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Executive summary

The main goal of the SANEME project was the incorporation of molecules or other nanometre-sized components (such as nanocrystals) between conducting electrodes on a silicon wafer. The gap between the electrodes was to be made using a single-step, parallel process, with the nanoscale elements assembling themselves within these “nanogaps”. This allows such “nanoscale elements” to be directly connected to conventional electronics. This hybridisation of new and existing technologies is of critical importance, as it is widely recognised that the first manufacturable molecular nanoelectronic circuits are likely to be bolt-on technologies on top of a CMOS chip, with hybrid CMOS/intramolecular electronic chips providing robust input/output interconnections to conventional integrated-circuit architectures.

The principal objectives of the SANEME project were:

- Fabrication of individual metal junctions and the incorporation and assessment of nanoscale elements within them
- Development of selectively-etched nanogaps on silicon wafers using the silicon as electrodes
- Development of selectively-etched nanogaps on silicon wafers using silicide as electrodes
- Electrical analysis of nanoscale elements self-assembled in nanogaps
- Synthesis of functional conjugated molecules with end groups
- Synthesis of Cd chalcogenide nanocrystals and anchors
- Physical and electrical characterisation of molecules and nanocrystals on surfaces
- Modelling of energy levels of molecules and nanocrystals with anchors
- Modelling of TSRAM architecture with nanoscale elements
- Fabrication and electrical assessment of MOSFETs connected to nanoscale RTDs

The coordinating partner was the University of Cambridge, UK (Dr Chris Ford and Dr Neil Greenham at the Cavendish Laboratory, Department of Physics, and Prof. Paul Raithby, originally in the Department of Chemistry and now in the Department of Chemistry at the University of Bath, UK). They carried out the synthesis of nanocrystals and molecules, and fabrication and measurement of nanogaps. The other partners were Chalmers University of Technology, Sweden (Prof. Stefan Bengtsson and Dr Per Lundgren, fabricating, processing and measuring silicon samples), the Mikroelektronik Centret (MIC), Denmark (Dr Kurt Stokbro, performing STM characterisation on surfaces and quantum-chemical modelling), and the University of Mons-Hainaut (UMH), Belgium (Dr Jérôme Cornil, performing quantum-chemical modelling).

A selective-etching technique to produce nanogaps of width below 10nm (comparable to the size of conjugated organic molecules and nanocrystals) has been devised and a patent filed. We have reliably produced gaps between GaAs layers of approximately 7nm. Polysilicon MOS (silicon-oxide-silicon) structures were also prepared and etched selectively to partially remove the thin thermal oxide and hence form a nanogap of width below 10 nm (comparable to the size of conjugated organic molecules and nanocrystals). The nanogap fabrication has been investigated, using various chemical treatments to activate the nanogap. We have measured current-voltage (I-V) characteristics of such nanogaps (with a small contact area to minimise leakage effects), with and without included nanocrystals. The nanocrystal samples show an apparent negative differential resistance but only on the first sweep of bias voltage, indicating that the cause may in fact be time dependence rather than quantised energy levels. Methods of anchoring nanocrystals to a silicon surface would need to be found in order to improve this. We have investigated the leakage currents and shown that they are not excessive. A model for the electrical properties of the intrinsic device has been developed.

We have also developed a method of using the selectively etched gap as a template for the formation of a gap between metal electrodes. We have used gold to provide a well-characterised surface for self-assembly of nanoscale elements that are terminated with anchor molecules. This offers the prospect of easy attachment of molecules to a silicon device, without having to perform complicated chemistry to attach the molecules directly to the silicon.

Silicidation (incorporation of a metal into silicon) of the silicon surfaces of the nanogap structure using Ni seems promising. We have demonstrated, for the first time, that molecules with a sulphur atom at each end will self-assemble on a nickel silicide surface. Such molecules have been used to attach CdSe nanocrystals to silicide surfaces. These results are encouraging for the future use of silicide electrode structures for molecular electronic devices containing either nanocrystals or conjugated molecules.
We have developed a method of attaching nanocrystals in selectively etched nanogaps with semiconducting electrodes. To avoid, for the present, the difficulties of anchoring molecules or nanocrystals on to silicon, we have used a GaAs heterostructure, and have shown that thiol-ended molecules will bond well to a GaAs surface. The properties of 5 nm nanogaps have been investigated, to minimise the leakage current through the supporting AlAs insulator.

