Robust Advanced Materials
for metal SupportEd
Solid oxide fuel cell

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This report corresponds to the final publishable summary report of the FCH-JU RAMSES project (grant agreement no.: 256768).

1 Summary of results and conclusions

1.1 Introduction

Metal supported cells (MSC) are considered as the next generation of Solid Oxide Fuel Cells (SOFCs). They are supported by a metallic support on which the electrochemically active ceramic layers constituting the cell are deposited as thin layers. Since both the electrodes and electrolyte ceramic layers are thin, a step is done towards the cost-efficiency because the amount of the expensive ceramic materials is decreased to minimum. The use of a thin electrolyte (< 10 µm) enables high performances at low operating temperature. Since the anode is thinner it is in addition less sensitive to re-oxidation. The metallic support, which is a good thermal and electrical conductor, helps in increasing the thermal cycling resistance and the current collecting, respectively. Furthermore, the ductile behavior of metal constituting the support of MSCs will provide increased strength to this type of cell compared to electrolyte and anode supported cells. The above advantages in terms of robustness and cost-efficiency lead to numerous developments of this type of SOFC worldwide (1-27). However, improvement of their performances for low temperature operation (700°C and below) is a key point, as well as durability and manufacturing route which has to be compatible with the metal substrate.

1.2 Scientific Approach

The European funded project called RAMSES addresses the development of MSCs for both planar and tubular designs since both present advantages. Indeed planar cells can reach higher power density but cycling resistance is significantly higher for tubular cells. For planar cells, two cell configurations are considered: the anode side option (named anode side metal supported cell, AMSC), and the cathode side option (named cathode side metal supported cell, CMSC), in which the first electrode deposited on the porous metal substrate is the anode or the cathode respectively. Up to now, only the anode side option has been considered by teams working on MSC around the world (1-27). However, the oxidation resistance of the porous metallic substrate differs depending on the proportion of H_2O and H_2 in the anode compartment. At the outlet of the anode side the environment is strongly corrosive (high p_{H2O}/p_{H2} which is more corrosive than air). The CMSC option offers an alternative to this accelerated oxidation due to the relatively homogenous (low p_{O2} gradient) air environment. The problem of Cr poisoning of the cathode by the metal support has to be solved, by using a barrier layer and/or coating of the metal surface. For tubular cells, only the anode side option is considered.

In the project, developments include both the metal substrate and the ceramic active layers of the cell. Regarding the development of the porous metal substrate, the main challenge is to obtain a controlled and optimized microstructure for gas distribution to the electrode as well as for current collection, but also a stable microstructure and a (very) low corrosion rate below 700°C. Regarding the cell development, the best performing cell materials are optimised and customised to the specific MSC manufacturing and functional requirements. The strategy of the project is a piece by piece validation of all cell components, before a progressive integration into complete cells. Targets are set for each component. In the present document, the development of the metallic substrate and coating, anode, electrolyte and cathode are reported as well as the development and test of tubular and planar cells. Finally, the inspection technique developed to evaluate electrolyte quality is presented. More details can be found in (28-33).

1.3 Experiments

Metallic substrates and coatings

Activities at HÖGANÄS have been dedicated to the development of a stainless steel metallic powder appropriate for the manufacturing of the porous metallic substrates. Major attention has been paid to their mechanical and oxidation resistance but also to their compatibility with the different subsequent process steps. The development includes protective coatings for the metallic substrate to enhance its oxidation resistance. The primary purpose has been to work with rather simple alloys that can be produced by cost effective water atomization even though some comparisons with gas atomized powders have been performed. The metal powders were manufactured in several different sieve cuts to investigate the effect of this parameter on the subsequent properties.

Ten different metal powders with variations in the element composition and concentrations as well as in production route were produced and analyzed by HÖGANÄS. A water-atomized ferritic stainless steel powder consisting of Fe + 22% Cr and a Si content <0.15 wt% has been selected according to the criteria of lower cost, better shrinkage properties and adequate oxidation resistance. After drying, the green sheets made by tape casting were pre-sintered in hydrogen for 30 minutes at 1100 and 1150°C giving shrinkage of 2.5 and 5%, respectively. Observed porosity after co-sintering of the porous metal support, electrode and electrolyte is in the range of 30–40%, which is in agreement with the target for MS-SOFC (Figure 1). The shrinkage properties of these materials have been investigated through geometrical measurements of the samples.

