MICROSILICA IN CONCRETE

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SYNOPSIS
Microsilica is a by-product from the ferro alloys industry. It consists of extremely fine, amorphous silica particles. Microsilica affects the properties of fresh concrete, the pore size distribution and the hydration products formed during hydration, and hereby the mechanical properties and durability of the hardened cement based products. The literature on the properties of microsilica in concrete is reviewed.

Key-words: Microsilica, concrete properties, microstructure, durability, review.

1. INTRODUCTION

Microsilica is a by-product from the ferro alloys industry. It consists of extremely fine, amorphous silica particles. Microsilica has been given a variety of names such as silica dust, silica fume, condensed silica powder.

Until recently microsilica has been considered a waste material, and large quantities have been dumped. Nowadays it is realized that microsilica possesses unique properties and it is considered a valuable material in the fertilizer industry, refractory industry, plastics industry, and in the cement and concrete industry.

Microsilica affects such vital properties of cement based products as the rheological behaviour of fresh concrete, the pore size distribution and the hydration products formed during hydration and hereby the mechanical properties and durability of the hardened cement based products.

The reason for these interesting properties may be attributed to a microfiller effect and a pozzolanic effect of microsilica in cement based products.

> Microfiller effect. The silica particles are a 100 times smaller than the cement grains, and are, therefore, easily introduced into the spaces between the cement grains reducing the space available for water and acting as nucleation sites for hydration products.
> The microsilica particles densify the cement paste by substituting water.
>
> Pozzolanic effect. As the particles are amorphous silica with an extremely high specific surface, they react chemically with ions in the surrounding alkaline pore solution forming reaction products which resemble the hydration products found in hardened cement paste.

2. MICROSILICA PRODUCTION AND CHARACTERISTIC PROPERTIES

2.1 Production of ferro-silicon alloys and microsilica

Microsilica emerges as a by-product during the production of silicon metal and ferro-silicon alloys in electric arc furnaces (see Figure 1).
Pure silicon metal is produced by reducing quartz by coal at high temperatures (2000°C). Iron is added to the raw mix when ferro-silicon is produced. The molten silicon or ferro-silicon is tapped through the bottom of the furnace.

The reactants are heated by passing electric current through the charge in the furnace. The electricity demand is high: approximately 13.000 kWh are needed per ton ferro-silicon alloy produced.

