Danish Solid Oxide Fuel Cell project: DK-SOFC 1997-1999

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Preface

It was decided medio 2000 at the 22th DK-SOFC programme board meeting to change the format of annual- and final reporting from the Danish Solid Oxide Fuel Cell projects. From then on, a compilation of reprints of papers published in the open literature will be printed and distributed each year. At the end of each three-year period also a summery, like the present one will be made. These will be sent to the sponsors, collaborating institutions and other interested parts.

Executive summaries of the individual projects will also appear every year, but these will have a restricted circulation. The present summary report covers the years 1997-1999, but due to the time constant involved in publishing, several results from the project, first appearing during the year 2000, will also be referred to. It is our intention to make scanned versions of the publications available on CD in the future.

The four main goals for DK-SOFC in the three year period were:

1) lowering of the operation temperature from about 1000°C to 800 - 850°C keeping the cell area specific internal resistance (ASR) below 0.4Ωcm^2;
2) development of a mechanically strong anode supported cell with a gas tight 20 - 40 μm YSZ electrolyte;
3) achieve a improvement of cell materials and contacting to metallic interconnects in order to make a basis for further improvements in a following programme, e.g. development of a zirconia based electrolyte with improved conductivity;
4) obtain know-how and patents as a basis for a commercialisation of SOFC in co-operation with Danish industry and electricity utilities.

All four main goals have been achieved. The DK-SOFC programme is continued with the main goal of preparing the SOFC scale-up and commercialisation through demonstration of a Danish cell production technology. This activity started by the beginning of year 2000.

Seven persons at AFM, Risø have contributed to the writing of the individual résumés. The name of the person responsible for a given paragraph is shown in parenthesis after the paragraph title. The full length papers and Ph.D. theses referred to in this summary report can be obtained from the authors. The layout of this final report was cast by Carsten Bagger, the project leader of DK-SOFC during 1/1 1998-2001. Carsten Bagger regrettably died at the age of 55 on January 20th 2001.

Finn Willy Poulsen, Editor

Research-groups in DK-SOFC and contact person(s)

Afdeling for Materialeforskning, Forskningscenter Risø (Carsten Bagger=, Søren Linderoth, Mogens Mogensen); Institut for Kemi, DTU (Torben Jacobsen); Kemisk Institut, Syddansk Universitet(Eivind Skou); IRD (John Engell); Haldor Topsøe A/S (Niels Christiansen)

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Dansk resume

(C. Bagger, M. Mogensen, F.W. Poulsen)

Slutrapport for brændselscelle-projektet DK-SOFC 97-99

Med afslutningen af 3-årsperioden 97-99 skal ifølge tidligere vedtagen skik udsendes en slutrapport, der dækker perioden. Efter perioderne 90-92 og 93-96 udsendtes klassificerede slutrapporter, der i nyskrevet, mangesidet tekst med referencer detaljeret opsummerede udviklingsforløbet for brændselscelle-forskningen. En uklassificeret rapport blev udformet på grundlag af den klassificerede og kunne bruges offentligt (bestilles fra Biblioteket, foræres til vennerne). Det var et meget stort (læs enormt) stykke arbejde at lave disse slutrapporter og nyttevirkningen heraf har ikke stået i et fornuftigt forhold til indsatsen. Ved afslutningen af 97-99 har vi derfor foreslået og af følgegruppen (følgegruppemøde 22) fået accepteret at udsende en samling af vore publikationer gennem de tre år sammen et meget kort summary af materialet. Da publikationerne er uklassificerede undgår vi diskussioner om klassificerede oplysninger. Vi kunne tænke os at forme denne rapport som et pænt "visitkort" for DK-SOFC, hvorfor den udbygges med en referenceliste for offenlige publikationer for de foregående år (instruks fra Casten Bagger, juli 2000).

De 4 hovedmål for DK-SOFC 97-99

1) sænkning af driftstemperaturen fra 1000°C ned til 800 - 850°C , under bevarelse af en areal specifik indre modstand (ASR) for cellerne på højest $0.4\Omega cm^2$,
2) udvikling af mekanisk stærke anode-bårne celler med 20 - 40 μm tyk gastæt YSZ electrolyte;
3) fortsat forbedring af cellekomponenter (herunder zirconia-baserede elektrolytter med bedre ledningsevne) og forbedring af kontakt mellem cellematerialer og metallisk interconnect;
4) opbygning af know-how og udtagning af patenter, der muliggør kommercialise-ring af SOFC teknologien sammen med dansk industri og elproducenter.