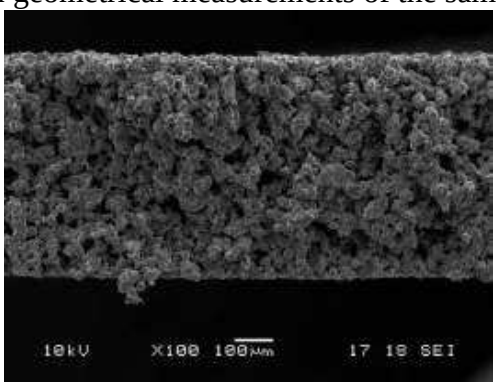


Figure1. Cross section of a pre-sintered (1150°C) porous metal support

Regarding the coating, two options were considered: the first one so-called “pre-coating” consists in the deposition of the coating on the pre-sintered metal support before the deposition of the subsequent ceramic layers of the cell; and a second one so-called “post-coating” where the coating is applied on the metal substrate once the complete cell has been manufactured.

For the “pre-coatings”, the pre-sintered porous metal supports were cleaned before coating in a sequential process using acetone, ethanol and 0.1 M HNO_3 in an ultrasonic bath to get good adhesion of the coating layer to the porous metal. The pre-coating of the porous metal was subsequently performed by infiltration under vacuum. Different solutions were used to create reactive layers coated on the metal support; *e.g.*, the $La(Mn_{0.5}Co_{0.5})_{0.8}$ coating solution was made by dissolving $La(NO_3)_3$, $Mn(NO_3)_2$ and $Co(NO_3)_2$ in isopropanol. The coated samples, after drying in air, were cured at 900°C in air between each coated layer using a vertical preheated tubular furnace. The samples were immersed into the preheated furnace allowing less than 1 minute curing time per

layer. Due to a very short time at 900°C, no significant oxidation of the metal support was observed (see hereafter).
The "post-coating" experiments were done by infiltration of a lanthanum-manganese-cobalt, a cerium or a cerium-manganese solution in isopropanol under vacuum. Various numbers of layers (with drying in air between each layer) and concentration of the solution were tested using cleaned metal supports or fabricated half cells (metal supports/anode/ electrolyte). Excess solution must be avoided since it can penetrate into the anode and partly into the electrolyte. Post sintering of the coated sample was performed in 15% H_2/2 % H_2O/Ar at 700°C since a fast curing at 900°C in air cracks the electrolyte.

Characterization of the coated and uncoated metal supports was studied using FEG-SEM (FEI Nova NanoSEM 650), FEG-SEM-EDS (ZEISS LEO 1530), XRD (Bruker D8 Advance, equipped with 1D LynxEye detector to detect minor phases, θ-2θ scan mode, Cu-Kα X-ray) analysis.
The oxidation behaviour of the metal substrates at operating conditions was studied both for CMSC and AMSC options. In addition to the coated and uncoated metal supports, the oxidation behaviour of a pre-oxidized metal support (cured at 900 °C in air) has been studied. Some coated and pre-oxidized metal supports were annealed in Ar and H_2/Ar at different temperatures in the range of 1100-1350°C in order to see how the coating could tolerate co-sintering conditions for metal support, anode and electrolyte. The oxidation experiments were carried out at 600°C for several hundred hours (up to 500 h) in gas mixtures simulating the most oxidising conditions received on the cathode and anode sides, *i.e.*, air and wet hydrogen (80vol% H_2O). The wet hydrogen atmosphere was simulated by an Ar-2%H_2-8%H_2O mixture. The materials were evaluated mainly from the weight increase and post-mortem characterisations. SEM-EDS was used to determine the nature of the oxide layers formed and the oxide thickness. The porosity after oxidation was also checked. XRD analysis was additionally carried out to confirm the nature of the oxide layers.