We have used shadow evaporation to produce arrays of similar gaps between metal (gold or gold palladium) electrodes. The sizes are between 2 and 6 nm. We have measured current-voltage characteristics of nanocrystals with diameters between 2 and 4 nm, self-assembled in these gaps using short linker molecules to anchor the molecules within the gaps. Some of these show electrical properties and their suitability for an application in nanoelectronic devices. I-V characteristics collected at different general behaviour for a given type of sample is consistent.

The yield of working devices, and the limited reproducibility for a given sample, and between equivalent samples, but the main problems are perhaps corresponding to alignment of the molecular orbitals with the Fermi energy in one of the leads. The main problems are the yield of working devices, and the limited reproducibility for a given sample, and between equivalent samples, but the general behaviour for a given type of sample is consistent.

Selected molecules and nanocrystals have been investigated by scanning tunnelling spectroscopy (STS) with regard to their electrical properties and their suitability for an application in nanoelectronic devices. I-V characteristics collected at different molecules or nanocrystals have been measured and interpreted. The I-V characteristics are found to be strongly dependent on whether the semiconductor substrate is doped heavily p- or n-type.

We have used electronic structure calculations of small phenylene ethynylene oligomers to identify molecular features required to promote an NDR signal in molecular junctions; according to these calculations, resonant tunnelling processes can be generated in conjugated wires by introducing a twist or a saturated spacer along the molecular backbone. This is further supported by sophisticated I-V simulations, thus pointing to the fact that simple molecular modelling can prove very useful to design wires with specific functionalities and electrical characteristics for use in molecular junctions. The calculations have also rationalized a large number of recent experimental data, thus providing a unified picture of the NDR behaviour in molecular wires.

We have also developed a new modelling program (TranSIESTA) to calculate transport through molecules attached to realistic substrates. Arrays or individual molecules can be handled. This has produced important insights into the causes of the switching in molecules consisting of chains of one or more benzene rings.

During the course of the project we have developed the synthesis of new “long-chain” organic molecules with lengths of between 3 and 5 nm that can span nanogaps. These molecules have been designed to make them soluble in most organic solvents so that they are easy to deposit onto the surfaces of supports and into nanogaps. The molecules are attached to metal surfaces through specially designed “crocodile clips”, that are molecular units that contain either sulphur or nitrogen atoms and form strong bonds to the surface. The electronic properties of the “long-chain” molecules have been altered by altering the organic functional groups along the chains, in one of two ways: (i) by introducing donor-acceptor groups on either side of the molecules which introduces a polarity perpendicular to the chain direction, or (ii) by using steric control to introduce a twist into the molecules along the chain, and by altering the angle of the twist altering the electron flow along the chains. In the latter stages of the project, transition-metal-containing units have been introduced into the chains at regular intervals. The introduction of the metals means that it is easier to grow longer chains, and that there is potentially greater control over the electronic properties of the chains because the redox properties of the metals can be tailored to enhance or decrease the electron flow.

Computer simulations were used to determine the performance required for molecular nanoelectronic devices to be useful in computer circuits. One of the possible applications of molecular electronics in combination with silicon technology is for the refresh of DRAM cells. The simulations show that no molecules with resonant electrical properties published so far in the literature have suitable properties for this particular application. Thus the aim should be to find molecules which show NDR at lower applied voltages and lower current levels than the molecules published so far. If this is successful, the merging of silicon technology and molecular electronics (for instance for new generations of DRAM cells) is a realistic future path of microelectronics. This work has been published (Berg et al., Solid State Electronics, 44 2247-2252 (2000)).
Various paths for integration of MOSFETs and nanoscale elements assembled in nanogaps have been investigated. These approaches include the MOS-type nanogap structure, a test chip design intended for foundry manufacturing and a demonstrating measurement of a DRAM cell built from discrete devices connected to nanogaps with non-NDR (Negative Differential Resistance) nanoscale elements.

It was not possible to produce working devices with NDR characteristics reliably, so the final goal of a MOSFET connected to nanoscale RTDs was not achievable. However, much has been developed that bodes well for the future, although we, and all other research groups in this field, are finding that achieving reproducibility and control of such systems is not as easy as it had appeared at first.