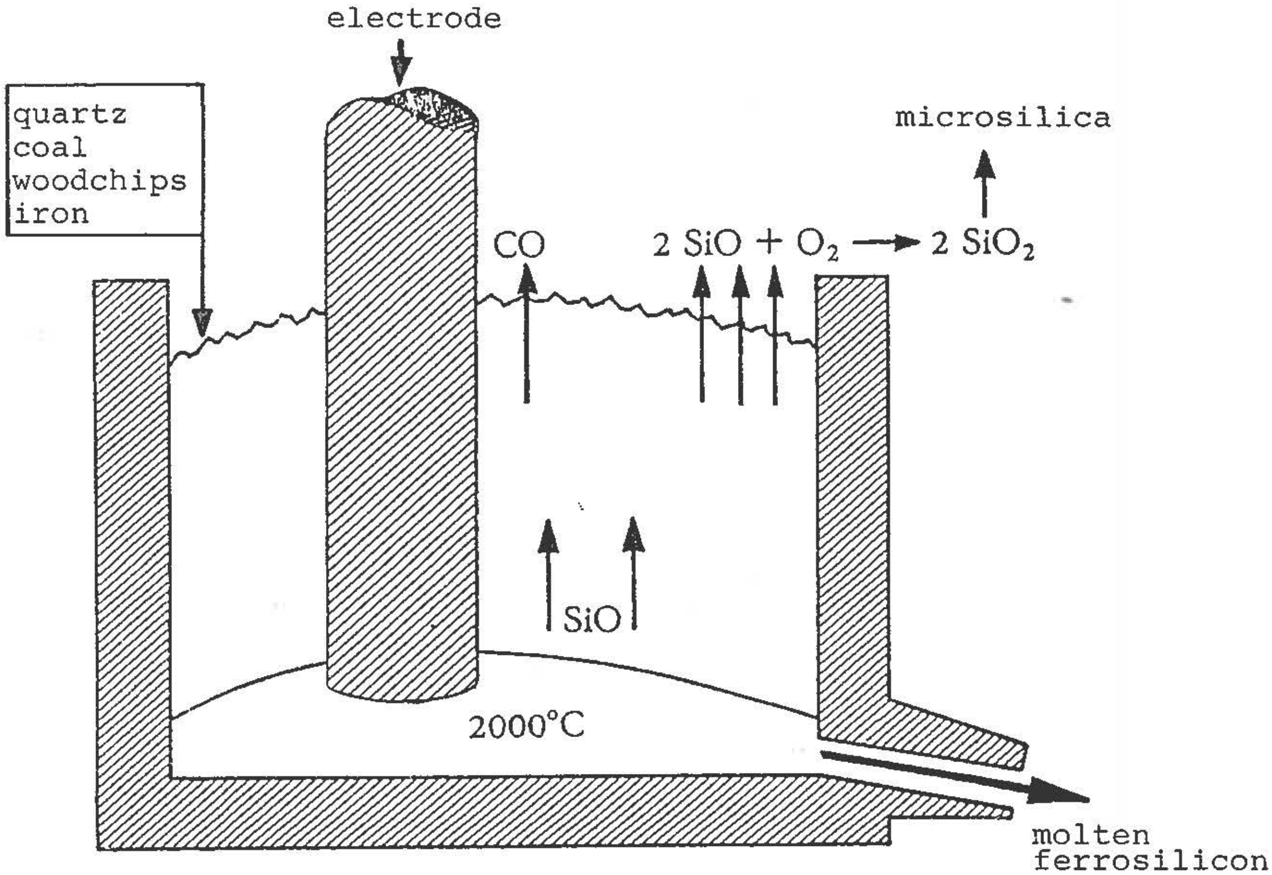

FIG. 1. Sketch of microsilica formation in an electric arc furnace

An idealized overall reaction in the furnace /1/ /2/ may be written

$$SiO_2 + 2C + xFe \rightarrow Fe_xSi + 2CO.$$

The reduction of quartz to silicon is not complete, however, and it is not direct. In a particular zone of the furnace, SiO gas is produced. Some of this gaseous SiO reaches the top of the furnace together with other combustion gases, and here it is oxydized to SiO_2 by the air and condenses as very small particles: microsilica. The silica fume is then collected in large baghouse filter installations.

Microsilica has a very low bulk density and the particles are very fine. It is, therefore, difficult to handle and transport. Consequently, it is often used in a compacted form /3/, as small nodules made by an agglomeration process /4/, or as a water slurry containing for example 50% microsilica.

The amount of microsilica obtained varies, but it is typically 400-600 kg microsilica for each ton of ferro-silicon alloy. The annual worldproduction of microsilica is 1.2 million tons, one third of which is produced in USA, Norway and Japan.

2.2 Characteristic properties of microsilica

Microsilica particles are spherical in shape and have an average diameter of 0.1 μm. The particles may be found in loosely bound agglomerates and in strong, primary agglomerates up to 0.5 μm /5/.

Its specific surface is very high. The specific surface, as measured by the BET-method, may vary from 15 m^2/g to 25 m^2/g. A typical value is 20 m^2/g.
Its specific weight is about 2.2 g/cm^3 and the bulk density varies from 0.2 g/cm^3 for fume right from the filter to 0.6 g/cm^3 for compacted microsilica.

	Si-metal	75% FeSi
SiO_2 %	94 - 98	86 - 90
C %	0.2 - 1.3	0.8 - 2.3
Fe_2O_3 %	0.02 - 0.15	0.3 - 1.0
Al_2O_3 %	0.1 - 0.4	0.2 - 0.6
CaO %	0.08 - 0.3	0.2 - 0.6
MgO %	0.3 - 0.9	1.0 - 3.5
Na_2O %	0.1 - 0.4	0.8 - 1.8
K_2O %	0.2 - 0.7	1.5 - 3.5
S %	0.1 - 0.3	0.2 - 0.4
LOI %	0.8 - 1.5	2.0 - 4.0

FIG. 2. Chemical analyses of microsilica from the production of silicon metal and 75% ferro-silicon alloy.

