Figure 10. Installation of wear-resistant Densit® end screw in powder pump.

Figure 11. Thin sheet form tool made of Densit®.

Figure 12. Mill wheel with Densit® as cement paste.

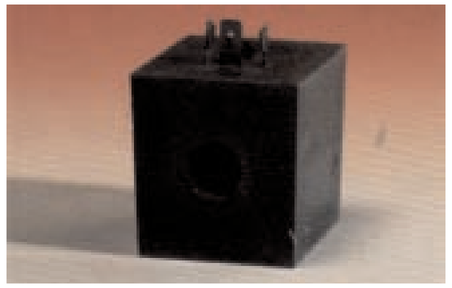


Figure 13. Experimental solenoid valve embedded in Densit® instead of epoxy.

Figure 14. Slotted grating for piglets.

This concentration of efforts in selected areas for more than 3 years has resulted in a competitive product range in each of the 4 sales areas, and further products are currently being developed. The sales work has led to such good results that Densit a/s is now operating at a profit. The share of the company's export sales currently stands at approximately 70%, but since sales take place on markets that are not typical for Denmark,

Figure 15. Safe constructed in Densit® following a successful test. The material is used today by a majority of the leading safe manufacturers in Europe.

the natural export share is considerably higher. There is thus every reason to expect continued positive development of the current business foundation.
Greater efforts with regard to product development are now planned. In the area of material technology this will take place with a Japanese licensee, with whom a close technical partnership has been forged. The development of new applications and products of Densit® will to a great extent take place in collaboration with partners from industry.

Patent situation

3 November 1988 marked the 10th anniversary of the first Densit® patent application. Since that time, fundamental Densit® patents have been issued in the chief industrial nations in Europe, in Japan, in the USA, as well as in a number of other countries.
The processing of the patent applications has taken time, in part because the applications are comprehensive, complex and protect a wide area, in part because the patent authorities in many regions are overstretched. Indeed, the Danish patent was not issued until 16 May 1988 as no. 151378.
These patents appear to be robust. In early summer 1988 a German court found in favour of Densit A/S on all counts in a case brought against a German company for patent infringement. The German company had introduced a product onto the market with properties that experience has shown can only be achieved within the scope of the Densit® patents. A closer examination, carried out by CBL, was able to provide evidence of this, and during the subsequent legal proceedings this proof was sufficient to convince the German court that a patent infringement had taken place.
Development over the last 10 years has shown that it was a good idea to apply for wide-ranging patent protection of Densit® materials, despite the fact that the development from the basic material technology to the final marketable product has proved to be considerably more investment-intensive than originally envisaged.
The development of Densit® often takes place in collaboration with partners, and there is thus a need to be able to guarantee these partners a share of the commercial results of their efforts. This can take place through patent licence agreements with exclusive rights. In this way it is possible to prevent the sale of material and commercial exploitation from being taken over by other parties as soon as the technical development and marketing development – which both demand heavy investment – have been completed.