<u>Anode</u>

The development of anode (composition and microstructure) was carried out from half symmetrical cells composed of an 8YSZ electrolyte support (120 µm) and symmetrical electrodes screen printed on each side. An additional thin layer (10 µm) of pure NiO was deposited on the active layer in order to collect the current. The half-symmetrical cells were characterized by electrochemical impedance spectroscopy (EIS) in a 2%H_2/Argon atmosphere humidified at 3% at 600°C to estimate the anode ASR. Most of half-symmetrical cells were observed by SEM in order to establish the link between electrochemical performances (ASR) and microstructure. Concerning the anode composition, the effect of an addition of Mg has been evaluated, since it has been shown that it increases the performances of anode for low temperature operation and improves the ageing behavior due to a better microstructure stability (34). Several parameters have been studied at CEA such as anode composition (NiO/8YSZ ratio, Mg addition, …), sintering temperature and procedure (basic sintering or co-sintering).

<u>Electrolyte</u>

The objective of BAIKOWSKI was to develop a specific Yttria Stabilized Zirconia (YSZ) having a sintering behaviour compatible with the assembly of metal support and substrate side electrode. Mainly, the powder has to densify around 1200°C, and parameters like particle size, morphology, crystallinity, etc. have to be adjusted in order to reach this target. A comparison of zirconia powder with 8mol% of Yttria (8YSZ) developed by BAIKOWSKI, named 8YSZ B, with the commercial

Tosoh's powder (referred as 8YSZ T), considered today as a reference in the SOFC manufacturing has been made. The characterization of sintering behavior and ionic conductivity on dense materials was done in comparison to the Tosoh reference powder. Gas-tightness measurements on sintered pellets were also carried out.

Cathode

Cathode materials with improved performance at low temperature have been chosen. The selected compositions were the nickelates $Ln_2NiO_{4+\delta}$ (*Ln* = La, Pr or Nd).Works have been focused on the optimization of the cathode material, and of an appropriate barrier layer to avoid reactivity with the electrolyte. Commercial submicronic powders (Marion Technologies Comp.) were used as starting materials for preparing porous $Ln_2NiO_{4+\delta}$ cathodes. The barrier layer in between the cathode and the electrolyte was $Ce_{0.8}Gd_{0.2}O_{1.90}$ (GDC) from Rhodia Comp.
Constraints due to the MSC design have been considered, such as sintering at lower temperature, as well as stability in low pO_2 sintering atmospheres. Thus, the stability of the cathode materials has been studied as a function of oxygen partial pressure and temperature using thermogravimetry analysis (TGA) measurements and high temperature X-Ray Diffraction (XRD). Several sintering conditions have been investigated, and characterized at the scale of half symmetrical cells through EIS measurements at CNRS.

Complete cells

First tubular cells are considered. Initial work was devoted to select the porous metal substrate, which provides the best manufacturability and performance by using the standard processes and ceramic materials used for the state of the art MSC technology at IKERLAN. Tubular cells of 50 mm length, 14 mm diameter were considered. First, screening of the metal substrates developed by HÖGANÄS in the frame of the RAMSES project has been made in comparison to IKERLAN standard CROFER22APU® tubes. Secondly, they have been validated into tubular cells with standard materials for the other layers, and finally RAMSES electrolyte developed by BAIKOWSKI was implemented. Cells were prepared using the HÖGANÄS metal powder previously selected and by forming porous tubes by gravity methods. A diffusion barrier layer (Yttria Doped Ceria, YDC) was deposited on tubular porous metal substrates by dip coating. A NiO-YSZ (Tosoh) standard anode was subsequently deposited by dip coating and then the BAIKOWSKI electrolyte layer was applied (by powder spray). The half-cell was co-sintered in 10%H_2-Ar between 1350 and 1370ºC. After co-sintering, the reference cathode layer (LSF-SDC composite) was applied by dip coating and fired *in situ* before electrochemical test at 950ºC prior to testing. Their electrochemical performances have been evaluated through i-V curves from 800°C (for a purpose of comparison with IKERLAN standard tubular cells operated at this temperature) down to 600°C in H_2/3%H_2O. A durability test has been carried out at 700°C for 500h under galvanostatic control with a current corresponding to 0.7 V. In addition, the tolerance of the cell towards thermal cycling has been carried out, with 500 thermal cycles. A heating and cooling rate of 10 °C min^{-1} was applied. Due to the inertia of the furnace, this rate was reached during cooling only down to 400°C. Below 400°C, the cooling rate decreased down to only less than 1°C/min. In order to be able to perform 500 cycles in a reasonable testing time, it was then decided to make the thermal cycles between 700°C and 80°C and not to room temperature. It will not affect too much the result, since the effect of cycling is more severe for temperatures above 100°C.
Upscaling activities have then been carried, with cell having a length up to 11 cm and a diameter of 14 mm. In spite of the promising electrochemical results obtained with 8YSZ from BAIKOWSKI,