Alle fire mål blev nået. Startende år 2000 er SOFC-projektet fortsat med det hovedmål at muliggøre opskalering og demonstration af SOFC-celler.

Opbygningen af denne slutrapport

DK-SOFC projektets relation til parallelt løbende EU-projekter beskrives i afsnit 1.
I afsnit 2-5 redegøres for udvikling af celler, små stakke, interconnect, anoder og katoder. Den nødvendige mere langsigtede udforskning af alternative SOFC-materialer beskrives i det sjette afsnit;- og endelig opsummeres i afsnit 7 de mere teoretiske og eksperimentelle fremgange. De 7 resumeer er skrevet af 7 forskellige personer, hvorfor stilen uundgåeligt varierer. Reference-listerne for hvert af de fire år fra 1997 inkl. 2000 er tilstræbt at være komplette. Dette medfører at en række publikationer er medtaget, som også vedrører fra EU- og NEFP- finansieret forskning. Det fremgår af de respektive publikationers "Acknowledgement"-sektioner, hvilke sponsorer, der er tale om. Det skal her understreges at vor deltagelse i internationale SOFC -projekter skyldes det teknologiske og videnskabelige niveau vi nået via den lange og kontinuerlige støtte fra Energistyrelsen til DK-SOFC.

Common abbreviations and acronyms

AC alternating current
ACC anode current collector (layer)
ASR area specific resistance of electrode or entire cell
B.E.T. method for determining specific surface area
C cathode
CCC cathode current collector
CG4 40% gadolinium doped ceria for anodes
CORE-SOFC EU project 2001-2004
CV cyclic voltammetry
DC direct current
DTU Danish Technical University
EFP Energistyrelsens energiforskningsprogram
EIS electrochemical impedance spectroscopy
HTAS Haldor Topsøe A/S
ICM interconnect material
IDUSOFC improved durability of SOFC, EU project 1997-2000
IEA International Energy Agency, initiative for SOFC collaboration
IRD Danish R&D Company in Svendborg
IS impedance spectroscopy
LOCO-SOFC, low cost fabrication of SOFC, EU programme
LSCV strontium doped lanthanum chromate with vanadium, interconnect material
LSFM strontium doped lanthanum cobaltate/ferrate for cathode
LSM strontium doped lanthanum manganate, cathode material
MF-SOFC EU-project 2000-2003
NDE non destructive testing for defects
NEFP Nordic Energy Research Programme
OCV open circuit voltage
SOFC solid oxide fuel cell
TEC thermal expansion coefficient
TG thermogravimetry
TPB triple phase boundary
TZ8Y Tosoh standard 8 mole % Y_2O_3 doped zirconia
XRD x-ray (powder) diffraction
YSZ yttrium doped zirconia for solid electrolyte

Overview of SOFC programs, Relations to EC-projects, Assessment of Cell/Stack economy

(Søren Linderoth)

The Danish EFP 1997-99 program aims at establishing a Danish production of Solid Oxide Fuel Cells, with an option for establishing Danish stacking technology, Bagger et al, 1999) and (Mogensen et al , 1999). The program builds on results generated partly in the EFP 1993-1996 program, and leads on to another EFP program scheduled for 2000-2004. A total of 200 Mkr (approx. 27 M Euro) was invested over the period 1990-1999.

Besides the national efforts, the participants have at the same time been involved in EU-projects on SOFC. In the IDUSOFC (Improving DUrability of SOFC, 1996-98) the focus was primarily on the investigation and improvement of the component life while keeping or reducing the costs. This project was followed by LOCO-SOFC (LOw COst fabrication of SOFC, 1997-00) where materials- and fabrication cost was focussed upon, while improving the overall performance. Based on achievements in the above mentioned projects Risø is now engaged in MF-SOFC (2000-03) and CORE-SOFC (2001-2004).

An earlier systems analysis pointed out that the ceramic interconnect module with gas channels was the cost-controlling component. A central breakthrough achieved in the program has been the general lowering of the operation temperature to accommodate cheap interconnect materials(Bagger et al, SOFC VII, 1999).[1] By improving the electrodes and establishing a 20 μm electrolyte on an anode-support, internal resistances of about 0.3 Ωcm^2 have been achieved at 850°C. This is equal to what was earlier measured at 1000°C on electrolyte-supported cells. Through this lowering of the temperature, the interconnect material is no longer limited to ceramics or advanced alloys (Plansee), ferritic steels are applicable. Thereby the calculated stack cost has been reduced to about 300 Euro/kW, as compared to the 500 Euro/kW set forward by an EC strategy for 2005 (ref.: A Fuel Cell Research, Development and Demonstration Strategy for Europe up to 2005, 1998 Edition).