CES/Densit a/s

Literature

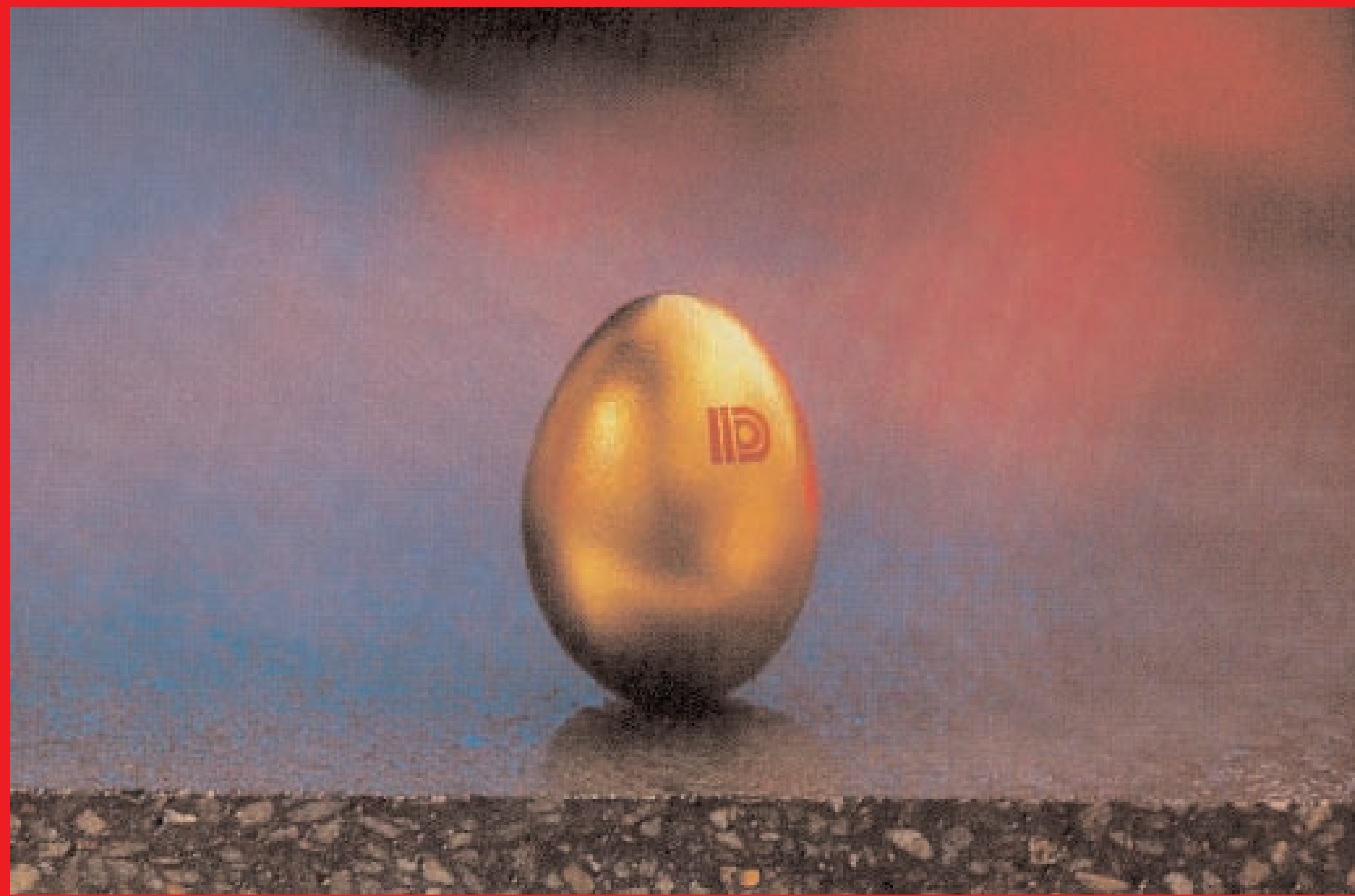
[1] Danish Patent no. 151378
[2] Bache, H. H. "Densified Cement/Ultra-fine Particle-Based Materials". Paper presented at the Second International Conference on Superplasticizers in Concrete, Ottawa, Canada. June 10-12, 1981.

CtO Cementfabrikkernes
tekniske oplysningskontor
Rørdalsvej 44
Postboks 165
9100 Aalborg
Telf. 08 16 77 77
AALBORG PORTLAND

CONCRETE ENGINEERING

10/29/1988

From CtO's field of operations



Densit® patents - their background and importance

On 3 November 1988 it is 10 years since the first Densit® patent was applied for. This marked the first result of highly focused development work on the part of Aalborg Portland that began some 20 years ago.

The following is a summary of the development process as the basis for the establishment of the patent rights, subsequent technical and commercial development in Densit a/s and the current patent situation.