Microsilica has a gray colour which may vary with the operating conditions of the furnace.
The particles are substantially amorphous, but microsilica may contain minor quantities of crystalline materials, such as silicon carbide, pure silicon, various ferro-silicon alloys and crystalline silica.

The chemical composition of microsilica varies with the type of alloy produced /47/.
Chemical analyses of microsilica from the production of pure silicon metals and 75% ferro-silicon alloy are shown in Figure 2.
It appears that microsilica is almost pure silica with minor quantities of alumina, iron, lime and alkalies.
The carbon is present as tar and as ultra-fine particles of graphitic carbon.

3. THE PROPERTIES OF FRESH CONCRETE

The properties of fresh concrete are strongly affected by the addition of microsilica.
The workability, the cohesiveness and the tendency to segregation and bleeding are changed considerably.

The addition of small amounts of microsilica (5%) has a minor influence on the water demand of an ordinary concrete mix.
At higher dosages of microsilica, on the other hand, the water required to maintain a certain slump of the concrete seems to increase linearly with the amount of microsilica /6/ /7/.
However, this increased water demand can be easily eliminated by using a suitable water reducing agent as illustrated in Figure 3 /8/ /9/.

The water reducing agent is seen to have an enhanced effect on microsilica containing concrete.

Figure 3 illustrates that a favourable use of microsilica is closely bound up with the use of suitable water reducing agents. It is of vital importance to the beneficial effect of microsilica addition that the microsilica is well dispersed in the cement paste.

High dosages of water reducing agents result in a decrease of the water demand. This effect has been used to develop a new type of cement based materials, which are extremely dense and strong.
These new materials are termed DSP, referring to "Densified Systems containing homogeneously arranged, ultra-fine Particles" /10/.

The effect of microsilica on the rheology of fresh concrete may be summarized as a "stabilizing effect".
This includes the observations that the tendency to separation and bleeding is reduced and the viscosity and internal coherence are increased.
These changes in properties have proven to be useful in practice in relation to under-water casting and pumping of concrete, and for shotcrete.

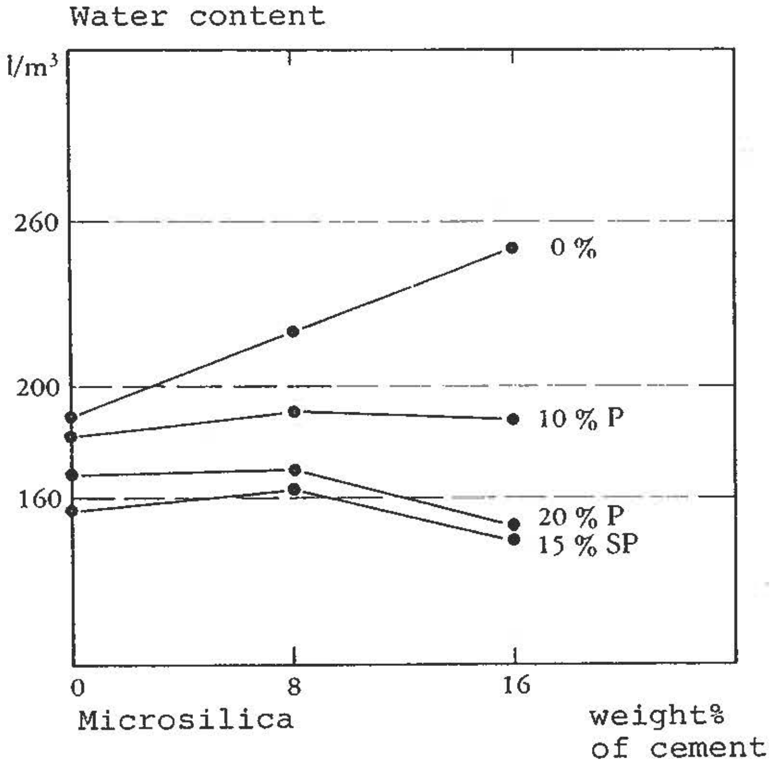

FIG. 3. Water content of concrete to maintain 12 cm slump as a function of microsilica addition and use of ordinary plasticizer (P) and superplasticizer (SP) /8/. The admixture dosages are percentages of the microsilica.