Arguments have been presented for considering SOFC technology not only in fossil power generation, but also as a power storage device (reversible electrolysis) in the renewable energy discussion (Mogensen anf Bagger, 1998).

Cell- and stack development and performance

(Peter Halvor Larsen)

Cell development

The DK-SOFC high temperature cell (1000^{o}C) is based on a 200 μm thick electrolyte as the supporting element. However, the thermal activation of the electrolyte conductivity necessitates a thickness reduction to approximately 15-25 μm to achieve a sufficiently low conductivity allowing operation in the lower temperature range.

Such a thin electrolyte is incapable as cell support. Therefor a cheap, scaleable method for producing thin, strong, flexible and efficient anode- supported cells has been developed, (Primdahl, Jørgensen, Bagger & Mogensen, 1999) and (Primdahl, Jørgensen, Bagger & Kindl, 2000). The manufacturing method is based on tape-casting and spray painting.
The thin anode-supported cells have the following characteristics:

- The NiO/3YSZ supports are 200-250 μm thick and the 8YSZ electrolytes are 10-25 m thick.
- Cell performance of 420 mΩcm^2 at 850^{o}C in hydrogen vs. air was demonstrated.
- The fracture strength of the half-cells (anode support + electrochemically active A-layer and electrolyte layer) was observed to exceed 150 MPa.

Non-Destructive Evaluation of Electrodes

During development and testing of new advanced ceramic materials for SOFC it is essential to ensure that components are free of defects. For the performance of the electrodes it is important that there are no delaminations between substrate and electrode layers.
Results from two NDE techniques: ultrasonic through-transmission and Lock-in thermography applied on SOFC cells were evaluated, (Borum et al, 1998). Both techniques were able to detect small delaminations between electrolyte and electrode. However, the sensitivity for small defects, approximately 1 mm in diameter, was higher for the ultrasonic through-transmission than for the lock-in technique. Also thickness variations of the electrode layers can be detected with the ultrasonic through-transmission technique. Specimens with artificial defects were used to evaluate and compare the NDE-techniques. The thermographic Lock-in technique has the highest potential for on-line measurements, despite the lower sensitivity. Ultrasonic through-transmission has a higher probability for defect detection but requires immersion and access to both sides of the specimen.

Ceramic-Metal Composite interconnects

A recent practice is to fabricate an SOFC interconnect as a ceramic-metal composite plate, where the metal side (Cr-Fe alloy) and ceramic side (Chromite) are exposed to the reducing atmosphere and air, respectively. The use intermediate layers of relatively cheap gold sheets of μm thickness and $LaCoO_3$ allow to dramatically decrease the interface resistance between metal and ceramic and low values of less than 100mΩcm^2 are obtained. However, even cheaper and better contacting methods are being persued in the current projects.

Development of Sealing Materials

Formation and properties of sealing materials for SOFC stacks that fulfil the necessary requirements has been investigated. The work comprised analysis of sealing material properties independently, in simple systems as well as tests in real SOFC

stacks (Larsen, Ph.D. Thesis 2000). The analysed sealing materials were based on pure glasses or glass-ceramic composites, having B_2O_3, P_2O_5 or SiO_2 as glass formers, and the following four glass systems were investigated: $MgO/CaO/Cr_2O_3$-Al_2O_3-B_2O_3-P_2O_5 (Larsen & James, 1998), MgO-Al_2O_3-P_2O_5, MgO-Al_2O_3-P_2O_5-SiO_2 (Larsen, Berg & Poulsen, 1998) and BaO/Na_2O-Al_2O_3-SiO_2.
All the phosphate based systems showed reasonable chemical stability with respect to the SOFC components to which they were sealed, and a composition based on $30MgO$-$15Al_2O_3$-$55P_2O_5$ performed reasonably well during short-term stack test. However, volatile losses were observed, and this was especially the case under anodic conditions (low pO_2). During heat treatment at 1000^oC devitrification of the glasses was observed, resulting in a significant change of the material properties. Stability analysis of the crystalline phases in or close to the glass-forming region of the ternary MgO-Al_2O_3-P_2O_5 system revealed, that only the orthophosphates were stable under anodic conditions. A comparative study of the effect of volatiles from phosphate and silicate based glasses revealed that severe reactions took place between volatile P_2O_5 and the anode components, whereas no reaction was seen in the case of silicate based glass. Silica based systems were found to provide long term stable seals, this was especially the case for composite seals having a ceramic filler material. A patent on glass sealings has been applied for (Larsen, Larsen & Bagger, 1999)

Development of cathodes

(Mette Juhl Jørgensen)

The work has mainly involved fundamental studies of the classical SOFC cathode material lanthanum strontium manganate ($(La_{1-x}Sr_x)_{1-z}MnO_{3\pm\delta}$, LSM), which is of pervoskite type ABO_3. In addition the oxygen stoichiometry and oxygen transport kinetics of Fe doped LSM ($La_{1-x}Sr_xFe_{1-y}Mn_yO_{3\pm\delta}$, LSFM) has been investigated using thermogravimetry.