Development of Densit® materials

Since the emergence of modern concrete at the end of the last century, work has gone on in many parts of the world in an effort to manufacture stronger concrete.
Just 10-15 years ago what was at that time designated high-strength concrete was characterised by compressive strengths of 60-80 MPa. The latest concrete technology enables the production of mortars and concretes with compressive strengths of up to approximately 250 MPa.
Aalborg Portland has made a significant contribution to this development, which in its case began at the beginning of the 1960s with the establishment of the Concrete Research Laboratory in Karlstrup (BFL), and subsequently continued at the Cement and Concrete Laboratory in Aalborg (CBL).
According to traditional concrete technology, the compressive strength of concrete more or less exclusively depends on the quality of the cement paste, since the strength of the aggregates is assumed to have secondary importance (fig. 1). The quality of the cement paste is in turn assumed to be a more or less clearly defined function of the water-cementitious material ratio or – in other words – of the packing density of the cement prior to hydration.
Engineer H. H. Bache led Aalborg Portland's research into this area. At one point Bache based his research on theoretical considerations and results of experiments with particle systems of other materials. These indicated that the strength of the cement paste would be increased if the size of the pores and cracks that occurred could be reduced. This could possibly be achieved by working with considerably finer cement or by filling the pores and cracks in ordinary cement with significantly finer materials, e.g. ultra-fine ground cement.
At that time, however, practical trials with fine ground cement did not lead to the desired results since surface forces made it impossible to arrange the extremely fine cement particles in a sufficiently densely packed structure.
It was – again based on theoretical considerations and experiments with other particle systems – recognised that through the addition of surface-active substances (plasticizers) and particularly intensive mixing, it would be possible to achieve a more densely packed structure, also in the case of fine particle systems. However, plasticizers only had a limited effect on the reduction of surface forces between cement particles, and this method did not achieve significant improvements in the strength of systems containing ultra-fine ground cement.
At the beginning of the 1970s admixtures appeared that were more surface active in cement systems – the so-called superplasticizers (SPT) (fig. 2). These were used both in order to make the concrete more fluid and to manufacture high-strength concrete through the reduction of the water-cementitious material ratio. This led to the Japanese researcher Hattori producing concrete in the laboratory with compressive strengths of up to 120 MPa with a water-cementitious material ratio of 0.25.

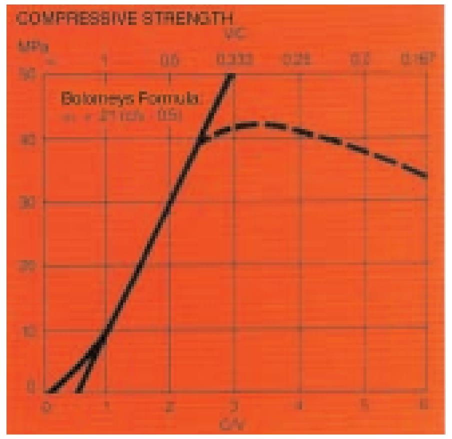


Figure 1. Compressive strength of conventional concrete as a function of the water-cementitious material ratio.

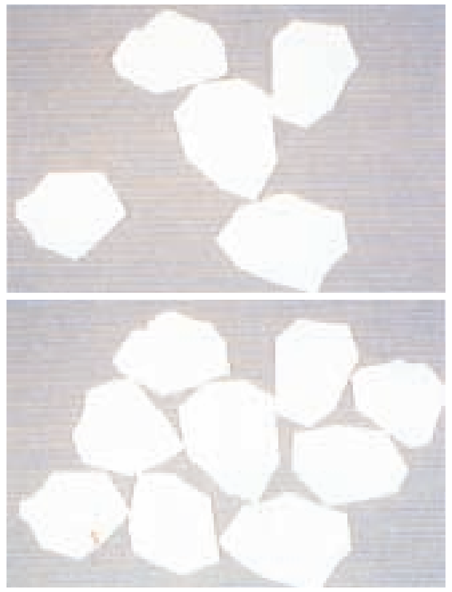


Figure 2. Packing of cement grains in conventional concrete – without (top) and with (bottom) superplasticizing admixtures.