The observed "stabilizing effect" may be explained by following mechanisms:

- The presence of ultra-fine particles reduces the rate of internal liquid flow in the paste.
- The internal coherence increases as the number of contact points increases.
- Cement grains and microsilica particles are bound together by reaction products.

4. HYDRATION AND HARDENING

4.1 Hydration mechanisms and reaction products

Microsilica must be considered an active reactant during the hydration of microsilica containing cement pastes.
The action is chemical as well as physical.

Microsilica reacts chemically with the ions in the pore solution. Chemical analyses of pore solutions expressed from microsilica containing cement pastes /11/ /12/ show that microsilica addition causes profound changes in the chemistry of the pore solution.

After 10 days of hydration of cement pastes with 5% microsilica and w/c = 0.4 the concentrations of K^+, Na^+, and OH^- are approximately half of those obtained in neat cement pastes by 2½ months /11/.

Higher replacement levels result in greater rates of reduction of alkali and OH^- ion concentration with time and lower levels for the equilibrium pH. A pH of 12.2 was found after 5 months hydration in cement pastes having 30% microsilica and a water to powder ratio of 0.4. A pH of 11.9 was found after 84 days hydration of cement pastes with 30% microsilica and a water to powder ratio of 5 /12/.

Microsilica's pozzolanic properties have been proven by the detection of well crystallized CSH I after 7 days reaction of microsilica in calcium hydroxide solution at 38°C /13/.
The pozzolanic properties of microsilica in cement paste have been demonstrated by measuring the calcium hydroxide content of cement pastes containing various amounts of microsilica /14/. The result is shown in Figure 4. The calcium hydroxide content was measured by thermogravimetric analysis.

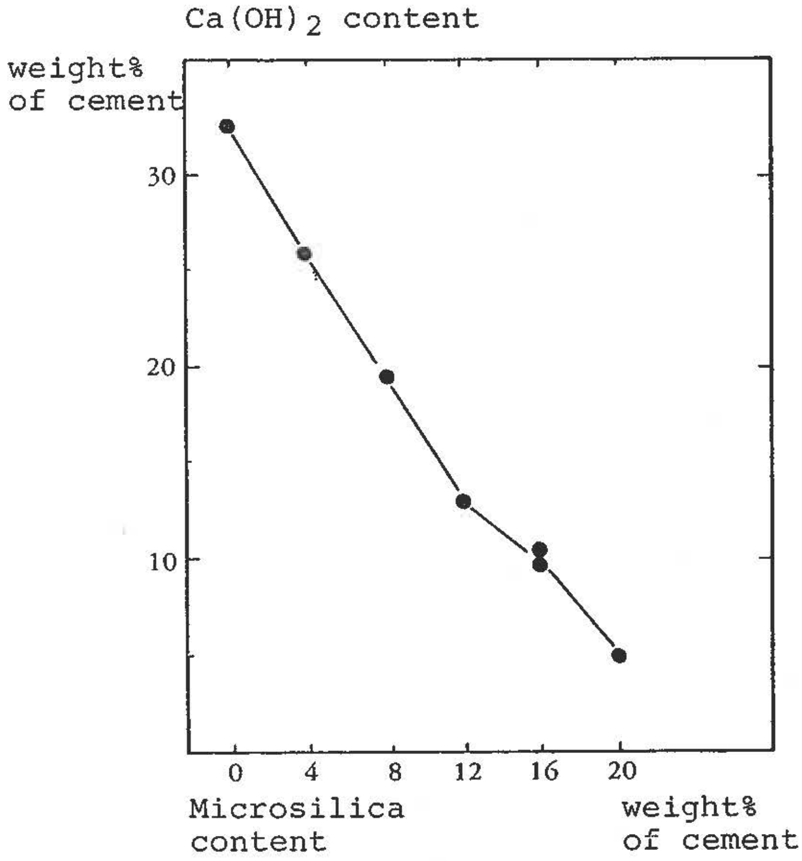

FIG. 4. Calcium hydroxide content in samples of cement paste + microsilica /14/.

The pozzolanic activity of microsilica in cement pastes has been reported to be very small /15/, medium /16/ /17/ and strong /14/ /18/ /19/.