Model experiments were performed with a cone shaped LSM electrode pressed into contact with an yttria stabilised zirconia electrolyte (Odgård & Skou, 1997). The results suggest that the active reaction zone is extended onto the electrolyte surface, when the electrode is polarised. Impedance analysis suggested that a charge transfer process is rate limiting at high frequency and that a mass transport process is limiting at low frequency.

Electrochemical characterisation of potential SOFC cathode materials often involves impedance spectroscopy (EIS) (Jørgensen, 1999). Using EIS care must be taken during interpretation, as there are several factors which are not related to the reaction under study (oxygen reduction), which may influence the measurement results. These are among other things the parameter settings on the equipment and that the electrode polarisation resistance is time dependent during current flow. Further, the reproducibility of both the overall polarisation resistance and the frequency distribution of the impedance is limited from sample to sample. Despite these limitations EIS is considered to be an important tool with respect to kinetic investigations but its limitations must be taken into account during interpretation of the results.

The variation in oxygen stoichiometry of $(La_{1-x}Sr_x)_{1-z}MnO_{3\pm\delta}$, $x \leq 0.15$, $0 < z < 0.10$, $y \approx 0$ with oxygen partial pressure has been studied. Linear sweep voltammetry measurements are performed on an oxygen pumping cell. The oxygen stoichiometry depends on whether the A-site charge deficiency is imposed by Sr-doping or by A-site vacancies. In air at temperatures between 25 – 1000°C $LaMnO_3$ will expel Mn_3O_4 when A/B is less than 0.96. The phase segregation may influence the long term stability of a working SOFC if large overvoltage variations are encountered.

The kinetics of oxidation of LSM in oxygen has been studied using time resolved synchrotron x-ray powder diffraction methods (Andersen et al., 1998) The gas/solids reaction

$$La_{1-x}Sr_xMnO_{3.00} + \delta/2O_2 \rightarrow La_{1-x}Sr_xMnO_{3.00+\delta}$$

was studied. The solids remain of single phase during the reaction. The structural rearrangement of the solid during the reaction is fast. The oxidation of LSM with x = 0 or x = 0.10 is faster than that of LSM with x = 0.15.

Oxide ion transport in $La_{1-x}Sr_xMnO_3$ was investigated using thermogravimetry, (Kjær & Skou, 1998) and (Mikkelsen & Skou, 2000). The processes involved are surface exchange of oxygen and oxygen diffusion. The temperature dependence of the diffusion reaction is much larger than the temperature dependence of the surface reaction. The surface reaction gets more and more important as with increasing temperature. The results indicate that at temperatures above 830°C the surface exchange process is rate limiting.

Similar investigations were carried out on LSFM. LSFM is found to be overstoichiometric with respect to oxygen in air at temperatures between 700°C and 1000°C. The overstoichiometry decreases with increasing degree of substitution. Diffusion coefficients in the range of 10^{-11} – 10^{-15} cm^2/s are found. The values are independent of particle size and are thus assumed to be bulk values. The diffusion coefficients decrease upon increasing Sr- and Fe-substitution.

In summary the results indicate that some of the LSM type of materials may be unstable with respect to oxygen stoichiometry and phase segregation during operation, which may affect the long term performance of SOFC (Cassidy et al , 2000) and (Jørgensen, Holtappels & Appel, 2000) and (Holtappels et al, 1998).

Development of Anodes

(Søren Primdahl)

Substantial additions to the existing knowledge of limiting processes in anodes and performance of these have been generated. These achievements fall in three main groups:

♦ The importance of non-electrode related limitations during test and how to handle these contributions,

♦ Basic properties of Ni/YSZ cermet electrodes in H_2/H_2O and how to increase the performance of these,
♦ Basic properties of $Ce_{0.6}Gd_{0.4}O_{1.8}$ ceramic electrodes in H_2/H_2O and H_2O/CH_4 and how to make these applicable in cells.