With the appearance of SPT, tests with mixes of ordinary and ultra-fine ground cement were resumed at CBL. The idea was to produce cement pastes with a dense packing of normally ground cement combined with ultra-fine ground cement arranged in the gaps between the larger particles.
In spring 1978 the staff at CBL became aware of the potential of microsilica as a possible replacement for the ultra-fine ground cement, since it offered a number of advantages. A smaller particle size and a better grain shape (microsilica particles are spherical) would make the particles more suitable for packing in the gaps between the cement particles. The particles could be expected to make an active contribution in terms of the chemical structure, but would be much less reactive than corresponding ultra-fine cement, whose higher reactivity caused problems with dispersion and rapid setting.
At the time, microsilica had already been used in concrete for many years, in part as a convenient method of disposal (microsilica is a waste product from the metallurgic industry), and in part to improve the chemical resistance of concrete (fig. 3). The use of large amounts of microsilica in concrete, however, led to an increasing requirement for water and also declining strength.
On 8 May 1978 Bache produced the first concrete samples at CBL with densely packed cement and relatively large amounts of microsilica in the gaps between the cement particles (the ratio between cement and microsilica was approx. 3:1). Thanks to a heavy overdose of SPT and intensive mixing, the concrete was made fluid with a water requirement as low as 0.18 (water/cement + microsilica). The following day compressive strength testing of heat-cured test samples revealed that the strength of the concrete had reached a particularly promising level (128 MPa).
Initially, this marked the end of development work aimed at producing a "supercement" with a completely new level of strength and quality achieved by means of densely packed cement particles with uniformly distributed ultra-fine particles in the gaps between the larger particles (fig. 4). At the same time, it heralded the beginning of a development that has continued to this day, and which would enable the production of traditional mortar and concrete products with greatly enhanced properties. This ongoing development has also allowed the use of cement-bound materials of this type in a number of applications in which high-value materials such as metals and ceramics once reigned supreme.
The new cement pastes that were produced for the first time on 8 May 1978 demonstrated a number of properties that offered significant advantages.

Figure 3. Electron microscopy of microsilica..

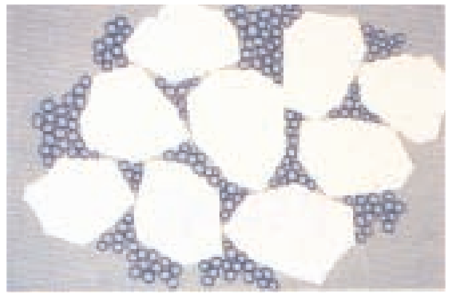


Figure 4. Packing of cement grains and microsilica in Densit®.

Their strength proved to be high in relation to aggregates such that the break – e.g. when a test piece was crushed in a compressive strength test - went through the aggregate and not around it, as is the case for ordinary concrete (fig. 5). This led to studies of the properties of mortars and concrete using strong, synthetic aggregates such as calcined bauxite. This in turn led to the development of materials with compressive strengths of up to 250-300 MPa and with other attractive properties such as good wear resistance, hardness, etc.
Furthermore, thanks to their high density, the new cement pastes had exhibited a high level of grip on reinforcement steel in the form of both ribbed bars and fine steel fibres. As a result of the materials' adhesive consistency it proved to be possible to incorporate large quantities of steel fibres, and the cement paste's powerful grip on the fibres meant that considerable toughness could be achieved in what is otherwise a particularly brittle material.
The cured material's high density resulted in a much greater resistance to chemical attack than is the case for traditional concrete and mortar products. The high density also led to other beneficial properties such as high impermeability with respect to chloride ions and high electrical resistance, two properties that in combination give the materials a high degree of corrosion resistance.