In summary, the SANEME project has made great progress, in developing the capabilities to synthesise suitable molecules, fabricate arrays of fairly-uniform nanometre-sized gaps between electrodes, self-assembling molecules and nanocrystals in the gaps and on surfaces, and measuring the electrical properties of these systems. This is a huge achievement given the incredibly small size of the structures being used. Sophisticated theoretical models of such systems have also been developed, which will make it much easier to decide in future which new types of molecule to look at in most detail. More work remains to be done, but the partners in the SANEME consortium are in a very good position to spearhead this research in the future.
Overview of work accomplished

In this section, the tasks worked on during the SANEME project in each work package (WP) will be described briefly, together with a discussion of the successes and problems.

WP1 Development of arrays of nanogaps and electrical analysis of nanoscale elements within them

The main aim of the project was to develop processes to fabricate, on a large scale, arrays of reliable nanogaps of a chosen size (~5 nm) with good yield and reproducibility and to incorporate nanoscale elements within them.

Firstly, in Cambridge we investigated metallic gaps on Si substrates, as their properties were moderately well known. For example, gold does not oxidise, and there is no depletion region. We adopted a recently devised technique of shadow evaporation past a step made from a layer of well-controlled thickness. This allows us to make arrays of gaps, of size between 2 and 6 nm, with adequate reliability. Arrays of metal gaps were optimised to have suitably sized gaps without too many shorts or excessive leakage current. Nanocrystals or short-chain functional molecules were self-assembled (using thiol end groups as linkers) in these gaps. They each showed interesting current-voltage (I-V) characteristics indicative of the discrete energy levels and charge in the nanoscale elements. The I-V characteristics of some devices containing nanocrystals with diameters 2.4, 3.5 or 4.2 nm show steps, indicative of Coulomb blockade of single-electron tunnelling through a double-barrier structure, or of quantised energy levels within the nanocrystal. We have also measured three-ring phenylene-acetylene oligomers, with a nitro side-group. These are so short that it is very hard to make reliable contact to them, but some showed switching at particular voltages. Three-ring molecules without a nitro side-group show a tunnelling gap around zero bias, but then the current increases fairly linearly on either side. A five-ring molecule without a nitro side-group showed a similar gap, together with steps at higher bias, perhaps corresponding to alignment of the molecular orbitals with the Fermi energy in one of the leads. The main problems are the yield of working devices, and the limited reproducibility for a given sample, and between equivalent samples, but the general behaviour for a given type of sample is consistent.

The other main approach pursued was the selective etching of semiconductor layered structures, with the aim of fabricating and characterising a CMOS-compatible silicon-silicon dioxide-silicon nanogap structure. The structure is a MOSFET gate stack allowing direct integration of MOSFETs and the nano-scaled elements. Nanogaps were made by selectively etching away the oxide from the side of the MOS structure. Nanocrystals were incorporated into the gaps, but without any anchor molecules to bind them in place. A small contact area was used to minimise leakage effects. There is enhanced conduction in the presence of nanocrystals, but negative differential resistance (NDR) was observed only in the first sweep of each nanogap, and appears to be largely due to some instability of the nanocrystals in the gap. We have investigated the room-temperature leakage currents and the effects on them of substrate doping and oxide thickness. Various environments were tried, and different chemical treatments were used to activate the nanogap. Many of the wafers had heavy doping in both the silicon electrodes, to minimise the depletion depth. The samples have been extensively characterised at Chalmers as well as in Cambridge.

We have developed a method of attaching nanocrystals in selectively etched nanogaps with semiconducting electrodes. To avoid, for the present, the difficulties of anchoring molecules or nanocrystals on to silicon, we have used a GaAs heterostructure, and have shown that thiol-ended molecules will bond well to a GaAs surface. The properties of 5 nm nanogaps have been investigated, to minimise the leakage current through the supporting AlAs insulator.

We have also developed a method of using the selectively etched gap as a template for the formation of a gap between metal electrodes. This offers the prospect of easy attachment of molecules to a silicon device, without having to perform complicated chemistry to attach the molecules directly to the silicon.