♦ Measuring impedance of high-performance Ni/YSZ cermet anodes using a reference electrode in a stable atmosphere typically reveals three distinct impedance arcs taken to represent three limiting processes (Primdahl & Mogensen, 1997). By varying the test geometry one of these arcs has been demonstrated to arise in conversion between H_2 and H_2O by the imposed AC amplitude (Primdahl, SOFC V,1997) and (Primdahl & Mogensen, 1998). Further studies demonstrated another of these arcs to arise in diffusion limitation in a stagnant gas layer outside the <50 μm electrode structure(Primdahl & Mogensen, 1999). Both effects are demonstrated to be suppressed in practice in measurements on symmetrical samples in one atmosphere using auxiliary electrodes. These findings are used repetitively in characterisation of anodes at Risø and have been recognised by electrode developers world-wide.

♦ The Ni/YSZ cermet electrode has been used over several years by most SOFC developers. The identification of conversion and diffusion impedance led to far better resolution of the last impedance arc for Ni/YSZ cermet electrodes, demonstrated to arise in the electrode itself. The electrode mechanism is generally believed to relate to the vicinity of a triple phase boundary (tpb) defined by the contact periphery of percolating Ni and YSZ in the presence of open pores. Earlier work on reviewing literature has indicated that H_2 is available on the surface of Ni in excessive quantities. Despite this an isotope effect is demonstrated for the electrode response at temperatures of 850°C and less, indicating a hydrogen access limitation. By cyclic voltammetry the absence of O-species on Ni below the potential for Ni/NiO has been documented (Zachau-Christiansen & Jacobsen, 1998) . General considerations of the solubility of species in Ni and YSZ combined with diffusion rates indicate that the electrode processes can occur within μm from the tpb, and that it is likely to involve both the Ni and the YSZ surface. Studies in CO/CO_2 atmosphere show periodic instability and suggest some kind of surface property variance with gas coverage.
The active thickness of such Ni/YSZ cermet electrodes in H_2/H_2O has been demonstrated to be about 10 μm at 1000°C (Brown et al, 1998, 2000) which have led to a technical treatment of the electrode as a 20 μm active zone with an overlying porous current collector. Addition of Mn to the active Ni/YSZ structure has been demonstrated to decrease the electrode impedance, thus increasing the cell performance. This effect has been patented (Primdahl et al, patents 1998, 1999). Most of the impedance characterisations of Ni/YSZ electrodes and the identification of gas conversion and diffusion impedances have formed part of the backbone of a Ph.D. study (Primdahl, Ph.D. Thesis, 1999). Despite these efforts, the exact mechanism and location of the limiting steps in Ni/YSZ cermet electrodes have not been established.

♦ Mixed conducting ceramic electrodes are considered for oxidation of relatively dry methane in SOFC. This interest in replacing the Ni/YSZ cermet electrode is mainly based in the catalytic properties of Ni, which will cause carbon deposition by methane cracking and thereby anode failure during operation. Another technical advantage of ceramics is low volume instability during change of pO_2 as opposed to

the nickel metal/oxide phase transition. $Ce_{0.6}Gd_{0.4}O_{1.8}$ (CG4) with a minimum of volume instability has been studied at Risø (Marina et al, 1999a-c). Due to earlier reports of extensive detrimental reactions at the CG4/YSZ electrolyte interface during high temperature sintering, an extensive study of possible interface layers has been conducted.(Jørgensen et al, SOFC V,1997). Some layers provided sufficient adhesion of the electrode, others did not severely reduce performance. The best compromise was found to be Ni applied under reducing sintering conditions. Inspired by cathode development work a layer of YSZ scales has been sintered onto the electrolyte before applying the electrode to cells (Jørgensen et al, 3rd SOFC Forum,1998) and (Jørgensen et al, SSI 1999). This type of electrodes exhibit adequate mechanical stability and good performance in H_2/H_2O. Additionally it was documented that CG4 is not a significant catalyst for oxidation or reforming of methane, and therefore the electrode should be provided with an additional catalyst for technical applications.

Modelling of materials/cell/stack performance, internal reforming and mechanical aspects

(Peter Vang Hendriksen)

Mathematical modelling has been used extensively as an assisting tool in the development work, addressing processes both at electrode-, cell- and stack-level.