Patent protection of the new materials

The new materials were initially designated DSP materials (Densified Systems containing homogeneously arranged ultra-fine Particles). At an early stage it was clear that the new material technology offered interesting commercial perspectives, but that realising such perspectives also required heavy investment and carried considerable economic risks. It was therefore natural to look at the possibilities for securing patent protection of these new materials.
It was clear that the DSP materials as such were new. The use of microsilica in concrete was at that time quite common, but no-one had previously produced materials with densely packed cement and microsilica uniformly arranged between the particles and with the resulting low water requirement and high strength. Neither was it thought that similar systems of other types of particles had been realised earlier.
The invention was absolutely sensational, and attracted enormous attention within the profession. According to traditional technology, the mixing of large amounts of microsilica in concrete increased the amount of water required and lowered the quality of the concrete. The attractive materials could only be realised through intensive and extended mixing, the use of extremely high doses of superplasticizers and the choice of chemically compatible components.
The invention of DSP materials was therefore thought to fully satisfy the conditions for patentability, and on 3 November 1978 the first of a number of patent applications with regard to DSP materials and their applications were filed.
In order to make it easier to identify the new class of cement-bound materials in connection with planned commercial development, the designation was changed to Densit® (based on the term density) from the less informative term, "DSP materials". The designation was submitted with a view to registration. With the aim of a geographically extensive exploitation of Densit® materials, the various patent and trademark applications were filed in all of the most important industrial nations in the world.

Densit® basic patent and its scope of cover

A total of 5 patent applications were filed in Denmark within 2 years of the basic invention having taken place. These applications covered both the materials and their applications in different areas. The first patent application was the most fundamental and broadest in terms of coverage. It is chiefly concerned with the microstructure of Densit® materials, and, via more than 50 patent claims, describes a large number of different types of material. These are characterised as consisting of densely packed fine particles between which ultra-fine particles are uniformly arranged.
The broadest patent claim covers materials in which the fine and ultra-fine particles can be of practically any type. A number of more specific patent claims concern materials in which the fine particles consist of cement and the ultra-fine particles consist of microsilica. Claims of this type cover everything from ready-mixed materials, cured objects and manufacturing processes to mixes of cement, silica, plasticizers and possibly aggregates, which after the addition of water and the mixing process form the final microstructure.
Some claims specify the limits for the composition of the protected cement-based materials, and of these the following can be derived:
Cement-based materials containing homogeneously arranged ultra-fine microsilica, densely packed cement particles and super plasticizers, typically in a quantity of at least 1 per cent by weight (calculated with respect to cement and microsilica) are within the scope of the patent when the weight ratio between the mixture's water content and its cement + microsilica content is less than 0.30. The cement particles can to a certain extent be substituted by materials such as fine sand, fly ash and fine limestone.

Densit a/s

Further work with Densit® materials within the first 3-4 years of their invention increased the likelihood of the new material technology offering exciting perspectives. In 1982 Aalborg Portland came to the conclusion that the realisation of this potential would be best achieved in an organisationally separate unit. Therefore at the beginning of 1983 Densit a/s was established with the declared aim of carrying out further development of Densit® materials, both technically and commercially.
During the early years, the primary task for Densit a/s was to develop the basis for future business. The plan was to develop new products – typically in collaboration with partners from industry – and to clarify market opportunities to the extent that a decision could be made on putting the products concerned into production and launching marketing activities.
In accordance with the definition of this task, these development activities were approached on a broad front. Development of new products was sought within a wide range of very different areas.

Building and construction industry (fig. 6,7)

- surfacing for industrial flooring, balcony renovation, corrosion protection, sewage renovation, drainpipes, paving stones, embedment mortars

Mine construction (fig. 8)

- silos, chutes, pipe bends, shaft linings

Wear protection (fig. 9, 10)

- pipes and elbows, screw conveyors, impellers, panels, cyclones, hydrocyclones, power station components

Form tools (fig. 11)

- thin sheet forming, vulcanising, vacuum forming, ceramic pressing, roof tile manufacture, concrete block machines, manufacture of moulded cardboard

Other (fig. 12, 13, 14, 15)

- safes, vault walls, electrical components, millstones, livestock stable equipment, machinery elements.

