibres is independent of the aspect ratio of wollastonite micro-fibres.

2. The pore structure in the hydrated cement-wollastonite and cement-silica fume-wollastonite composite systems varies slightly by the addition of wollastonite micro-fibres having different aspect ratio.

3. The overall total porosity in the hydrated cement-wollastonite and cement-silica fume-wollastonite composite systems as determined by mercury intrusion, helium gas pycnometry and isopropyl alcohol saturation methods is also independent of the aspect ratio of wollastonite micro-fibres.

4. The pore volume versus pore size curves for both cement-wollastonite and cement-silica fume-wollastonite composite systems have slightly different characteristics when the aspect ratio of wollastonite micro-fibres is increased. The volume of coarse pores in the range of 0.5 - 0.1 μm and the volume of very small pores in the region 0.01 - 0.005 μm increase slightly as the aspect ratio of wollastonite micro-fibres in the composite matrices increases.
5. Wollastonite micro-fibres having relatively large aspect ratio added to cement and cement-silica fume matrices appear to promote the formation of more large pores in both cement-wollastonite and cement-silica fume-wollastonite composites.

6. Portland cement based-binders reinforced with wollastonite micro-fibres having larger aspect ratio are tougher and more ductile than similar binders reinforced with similar fibres having lower size aspect ratio.

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