A silicide process using Ni has been developed for the selectively etched MOS device structure, to optimise the electrode properties by providing a metallic surface with minimal depletion and oxidation, and to provide an anchor point for attaching the molecules or nanocrystals. CMOS-compatible nanogap structures with silicided electrodes have been fabricated and characterised. The samples have been characterised by XPS and by electrical measurements. Silicidation does occur, resulting in sufficiently good electrical contacts and low surface-leakage currents to be a valid candidate for nanogap contact improvement. The assembly of alkane dithiols, and of the three-ring phenylene-acetylene oligomer with a nitro side-group, on nickel silicide surfaces has been demonstrated for the first time. We have confirmed the presence of self-assembled monolayers using FTIR and contact-angle measurements. Monolayers of both dithiols and conjugated oligomers have been used to attach CdSe nanocrystals to silicide surfaces, as confirmed by photoluminescence experiments. These results are encouraging for the future use of silicide electrode structures for molecular electronic devices containing either nanocrystals or conjugated molecules.
The successes of this work package are the development of a number of very promising techniques for making arrays of nanogaps quite reproducibly, and the ability to self-assemble nanocrystals and functional molecules within metal gaps made using shadow evaporation. This work has taken longer than expected, with reproducibility of the measurements being a major hurdle. This problem appears to have been encountered by all the research groups working on such systems around the world. Also, it was necessary to make smaller gaps (2-5 nm) than at first intended (10 nm), since suitable long molecules were found to be insoluble. Electric-field effects in such small nanogaps are problematic, causing leakage currents along surfaces and by direct tunnelling, and probably also electromigration. Nanocrystals are unstable in such electric fields unless anchored in the gap, so it is important to develop methods of attaching them to silicon. This capability is also vital in order to self-assemble molecules in silicon nanogaps. The silicon selective-etching techniques described above that use siliconide or provide a template for metal will, however, support such attachment, and self-assembly on silicon itself is a technique that only requires more time to develop.

WP2.1 Synthesis of conjugated molecules

The synthetic programme at Cambridge, and later Bath, in the UK commenced with the repetition and development of the synthetic routes reported by Tour et. al. to prepare phenylene-based, conjugated long-chain molecules with lengths of between 2 and 5 nm so that these molecules could be tested by the groups in Cambridge, Chalmers and the Microelectronics Centre in Denmark. The first year was taken up with optimising the reaction conditions, overcoming solubility problems, and developing techniques to separate and purify the “Tour wires”. Leading on from further work by Tour and Reed, phenylene-based long-chain molecules with either electron-withdrawing or electron-donating substituents on the central ring were synthesised for use by the “measurement” groups. As the project developed, theoretical calculations from the Mons group indicated that it would be fruitful to prepare long-chain molecules that contained partially conjugated ring systems that were twisted relative to the chain direction. During the second year of the project syntheses to produce molecules with this inherent twist were developed.

Work on the synthesis and characterisation of linear, long chain organic molecules that act as spacer groups continued in the third year of the project, although not all the problems associated with the isolation of the longer chain molecules in a pure form could be overcome because of the difficulty in separating the target molecules from the reaction mixtures. The introduction of alkyl side chains on the aromatic rings of some of the units in the long-chain molecules improved the overall solubility of the larger molecules. Donor and acceptor side groups were also introduced onto the central aromatic rings of the chains to form second-generation molecules. These side groups introduce polarity in a direction perpendicular to the extended molecular chain, and also increase steric interactions along the chains. This introduces a twist into the molecules in their ground state and reduces the level of conjugation. This work was extended to introduce a range of conjugated, extended aromatic systems in place of the central arene ring. This imparts different degrees of twist to the chains, with a view to correlating the measured angle of twist with the electrical properties of the materials, and with the associated computational calculations.

The central arene ring, in the long-chain molecules, has also been replaced by metal-containing fragments, such as Pt(PR₃)₂, that retain delocalisation along the chains, but introduce different electronic properties. The spin orbit coupling associated with the presence of the heavy metal, makes a spin crossover from the excited state singlet to the excited state triplet possible, facilitating the occupancy of the triplet state.

Further investigations have been undertaken into the chemistry of the anchor molecules, used to link the conjugated molecules to the surface of the nanogaps. Initial work with thiol anchors showed that these were versatile groups for forming linkages to most metal surfaces. Work in this area has continued to establish the exact nature of the interaction between the metal surfaces and the thiol end groups, and a range of model compounds, mimicking this interaction, have been prepared. In addition, other end-groups, including nitrogen donor molecules have been investigated synthetically, so that a range of molecules that may attach to other surfaces may be developed.