The result of the wealth of practical experience with Densit® materials in different areas of application was first and foremost better knowledge of the limits for the use of the materials. In each area of application a basis was established for an assessment of, firstly, the potential competitiveness of the various Densit® solutions and, secondly, which technical and marketing efforts were required in order to launch the Densit® products onto the market.
In parallel with development efforts, sales activities were commenced for products that were deemed to be ready for introduction onto the market, and a number of Densit® materials in the form of ready-mixes with aggregates, etc., were developed and put into production.
By the middle of 1985 Aalborg Portland had invested considerable sums of money in technical development and the development of business opportunities within the area. Densit A/S's financial results were, however, not yet satisfactory, and a decision was thus taken to concentrate on a limited number of the most promising possibilities with the aim of achieving a positive result on the bottom line as quickly as possible.
In practice this decision resulted in efforts being concentrated on the following areas of application:
- the security industry (safes and bank vaults)
- industrial flooring and surfacing
- wear protection in selected industries
- the mining industry

in addition to the fact that marketing would initially be concentrated on the European market.

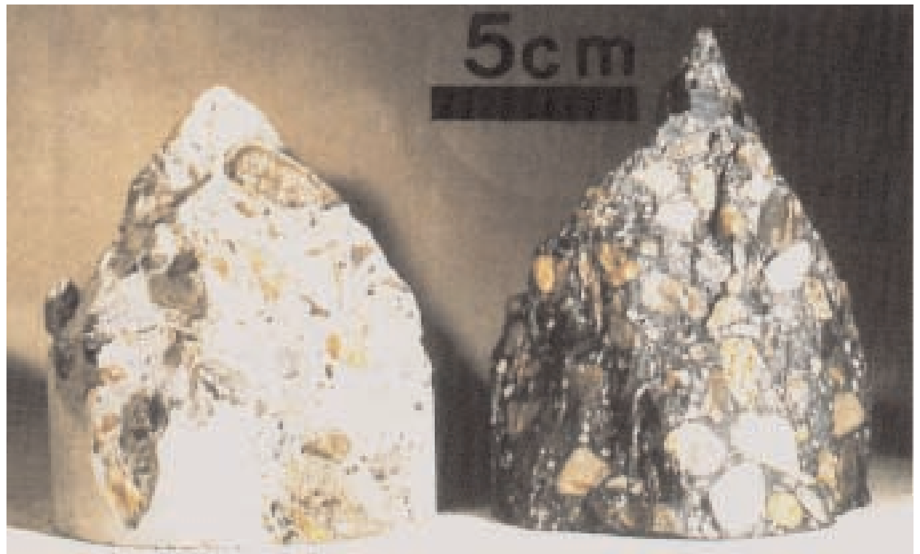


Figure 5. Crushed test samples of ordinary concrete (left) and Densit® (right).

Figure 6. Renovation of balcony walkway with Densitop®.

Figure 7. Corrosion protection of mast foundations.

Figure 8. Silo in German coalmine, 1100 m underground, lined with Densit® concrete.

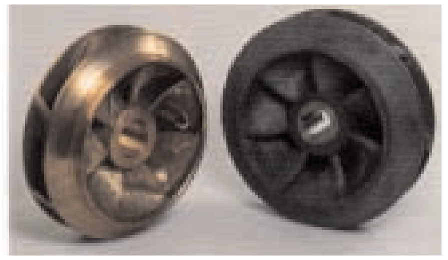


Figure 9. Impeller for same pump, constructed in bronze (left) and Densit® (right).

Apart from limited continuation of development efforts in the concrete products industry and the form tool area, the remaining development activities were put on hold.