its surface quality presented repeatability issues associated to the use of free powder metal supports. Presintered metal supports manufactured by HÖGANÄS were used instead of free powder tubes in order to reduce defects on electrolyte, due to their better superficial roughness and homogeneity. Although significant improvements in electrolyte quality were obtained with HÖGANÄS tubes, surface defects were not completely removed, observing a better electrolyte appearance with the use of 8YSZ from Tosoh. Electrochemical performances of these cells were measured through i-V curves as described previously. Thermal cycling test of a cell manufactured with HÖGANÄS support and BAIKOWSKI 8YSZ electrolyte is still in progress despite the end of the project thanks to its good behaviour, with 356 cycles done so far

Secondly works regarding planar cells have been carried out. Metal supported SOFC, with planar design, were produced by a multilayer approach comprising metal and metal/ceramic composite layers with different compositions.
NiO-YSZ/CeO_2/steel half cells were prepared by water-based tape casting technology. Vinylic- and Acrylic-base emulsions were used as binders to prepare the slurries for tape casting for steel and ceramic layers, respectively.
The anode was based on Aluminum-doped-NiO and 8mol%Y_2O_3 stabilized zirconia (8YSZ) in a NiO/YSZ=1 ratio. The electrolyte was based on 8YSZ with the addition of 2% Iron as sintering aid. A mixed $La_{0.4}Ce_{0.6}O_2$ (LDC)/steel intermediate layer was placed between the anode and the metal support in order to provide a good adhesion at the interface between the two layers.
Metal support, intermediate layer and anode were prepared by sequential tape casting of water based suspensions and presintered at 1100°C under 10%H_2/Ar atmosphere. The electrolyte was applied by screen printing and the half-cell was sintered at 1320°C under the same protective atmosphere. A ceramic tile was used as load on the laminate in order to keep it flat during sintering.
The cathode, consisting of $La_{0.6}$,$Sr_{0.4}CoO_{3-\delta}$ (LSC40) was then applied by screen printing and sintered in situ at 850°C during the electrochemical test. The electrochemical characterization, comprising i-V curves and Electrochemical Impedance Spectroscopy was performed on a 35 mm cell with active area 3.14 cm². Gold and Nickel meshes were used to collect the current at the cathode and the anode, respectively.

Inspection techniques

A preliminary study stated and classified main kind of defects that can occur during half cells manufacturing process and affect cell performances. Most critical issue directly related to the performances of the cell is of the “quality” of the electrolyte (microstructure, thickness, absence of cracks and ionic conductivity) and in particular its gas tightness. Electrolyte gas tightness can be directly measured with a permeation test, putting the half cell in the middle of a sealed 2 chambers test setup and measuring the gas (O_2) flowing through the cell from a chamber to the other whit a gas sampler. This kind of test presents many difficulties when implemented in a fully automated system in production lines, as it requires a robotized movement of the cell and its positioning in a sealed pressure chamber. This procedure would be too complex to be implemented in an automatic line with few seconds of cycle time and moreover there is the risk to damage the cell.
Non destructive indirect methods to measure electrolyte permeability have been then considered and evaluated by AEA, inspired by test methods performed by operators in SOFC production line, which performs penetrant fluid test and visual inspection of half cells on 100% of production. In the frame of the project, a prototype of a test bench has been designed and constructed to be able to automatically execute the penetrant fluid test performed by operator in production line and cell dimensional check.