Internal reforming
Internal reforming of methane in the solid oxide fuel cell stack is an interesting option as it allows significant simplification of the balance of plant and thereby reduction of cost as compared with external reforming. Having the strongly endothermic steam reforming process occurring inside the stack is not without problems, however. Ni is an excellent catalyst for the steam reforming process and as the Ni surface area in state-of-the-art Ni/YSZ anodes is very large, the steam reforming rate on Ni/YSZ anodes will be extremely high. This results in large temperature gradients in the stack, which may result in built up of detrimental mechanical stresses. This problem has been treated both at stack level (Hendriksen, 1997) and with a detailed description at the electrode level (Sunde, 1997). The treatment at the stack level is based on a continuum-model describing heat-, mass- and charge-transfer processes in the stack. In the stack level study the optimum steam reforming rate per unit cell area is deduced to set a target for the development of anodes suitable for internal reforming. The targeted level is ca. 10 times slower than for the DK-SOFC anodes at the time. An anode capable of operating with this steam reforming rate will results in a reduction of the maximum tensile stress in the electrolyte by a factor of two and a reduction in air flow of 40 % as compared with the reference case based on the 1997 state-of-the-art anode.

The electrode model (Sunde, 1997) is a one-dimensional model describing diffusion and chemical and electrochemical reactions in the anode taking its detailed composite character into account. Key parameters describing the electrode structure, Ni-fraction, Ni-particle size, electrode thickness and porosity were varied and the effects on steam reforming rate and the electrode polarisation resistance calculated. It was found that

the most important parameter for the steam reforming rate was the electrode thickness – the rate simply scales with electrode thickness. The thickness could be reduced down to 10 μm without affecting the electrochemical performance of the anode. Controlling the steam reforming rate by reducing the porosity of the anode or by increasing the particle size were found to be much less efficient than decreasing electrode thickness.

Design of three electrode set-ups for characterisation of electrode performance
To characterise a single electrode under polarisation one has to use a three electrode set-up where a reference electrode is placed on the cell in addition to the working electrode and the counter electrode. This is a classical technique in electrochemistry. In solid oxide fuel cells the solid character of the electrolyte and typical dimensions of the electrolyte on technologically relevant cells makes proper placement of the reference electrode a non- trivial task. This topic has been addressed in five papers (Primdahl, Hendriksen 1996), (Bonanos and Winkler, 1997), (Jacobsen and Skou, 1997) (Winkler et al. 1998) and (Hendriksen and Primdahl 1999). In these papers it is shown, that placing the reference electrode next to either working or counter electrode on thin electrolyte cells will result in highly erroneous determination of electrode polarisation resistances. The literature holds numerous examples of studies where this has been done. The above model-papers clearly show that such results can not be considered credible. In the 1998 and the 1999 papers specific requirements to three electrode cell geometries for these to be considered scientifically sound can be found.

Dimensional instability under reduction and its consequences.
The 500 W stack tested in the DK-SOFC project in 1995 used ceramic $(La,Sr)CrO_3$ interconnect plates. The plates were observed to bend under test and most of the plates were cracked after the test. This was a identified as a limiting factor for stack performance. The problem originates from an expansion of the material on reduction. The molar volume varies with oxygen content in the material, which is determined by the oxygen activity in the surrounding atmosphere. When used as an interconnect, the material is exposed to an oxygen activity gradient. This results in a strain distribution in the component and a built up of mechanical stresses. The prediction of the magnitude of such induced stresses and their mechanical consequences for stack and component integrity has been treated in two papers (Hendriksen and Jørgensen, 1996) and (Hendriksen et al., 1999). In the first paper the stress distribution in the bipolar plate (2D-treatment) is calculated under realistic operating conditions. From a comparison of the calculated stress level with reported strengths and calculated strain energy release rates with fracture energies the maximum tolerable expansion on reduction of the interconnect material is deduced. The material should expand less than 0.05 % on reduction (pO_2)~10^{-16}) to ensure stack integrity.

The above-described problem is in fact a very general one and may be a serious limiting factor for use of a number of non-stoichiometric oxides. The second paper (Hendriksen et al., 1999) addresses the problem of chemically induced strains and stresses in tubes of two other non-stoichiometric oxides : $(La,Sr)(FeCo)O_3$ and $SrFeCo_{0.5}O_x$, which are candidate materials for oxygen permeable membranes and SOFC cathodes.

Electrodes
The state-of-the-art SOFC electrodes are composites consisting of lanthanum strontium manganate (LSM) and yttria-stabilised zirconia (YSZ) for the cathode and Ni-YSZ for the anode. The electrode processes and the factors limiting performance on these types of electrodes have been discussed in a number of papers. Based on a review of experimental investigations in the literature (Mogensen and Skaarup, 1996) discuss the factors determining performance on both LSM/YSZ composite cathodes and Ni/YSZ-cermet anodes. They conclude that the LSM/YSZ cathode is a TPB-electrode, i.e. only a narrow zone around the TPB take actively part in the process. The route for optimising the cathode performance is thus to introduce as much TPB as possible per unit cell area. For the anode process it is concluded that the rate limiting step is not likely to be dissociative adsorption of H on the Ni nor surface diffusion. The rate limiting process takes place at the TPB or on the adjacent YSZ.