The hydration products formed in microsilica containing cement pastes resemble those found in ordinary cement pastes although certain deviations are found.
The presence of CSH I products has been reported /17/ and C/S values as low as 0.9-1.3 have been found by different techniques /19/ /16/ /14/ /18/.
The H/C value is probably also lower as it has been found that the amount of non-evaporable water in hydrating microsilicacement pastes remains constant, although the amount of calcium hydroxide continues to decrease and the microsilica particles continue to react /11/ /14/. These observations indicate a H/C of 1.

The physical effect of microsilica is reported as an acceleration of the hydration of cement /14/. This accelerating effect is assumed to be caused by the small particles acting as nuclei for hydration products.

This has been demonstrated /14/ by adding microsilica and fine calcium carbonate respectively to cement pastes and measuring the content of non-evaporable water.
The investigation shows that increasing amounts of microsilica result in increasing amounts of non-evaporable water at early ages.
Equal amounts of calcium carbonate and microsilica result in the same increase in the amount of non-evaporable water at early ages. Although fine calcium carbonate may not be completely inert the increasing content of non-evaporable water is interpreted as an accelerated cement hydration. This is in agreement with other observations of the reaction of pozzolanas /20/.

Recently a model for the reaction mechanisms of microsilica in cement paste has been proposed /21/. According to this model the microsilica particles lose their anhydrous nature by hydrating to a silica-rich "gel" in the alkaline pore solution shortly after mixing with water. The "gel-particles" may agglomerate to larger masses of "gel".
The "gel" then combines, at the "gel" surfaces, with $Ca(OH)_2$ to form additional CSH.

The high reactivity of microsilica towards alkali ions and hydroxide ions as described previously, and the fact that the pore solution of ordinary cement pastes few minutes after mixing has a high concentration of OH^-, K^+, Na^+ and Ca^{++} /22/ point towards a very early start of reaction of microsilica.

It is proposed, therefore, to consider microsilica as an active reactant of the cement paste like the various cement minerals and gypsum. It influences the hydration kinetics and the type of hydration products formed from the moment water is added.

4.2 Heat of hydration and high temperature curing

At present the effect of microsilica on the heat of hydration of concrete and the effect of elevated temperature curing on microsilica containing concrete have not been fully elaborated /23/ /24/.

Results from preliminary experiments /25/ indicate, however, that

- The chemical reactions of microsilica in cement pastes are exothermic.
- the specific heat evolution of microsilica (kJ/kg) in cement paste is of the same order of magnitude (or a little higher) as the specific heat evolution of cement.
- the normally used Arrhenius transformation of temperature time, which is valid for an estimation of the maturity of ordinary concrete at 20-60°C and 10-70% of the ultimate strength, may also be applied to microsilica containing concrete.
- prolonged heat curing is favourable to microsilica containing concrete.

As will be shown in a proceeding chapter the specific strength gain of microsilica (MPa/kg) in concrete is larger than the specific strength gain for cement. It is therefore possible to design a low-heat concrete fulfilling a specified strength requirement by substituting cement by microsilica /26/, and it is possible to design a concrete exhibiting high early strengths by adding microsilica and applying high temperature curing.

4.3 Plastic shrinkage

Microsilica containing concretes are sensitive to develop plastic shrinkage cracks if the surface is exposed to drying at early age /27/ /28/.
The crack formation is caused by internal stresses introduced by formation of the water-air meniscuses in the pore system at the concrete surface when there is no bleeding water and the rate of water evaporation is greater than the rate of water transport to the surface.
As the pores are smaller and the rate of internal liquid flow is reduced in microsilica containing concrete, such concrete is exposed more to plastic shrinkage.
In order to avoid such problems the concrete surface must be protected against evaporation as soon as possible, for example by applying curing compounds.

Chemical shrinkage or self-desiccation may create local, internal stresses during hydration. Chemical shrinkage related to the chemical reactions of microsilica is estimated to be half the size of the chemical shrinkage taking place during cement hydration /14/.
Other measurements on the bulk shrinkage of microsilica containing cement pastes /29/ indicate the shrinkage to be at the same level, or even a little higher, than the shrinkage of pure cement paste.