1.4. Results

Metallic Substrates and Coatings

Concerning the "pre-coatings" developments, different parameters such as *e.g.*, coating composition, number of coating layers, heat treatment between each layer, and heat treatment procedure affect the quality of the coating. The importance of the different parameters has been analyzed. It was experienced that a high quantity of Mn forms a thick protective spinel $(Mn,Cr)_3O_4$ layer with low conductivity. The La:Mn ratio was therefore varied between 0.7 – 1.0. The best result was obtained with La:Mn = 0.8. Adding more Mn gave a less homogeneous coating as observed by SEM. To avoid cracking of the coating during curing and the co-sintering steps needed for cell fabrication, investigations were carried out to match the TEC of the coating with the TEC of the alloy. The TEC of the alloy is ~$11–12\cdot10^{-6}$ K^{-1} (measured and calculated in the temperature interval 25–500°C) while TEC of $LaMnO_3$ is ~$11\cdot10^{-6}$ K^{-1} [35]. By substitution of Mn with Co in $LaMnO_3$ the TEC of the coating increases (TEC $LaCoO_3$ is ~$22–23\cdot10^{-6}$ K^{-1}). The most crack-free sample was $La(Mn_{0.5}Co_{0.5})_{0.8}$ coated up to 5 times, with subsequent fast curing at 900°C in air between each coating. It was experienced that the degree of cracking increased with number of coated layers. The coating did not reduce the conductivity of the metal supports (resistivity measurements using a multimeter).

The oxidation resistance tests at 600°C showed that the coating was very efficient to improve the oxidation resistance in wet hydrogen (AMSC option) for the pre-sintered metal support, as can be seen in Figure 2 where weight increase for coated and uncoated metal supports after 500 h in wet hydrogen (Ar-2%H_2-8%H_2O) has been compared. The oxidation resistance in air (CMSC option) was also improved by the coating but is still satisfactory without coating. The project criterion for the stability of the metal support (*i.e.* oxide thickness <3 µm after 500 h at 600°C) is fulfilled for uncoated and of course coated substrate in CMSC operating conditions and for coated substrate in AMSC operating conditions.

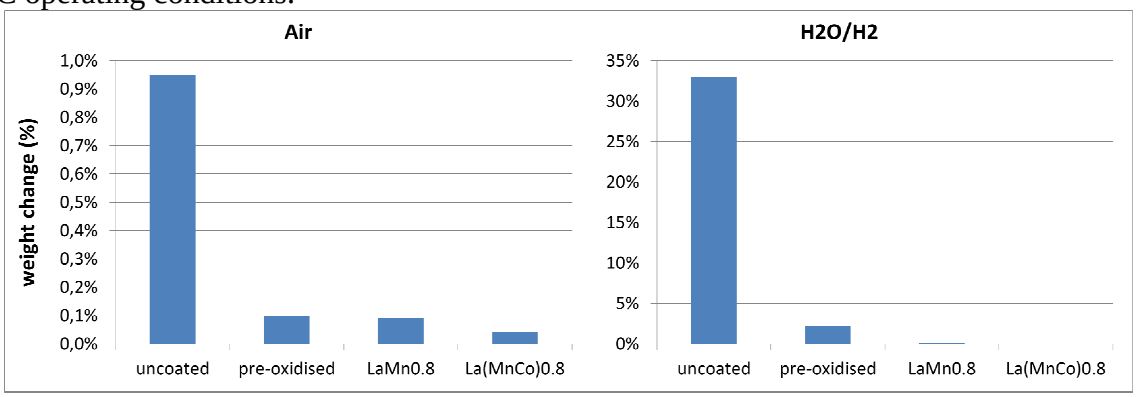


Figure 2. Weight increase of uncoated and coated substrates after oxidation at 600°C for 500h in air (left) and in Ar-2%H_2-8%H_2O (right). No visible plot in some cases means that the weight change measured is zero (below the detection limit).

However, it appeared that a subsequent heat treatment, similar as the one applied to sinter the ceramic layers that constitute the cell, decrease the efficiency of the coatings, the weight change increasing, up to 0.4% after 500h at 600°C in air for example. In addition, a significant drawback with both the coated and pre-oxidized samples is that nearly no shrinkage (below 1%) is obtained in the subsequent sintering steps. The low shrinkage is probably caused by the rather high amount of

coating present in the necks between the grains. This means that it will be impossible to co-sinter the coated or pre-oxidized metal substrate after deposition of the anode and electrolyte, since the electrolyte needs the metal support shrinkage to enable densification. However, the metal support needs the coating if there shall be any chance to fulfil the life time goals for the fuel cell.
This conclusion makes the "post-coating" option particularly relevant.

Concerning the "post-coatings" developments, metal supported half cells were coated with a cerium/cerium-manganese solution and subsequent annealing at 700 °C in 15% H_2/2% H_2O in Ar. The number of coating layers and the viscosity of the solution were varied in order to try to optimize the post coating procedure. The presence of CeO_2 (cubic) on the Ce-coated substrate was confirmed by XRD. The amount of CeO_2 present was however close to the detection limit of the Lynx Eye detector which shows that the layer is very thin. No CeO_2 was observed for the CeMn coated metal support, indicating that Cr_2O_3 formation is faster with this coating.