The performance of composite electrodes depends considerably on the particle size distribution of the constituents and the packing of the particles. Simulations of the polarisation resistance, the conductivity and the impedance of composite electrodes have been performed using a model representing the electrode by a three-dimensional random-resistor network. The results have been published in three papers by S. Sunde in 1996. The work was focussed on Ni-YSZ cermet anodes and the results are in reasonable agreement with experimental findings. For instance the model can explain how growth of Ni particles during operation of Ni-YSZ cermet anodes may lead to a decrease in performance due to changes in the percolation threshold which affects the electrode conductivity. Further, it was found that clusters of electrode material enclosed in a matrix of electrolyte material may give rise to high frequency "distortion" of the impedance.

Modelling of mechanical problems.
There is a number of mechanical loads to be considered when addressing the problem of ensuring mechanical integrity of cells and stacks. Cell/stack-failure may occur due to:
1) Stresses due to TEC-mismatches.
2) Stresses introduced on handling and mounting.
3) Stresses due to chemically induced strain (lattice expansion of interconnect)
4) Stresses due to non-homogenous temperature distribution under operation.

The cells must posses sufficient strength at room temperature to allow handling and mounting. The bending strength of YSZ electrolytes coated with Ni/YSZ-anodes and sintered at three different temperatures was studied by Sørensen and Primdahl (1998). It was found that the components sintered at the lowest temperature had the highest bending strength. From fracture mechanical modelling it was suggested that the loss in strength with increasing sintering temperature is due to development of cracks in the coating (caused by TEC-mismatches) and an increase in interface fracture energy with increasing sintering temperature. Due to the increased fracture energy of the interface in the components sintered at 1300 ^{o}C and 1500 ^{o}C, cracks in the coating penetrate into the structural backbone (the YSZ) forming sharp crack tips. These act as defects decreasing the bending strength of the component.

Treatments of consequences of chemically induced stresses and non-homogenous temperature distribution under operation have been summarised above.

Materials development, electrolytes and interconnects

(Mogens Mogensen)

Electrolytes

During the period studies were performed on stabilised zirconia and on various perovskite structured oxides. One type of studies is zirconia co-doped with Sc_2O_3 and Y_2O_3. This has a considerably higher (1.5 – 2 times) conductivity than the normally used TZ8Y according reports in the open literature. Such zirconia was produced by Viking Chemicals, and the high oxide ion conductivity was confirmed through conductivity measurements at Risø (N. Bonanos, unpublished results). Other investigations were about solubility and effect of the impurities, SiO_2, MnO_x and Fe_2O_3. It was found that SiO_2 has little if any detrimental effect in otherwise pure YSZ. If MnO_x is also dissolved in the YSZ then a glassy phase is formed and spread as a film along the YSZ grain boundaries. This decreases the ionic conductivity of the materials considerably (Appel & Bonanos, 1999). A study of solubility of Fe_2O_3 in zirconia at low temperature showed that by high intensity ball milling at room temperature, a solubility limit of 18.5 mol% was found (Jiang, Poulsen & Mørup, 1999). The solubility of Fe_2O_3 in zirconia is probably much lower at SOFC operating temperature, but the value has not been determined yet. NiO may react (dissolve) in YSZ during the initial sintering of the anode in air. The solubility and stabilising effect of Ni^{+2} dopant was addressed in a number of studies , also direct formation of Y/Ni-doped zirconia was accomplished by plasma techniques, see (Linderoth & Kusjukevics, 1997), (Kusjukevics et al., 1997) and (Kusjukevics & Linderoth, 1997).

An investigation of the effect of B-ion in perovskites (ABO_3, where A is a big and B is a small metal ion) of the type $La_{0.9}Sr_{0.1}B_{0.9}Mg_{0.1}O_3$ were made for B = Al^{3+}, Ga^{3+}, Sc^{3+} and In^{3+}. The results showed that Ga^{3+} gives by far the best oxide ion conductivity. By comparing the results with literature data and by analysing the results in terms of ionic radii it was revealed that the oxide ion conductivity in oxides with perovskite as well as with fluorite structure is mainly determined by how good the dopant ions match the host structure. In other words, the oxide ion mobility is maximal in a stress free lattice (Larsen, Poulsen & Mogensen, 1998) and (Lybye, & Mogensen, 1998) and (Lybye, Poulsen & Mogensen, 2000)

One of these perovskites, $La_{0.9}Sr_{0.1}Sc_{0.9}Mg_{0.1}O_3$, proved to be a proton conductor in moist atmospheres with low oxygen partial pressures below 800°C. The proton conductivity at 800°C was found to ca. 0.9 mS/cm in an atmosphere with pH_2O about 0.03atm. The parallel oxide ion conductivity was ca. 0.4 mS/cm at these conditions. At high pO_2 p-type electron conductivity is predominant in this oxide (Lybye & Bonanos, 1999).