Long term drying shrinkage is not affected significantly by the addition of microsilica /6/ /9/, provided that the water content is unchanged. When the microsilica containing concrete is exposed to drying at early ages, however, such concretes have a considerably higher drying shrinkage than ordinary concrete /30/.

5. MECHANICAL PROPERTIES OF HARDENED CONCRETE

Since the early fifties microsilica has been known to possess good strength properties /31/ /32/ /33/.
Many investigations have shown that the addition of small amounts of microsilica results in increasing compressive strengths, whereas addition of a larger amount (more than 5%) very often results in decreasing strengths due to an increased water demand /31/ /34/ /6/.
As previously mentioned, however, the application of suitable water reducing agents may enable the production of concrete at a fixed slump, with increased microsilica content without increased water content.
Such changes densify the cement paste and result in increasing strengths with increasing microsilica content.

In fact the very dense DSP materials, described above, have compressive strength values of 110-160 MPa (cast cylinders 10 x 20 cm) when ordinary aggregates are used, and 200-270 MPa when high strength aggregates are used /10/.

It has been found that the relationship between compressive strength and w/c-ratio that is normally used for ordinary concrete, is also valid for microsilica containing concrete, provided that the amount of microsilica is multiplied by a factor, which transforms the amount of silica to an equivalent amount of cement.

In order to quantify the effect of microsilica on the compressive strength of concrete the "cement equivalent factor" (k) is often used. Thus k is equal to the mass of cement necessary to add to a plain concrete to produce same strength increase as one unit mass of microsilica.
The cement equivalent factor is around 3 for the compressive strength at 28 days of concrete containing 300 kg/m^3 of cement and cured at 100% relative humidity and 20°C, which means that 3 parts of cement may be replaced by 1 part of microsilica.
Various examinations of the cement equivalent factor indicate that the factor increases with age and decreases with the cement content, the w/c-ratio and the microsilica content of the concrete and, moreover it decreases when curing conditions are changed towards shorter periods at 100% RH /9/ /35/ /34/.

At ordinary strength levels and low microsilica contents the ratio between tensile strength and compressive strength, the ratio between compressive E-modulus and compressive strength, the ratio between ductility and tensile strength and the Poissons ratio are similar to those for ordinary concrete /9/ /6/.
At higher strength levels, however, the material becomes more brittle, which means that increased reinforcement is required.

A comprehensive study of the mechanical properties of microsilica containing concrete has recently been published /36/.

6. PORE SIZE DISTRIBUTION AND DURABILITY

6.1 Permeability and pore size distribution

Several investigations have shown that the permeability of microsilica containing concrete is remarkably low, also in concrete with low cement content in which the effect of microsilica is the largest /9/ /33/.
Such reduced permeabilities are also found in products in which the total porosity is not reduced.

This effect is suggested to be a result of the very fine particles subdividing the water filled space between the cement particles found in ordinary cement paste /42/. During hydration of the cement grains and reaction of the microsilica particles the reaction products precipitate at the microsilica particles which act as crystallization nuclei.
The overall result from very early ages to later ages may be characterized as a "refinement" of the pore system (see Figure 5).