Oxidation testing of a cerium post-coated porous support gave less than 0.2% weight increase after 500 h of oxidation in air at 600°C. In wet hydrogen, Ce and CeMn led to a significant reduction of oxidation, 0% and 2% weight increase respectively, after preliminary testing for 100 h. From the evolution of weight increase observed for LaMn and LaMnCo coatings between 100 h and 500 h, the Ce post-coating is expected to still show a protective behaviour after 500 h.

Anode

The interest of Mg addition to reduce the anode ASR was shown as well as the positive effect of the co-sintering, which allowed decreasing the sintering temperature by 100°C. An ASR value of 0.37 Ω cm² is obtained at 600°C for a co-sintering at 1300°C, which almost fits the target of 0.3 Ω cm² fixed in the project. First implementation of this Mg-doped anode into tubular MSC cells seems promising, with a decrease of the cell ASR compared to the reference cell with conventional Ni-YSZ anode.
Nevertheless, in MSC configuration, the anode material, whatever the exact cermet formulation, is reduced by the low pO_2 due to the metal substrate once deposited on a green or a pre-sintered metal substrate and co-sintered. Even with a chemical barrier layer, the anode was found reduced. However, the barrier layer was efficient to prevent cationic diffusion between the anode and the metal substrate up to 1300°C. For the time being, this anode reduction in tubular AMSC is not so much detrimental when operating the cell somewhere in the range of 600-700°C, which allows obtaining satisfying power densities.

Electrolyte

The powder made by BAIKOWSKI presents a higher reactivity to sintering than the commercial Tosoh's one. Indeed, it presents a lower sintering temperature (-100°C), which is particularly interesting for the manufacturing of MSC which requires lower sintering temperature to protect the metallic substrate (Figure 1a). This result is observed in both oxidizing and reducing sintering conditions. It was checked that the sinterability in Ar/H_2 was as good as in air (see Figure 3a), as for Tosoh powder. The ionic conductivity is equivalent to the Tosoh 8YSZ in both sintering conditions (Figure 3b).
In addition, a higher density after sintering was observed, which could be explained by a higher green density, which also leads to a higher reactivity during the sintering. Finally, a similar gas-tightness was measured according to permeation tests. Thus, by using the BAIKOWSKI's 8YSZ, a

higher density is expected in the range of sintering temperature required for MSCs, around 1200-1370°C.

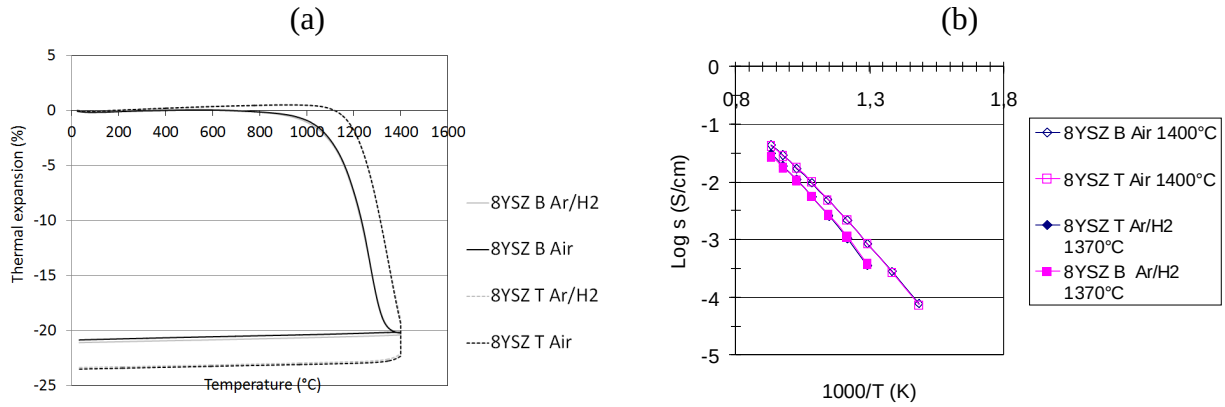


Figure 3. Comparison of BAIKOWSKI (8YSZ B) and Tosoh (8YSZ T) 8YSZ powders in terms of thermal expansion (a) and ionic conductivity (b) versus temperature in air and in Ar/H_2