The successes of this work include the synthesis of functional molecules of sufficient length to span the nanogaps (2-3nm), and the development of molecules containing parts that may rotate in an electric field, thereby potentially producing NDR. The problems faced have included the insolubility of chains containing more than 7 rings, and the poor purity of 7-ring samples. This has necessitated the development of smaller nanogaps than at first envisaged, delaying the time when electrical measurements of molecules could be made.
**WP3.1 Synthesis of Cd chalcogenide nanocrystals and anchors**

Cadmium selenide (CdSe) nanocrystals of diameters between 2 and 5 nm were made in Cambridge using chemical techniques to produce ligand-coated nanocrystals which are processible from solution. Selective bonding of the particles to gold surfaces was achieved using alkanedithiol linker molecules. Thiol-terminated conjugated molecules from WP2.1 were also used to attach nanocrystals to surfaces, and the effect of molecular length and rigidity on the assembly process was investigated.

Attachment of nanocrystals to semiconductor surfaces was an important target for device fabrication, and has been successfully demonstrated on GaAs surfaces using dithiol linkers. These assemblies were characterised using luminescence, infra-red spectroscopy, ellipsometry, and scanning tunnelling microscopy. The electrical properties of nanocrystals on metallic and semiconducting surfaces were investigated as described in WP2.2/3.3.

**WP2.2/3.2 Modelling of energy levels of molecules and nanocrystals**

At Mons in Belgium, we developed molecular electronic structure calculations to identify features required to promote an NDR signal in molecular junctions; according to these calculations, resonant tunnelling processes can be generated in conjugated wires by introducing a twist or a saturated spacer along the molecular backbone. This is further supported by sophisticated I-V simulations, thus pointing to the fact that simple molecular modelling can prove very useful to design wires with specific functionalities and electrical characteristics for use in molecular junctions. The calculations have also rationalized a large number of recent experimental data, thus providing a unified picture of the NDR behaviour in molecular wires.

At MIC in Denmark, we developed a new modelling program (TranSIESTA) to calculate transport through molecules attached to realistic substrates. Arrays or individual molecules can be handled. This has produced important insights into the causes of the NDR in phenylene ethynylene oligomers and benzene rings.

The successes of this work include the development of two different, complementary, modelling techniques for predicting and understanding the electrical properties of molecules attached to electrodes. It has been possible to compare these two techniques and to home in on the most likely explanation of NDR behaviour in molecular wires.

**WP2.3/3.3 Electrical characterisation of nanocrystals and molecules on surfaces**

At MIC in Denmark, we have used ultra-high-vacuum scanning tunnelling microscopy (STM) at temperatures down to below 10 K to investigate selected molecules and nanocrystals with regard to their electrical properties and their suitability for an application in nanoelectronic devices. I-V characteristics collected at different molecules have been measured and interpreted. Additionally the influence of the molecule’s coupling to the substrate has been studied by slight variations in the molecule-substrate coupling. I-V characteristics of nanocrystals on highly doped p- and n-type semiconductor substrates have been measured and interpreted.

We have shown that oligo phenylene ethynlenes (OPE) molecules form a standing monolayer on a GaAs substrate, but tilted with respect to the substrate. The monolayer growth saturated after about 21 hours, which is nearly two orders of magnitude slower than the growth of alkanethiol self-assembled monolayers (SAMs). We refer this slow growth to effects of steric hindering due to the stiff and more bulky structure of the OPE molecules compared to alkanethiols. Further, our results indicate that the thiol end group cannot be broken, and the structure of the as-prepared SAM consists of thiol-bonded molecules with an angle of 39° to the surface normal. At low temperatures, the films show negative differential resistance at voltages of typically 2.8 V, perhaps when the middle ring of the molecule is rotated. Since in both experiments the second thiol bond is not covalently bonded to the tip electrode, the reason for this difference is still not clear. Possibly a collective effect of the surrounding molecules constrains the rotation of the middle ring inside an OPE monolayer, while the molecule has a higher degree of freedom inside an alkanethiol matrix. As a result, we expect that the electronic behaviour of OPE molecules inside a nanogap will depend strongly on the dimension of the nanogap (i.e. on the question, how close it is to the case of an ideal SAM), and to the binding situation at the metal-molecule interconnects. Experiments with surface-sensitive techniques like scanning tunnelling spectroscopy (STS) will only give a first indication if a molecule shows a switching behaviour inside a nanogap or similar storage element.