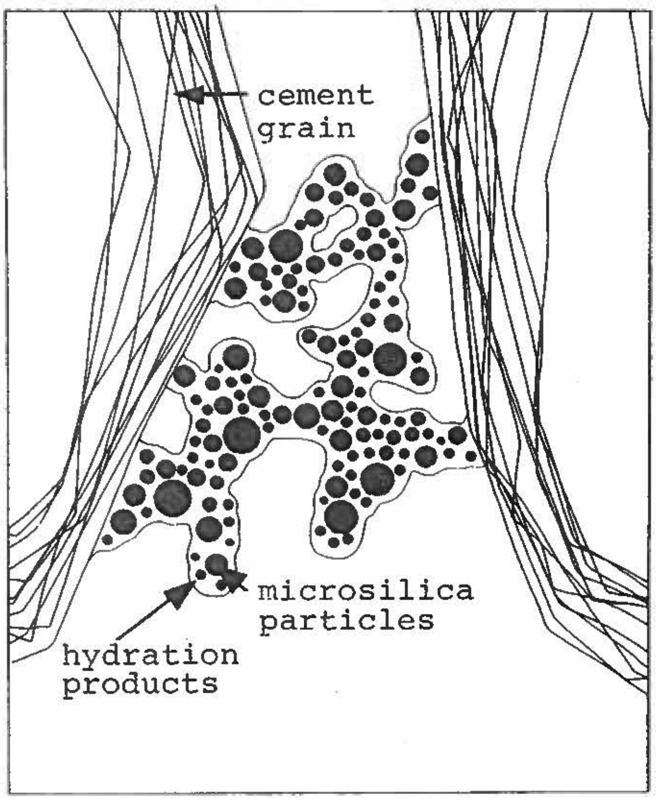

FIG. 5. Sketch illustrating the microsilica particles subdividing the space between the cement particles /42/.

The refinement is very clearly shown in a detailed study of the microstructure of microsilica containing cement pastes /14/. It was concluded that the total volume porosity of the pastes is practically independent of the amount of microsilica, whereas the gel porosity increases linearly with the microsilica content, and the pore size distribution of the capillary pores becomes finer with increasing microsilica content (see Figure 6).

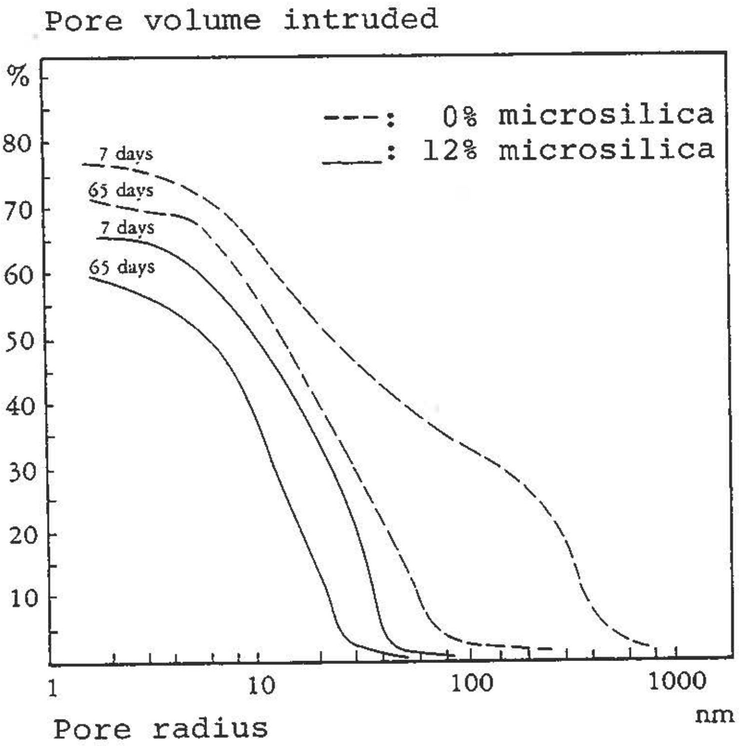

FIG. 6. Pore size distribution of cement pastes with and without microsilica measured by mercury intrusion porosimetry /14/.

The consequences of the refinement of the pore structure are clearly reflected in results from investigations on the durability of microsilica containing concrete.
As liquid flow and the rate of ion diffusion are considerably reduced in such materials, deterioration of concrete by mechanisms which involve mass transfer is correspondingly decreased.

6.2 Chemical attack

Deterioration due to chemical attack on concrete is considerably decreased when microsilica is incorporated.
20 years field exposure of concrete to ground water of low pH and high sulphate content has proved that concrete made from ordinary Portland cement and 15% microsilica has similar resistance as concrete made from sulphate-resisting cement /37/.
Various laboratory examinations have also proved the increased sulphate resistance /38/ /31/ of microsilica containing cement pastes.

The main reason for the increased durability is probably the reduced transfer rate of harmful ions.
An additional factor is related to the ability of the CSH products of low C/S-ratio to incorporate higher amounts of foreign ions in the lattice /19/. An increased amount of Al incorporated in the CSH products reduces the available amount of alumina for reaction with sulphates to produce ettringite.
Finally the lower content of the easily attacked calcium hydroxide is advantageous.