Another proton conducting oxide with perovskite structure is yttria doped strontium cerate. The influence of A-site stoichiometry on the sinter ability was studied on $Sr_sCe_{0.95}Y_{0.05}O_{3-\delta}$. The maximum density of 98% of theoretical was found for sample with s = 0.995. Also this material showed p-type electronic conductivity at high pO_2. The ionic conductivity in moist hydrogen at 800°C was in the range of 5- 10 mS/cm for optimal doped samples (Phillips et al., 1999).

Interconnects

Properties of multiple-doped lanthanum chromites have been examined as potential candidates as interconnects in SOFC stacks. The stability as a function of pO_2, the sinter ability, the conductivity and the oxygen diffusivity were studied within a range of conditions. The oxygen diffusivity was studied by conductivity relaxation. It was shown that vanadium oxide is an efficient sintering aid for the densification of lanthanum chromites. The composition $La_{0.8}Sr_{0.2}Cr_{0.97}V_{0.03}O_3$ is fulfilling most of the demands for an interconnect apart from a too high expansion on reduction at low pO_2 and high temperature. This expansion problem may be solved if the strontium content is slightly decreased and the vanadium content increased. By this a two-phase structure is formed, which is enough dimensional stable to fulfil the interconnector requirements. The main results on ceramic interconnects are given in (Hansen, Mogensen & Jacobsen, 1997), (Larsen, Hendriksen & Mogensen, 1997) and (Larsen, Hendriksen & Mogensen, 1998).

The oxidation of chromium containing metallic interconnects (Plansee-alloy) was studied (Linderoth & Larsen, 1998), and also ferritic steels for lower temperature operation were tested (Linderoth & Larsen, 2000). The possibilities for a successful application of ferritic stainless steel as interconnects at operation temperatures below ca. 850 ^{O}C look promissing.

Theoretical and experimental advances

(Finn W. Poulsen)

Several advances in the interpretation of impedance measurements on electrode reactions have already been mentioned in the paragraphs “Modelling of materials/cell/stack performance, internal reforming and mechanical aspects” , page 12 and “Development of Anodes”, page 10.

The first (and hitherto only) realisation of an *in-situ* diffraction experiment on an SOFC cell under polarisation was performed by synchrotron radiation at Hasylab, Hamburg, and reported in (Poulsen, Garbe et al 1997), (Poulsen, Sörby et al, 1997) and (Sörby et al 1998). Surprisingly, the lattice parameter of the LSM cathode changes instantaneously upon polarisation, indicating that the entire 50-70 μm cathode layer adjusts it oxygen stoichiometry, when the electrode is polarised and a cathodic, respectively anodic reaction proceed.

In a different synchrotron experiment at Brookhaven on the LSM cathode material itself (as loose powder) Norby et al. determined in great detail the temperature and partial pressure dependence of the LSM phases (Andersen et al., 1998).

Cathodes and ceramic interconnects are oxides that can change stoichiometry, when the partial pressure in the surrounding and/or the temperature is changed. For cathodes a rapid exchange is desirable, whereas for interconnects the opposite is the case (one wants to minimise the leakage current between adjacent cells in the stack).
The bulk chemical diffusion coefficient, D_O, and the surface exchange coefficient, k, for oxygen can be determined in the same experiment by conductivity relaxation

experiments: an equilibrated bar of the material is suddenly exposed to an atmosphere with a lower or higher partial pressure of oxygen and the change in conductivity is monitored. An advanced spread sheet program has been developed, from which D and k is obtained (Hansen et al, 1999).

Fluorite-, perovskite- and pyrochlore structure oxides are the archetype oxides of interest for SOFC. Defect chemistry modelling of these structures has advanced due to the "invention" of a full-proof mathematical algorithm, which allows the complete set of defect equations to be solved without using the conventional "Brouwer approximations" (Poulsen 1997; 1998a,b,c; 1999a,b), (Poulsen et al 2000), and (Bonanos & Poulsen 1999). The code(s) have been distributed to research groups in Oslo, Imperial College (UK), Twente (NL), Ekatarinburg and St. Petersburg (Russia) KFA Jülich and ETH (CH).

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