The successes of this work include the characterisation of the various nanoscale elements on the surfaces to be used in nanogaps. This shows us what will work, and where the problems lie. For example, surface depletion (band bending) effects have been shown to cause significant problems for nanocrystal measurements even on highly doped surfaces. The work has, however, highlighted how difficult such measurements are, partly due to the lack of reproducibility of measurements of individual molecules or nanocrystals, a similar problem to that found with nanogaps.
WP4.1 Modelling of TSRAM architecture with nanoscale elements

Simulations have been done to judge on the feasibility of the TSRAM cell with molecular RTDs as well as to determine the required properties of the molecular elements to achieve a functioning TSRAM cell. A model of the leakage currents and parasitic capacitances of the MOS-type nanogap structures was made. In view of the demands on the electrical characteristics of a functional TSRAM cell, the issue of control of the total leakage current is highly relevant. Five parasitic contributions besides the active edge (nanogap) current were included in the model.

This work has been successful in pointing the way to the types of properties required of nanoscale devices if they are to be useful in the medium term as add-ons to existing types of devices such as DRAMs. It shows that none of the known molecular systems have the right properties, and thus molecules that exhibit NDR at much lower voltages (<0.5 V) need to be found.

WP4.2/4.3 Fabrication and electrical analysis of MOSFETs connected to nanoscale RTDs

Various paths for integration of MOSFETs and nanoscale elements assembled in nanogaps have been investigated. These approaches include the MOS-type nanogap structure, a test chip design intended for foundry manufacturing and a demonstrating measurement of a DRAM cell built from discrete devices connected to nanogaps with non-NDR (Negative Differential Resistance) nanoscale elements.

We conducted design activities with the aim of rapidly allowing the production of a test chip using foundry services. The chip is a DRAM cell prepared for connecting two RTDs to transform the memory cell into a TSRAM cell. The cell also includes a read-out amplifier and input protection.

Extensive electrical characterisation of the MOSFET-based nanogap structures was performed. Some conclusions are that the surface leakage after activation can be controlled down to below pA level for non-conducting surface layers and that the edge activation leads to initially highly unstable edge currents for all treatments. After repeated measurements the edge current is stabilized and shows scaling with edge length.

We have demonstrated a CMOS-compatible MOS-type nanogap structure connected to a DRAM-like cell built of discrete devices.

The success of this work includes designing suitable test TSRAM cells and understanding the leakages and parasitic effects that will occur in such cells, so that when or if nanoscale devices exhibiting suitable NDR behaviour can be obtained, it will be quite easy to include them in a test TSRAM cell. Disappointingly, no working TSRAM cell has been demonstrated in the project. The main obstacle to doing this comes from the lack of stable nanoscale Resonant Tunnelling Diodes (RTDs). To date, no molecules or other nanoscale objects with the required properties have been published. However, the various discrete components of a TSRAM cell have been linked together as a preliminary Demonstrator, to show that there are no obstacles to making such a device when and if suitable nanoscale RTDs become available. The objectives of integration of nanoscale elements with conventional CMOS circuitry, and the TSRAM demonstrator remain a long-term goal that it turned out could not be achieved on the time-scale of the project. Before the project started it had seemed likely that suitable nanoscale elements would be fairly easy to obtain, but the experience of groups around the world in the last few years is that the initial pace of progress has slowed, as researchers try to move beyond one-off devices to being able to reproduce effects in many devices at will.

Information dissemination and exploitation of results

We have a web-site http://www.sp.phy.cam.ac.uk/SPWeb/research/saneme/.

The following papers have already been published, submitted or are in preparation:


The following papers were presented at conferences:
29. “A study on integration of molecular devices into CMOS-compatible technology”, J Berg, M S Kabir, P Lundgren, and S Bengtsson, abstract sent to Nordic Semiconductor Meeting, Tampere (2003). A patent (International Patent Number WO0237571, “Small-scale structures”) incorporating background IPR from the University of Cambridge and foreground IPR from both Cambridge and Chalmers was filed in November 2000. Unfortunately, despite approaching many companies for further funding of the patent, all regarded the work as not close enough to market to be viable, and so the patent has now lapsed.