6.3 Alkali-silica reactions

A low transfer rate of alkali ions from the outside into the concrete, for example when sodium containing deicing salts are applied, may increase the resistance of microsilica containing concrete to alkali-silica reactions.
Laboratory examinations and field tests have shown, that the incorporation of microsilica to high alkali cements reduces the expansion deriving from the swelling of alkali-silica gels /39/ /40/.
Besides the lower ion transfer rate, the reduced OH^--concentration of the pore solution, due to the high reactivity of microsilica, and the ability of the low C/S hydration products to incorporate alkali ions in the lattice, reduce the formation of expansive alkali-silica gel.

6.4 Frost attack

The frost resistance of concrete has also been found to be improved by incorporation of microsilica /41/ /19/ /31/ /9/. It is suggested that the favourable effect of microsilica addition is connected with the change in pore structure.

Examinations of ice formation in the fine pores of microsilica containing cement pastes by decreasing temperatures /14/ suggest that there is no ice formation taking place at ordinary freezing temperatures, even when the paste has undergone drying-wetting cycles and is tested in a saturated state.
Thus it is suggested that the microsilica containing concrete could be made resistant to frost action without air entraining agent.

6.5 Corrosion of reinforcement

The factors influencing the corrosion of reinforcement in concrete are of two categories. The first category of factors relate to the initiation of corrosion, and the second category to the continuation of corrosion. The development of cracks in the concrete due to corrosion depends on the type and the amount of reaction products formed during corrosion.
The influence of microsilica on these factors is very complex.

The corrosion may be initiated due to a reduction in pH to below 10.

Although the chemical reactions with microsilica do reduce the pH /11/, it does not appear likely that it will fall to below a critical value unless large quantities of microsilica are used together with high w/c-ratios.

A reduction in pH may also occur due to carbonation. The rate of carbonation of microsilica containing pastes should be increased due to a lower calcium hydroxide content. It should be decreased, however, due to a lower rate of diffusion.

The present information /9/ suggests that these factors cancel out and indicates that the rate of carbonation of microsilica containing concrete is the same as for ordinary concrete, in any case when the cement + microsilica content is the same.

The second cause of corrosion initiation is the presence of a high concentration of chloride ions which causes pitting corrosion at potentials and pH values which are normally passivating. It is well known that chloride penetration rate is considerably lower in concrete made from blended cement than in concrete made from ordinary cement /43/. Similar results have been found for microsilica containing concrete in a preliminary, minor investigation /44/.
This observation is explained by the lower permeability and the diffusion barriers provided by the fine pore structure, and by the capability of CAH products and CSH products to bind chloride ions by recrystallization or by ion exchange.
It should be mentioned, however, that the chloride-binding capacity of the cement paste seems to decrease with increasing content of microsilica /12/, and that the threshold value for chloride ions is reduced at reduced pH.

Cl^- diffusion coefficients for pastes of low w/c and high content of microsilica (DSP materials) are reported to be less than 1×10^9 cm^2/sec. in comparison with ordinary Portland cement pastes at 30×10^9 cm^2/sec /45/.

When corrosion is initiated other factors determine the rate of further corrosion. These factors are oxygen diffusion and the electrical resistivity of the paste. Due to the refinement of the pore structure, the oxygen diffusion rate is assumed to diminish with the microsilica content, whereas the electrical resistivity is increased by microsilica addition /9/.

The increased resistivity is probably also a consequence of the decreased ability of ions to move in the refined pore system of microsilica containing cement paste, and may be also due to a lower ion concentration /12/.

In summary the tendency for the reinforcement to corrode is considered to be smaller in silica containing concrete than in ordinary concrete, when microsilica is added to the concrete in order to improve other properties of the concrete.

7. APPLICATIONS AND CONCLUSIONS

In the cement and concrete industry microsilica is used in three ways.

1. Microsilica is used as a cement substitute to reduce the materials' costs.
   Microsilica has been used in this way for many years in Scandinavia with good results. It has been stressed, however, that during design of the concrete mix it is important not to use the high cement equivalent factor uncritically to reduce the cement content extensively, as other properties of concrete are not necessarily influenced in the same manner as the 28 days compressive strength /46/.

2. Microsilica is used as an admixture to improve the quality of concrete and to facilitate the concrete production.

3. Microsilica is used as a raw material for the production of new materials with properties quite different from ordinary cement based products (DSP-materials) /10/.

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