Cathode

Regarding cathode development, an extensive study has been performed on the chemical stability of the cathode materials upon low pressure conditions, as those requested to process the MSC cells. This study showed that under low oxygen partial pressure, $Pr_2NiO_{4+\delta}$ and $La_2NiO_{4+\delta}$ exhibit a better chemical stability than $Nd_2NiO_{4+\delta}$. For the integration of the nickelates as cathode materials into complete cells, the addition of a diffusion barrier layer was necessary. The cathode diffusion barrier layer selected was Gadolinia Doped Ceria (GDC). Works have been carried out to optimize this layer, especially regarding its sinterability in argon.
The most efficient way to decrease the sintering temperature was to consider the co-sintering of the barrier layer with the cathode. It has been shown that it was possible to reach the polarization resistance target of 0.20 Ω.cm² below 700°C by co-sintering the nickelate with the GDC barrier layer either at 1100°C in argon (Figure 4a) or *in-situ* at 950°C in air (Figure 4b) for both types of nickelates, and even at 600°C for $Pr_2NiO_{4+\delta}$.

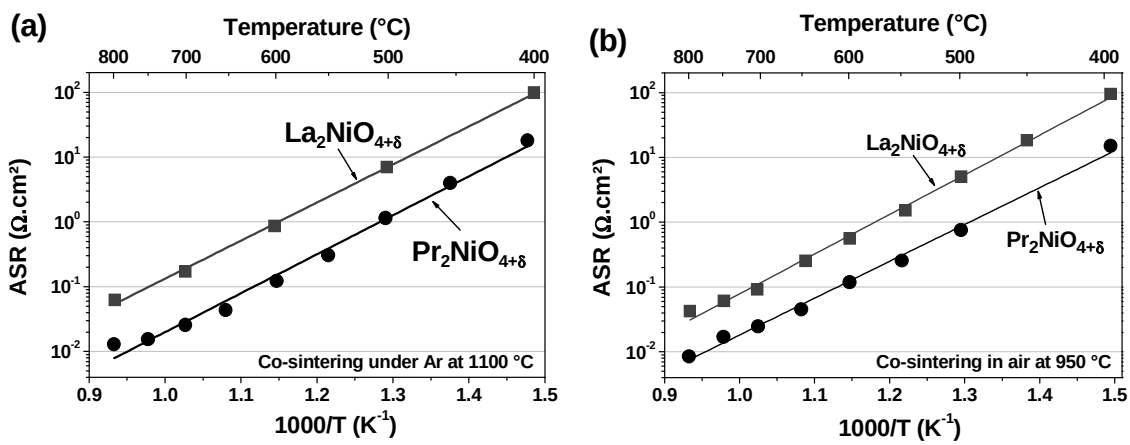


Figure 4. ASR measurements for two types of cathode sintering: a) Co-sintering of the nickelate/GDC on YSZ, at 1100 °C, 1h in Ar; b) *In situ* co-sintering of the nickelate/GDC on YSZ, at 950 °C, for 2 h in air.

Complete cells

Several tubular cells have been tested during the development and implementation of RAMSES materials into IKERLAN tubular MSCs. Figure 5 shows the i-V curves for such tubular cell measured at 600, 700 and 800ºC. An ASR of 1.56 Ω cm^2 was achieved at 600ºC whereas it was 0.40 Ω cm^2 at 700°C, that is to say much less than the target of 0.8 Ω cm^2. This result validates the integration of HÖGANÄS metallic powder as well as BAIKOWSKI electrolyte powder into IKERLAN tubular cells, since classical IKERLAN materials cannot lead to such a performance at low temperature. Two cells of this type have been submitted to a durability test of 500 h. The durability of one of them is shown in Figure 6 over the first 500h, with a stable voltage signal over time, that is to say no major degradation over such duration. The same result was observed on the second cell [28].
Figure 6 also presents the voltage signal over 500 thermal cycles carried out on one of the preceding cells. It can be seen that the voltage signal remains almost unchanged until a total duration of 2900 h, as well as Open Circuit Voltage (OCV~1.11 V) and ASR.

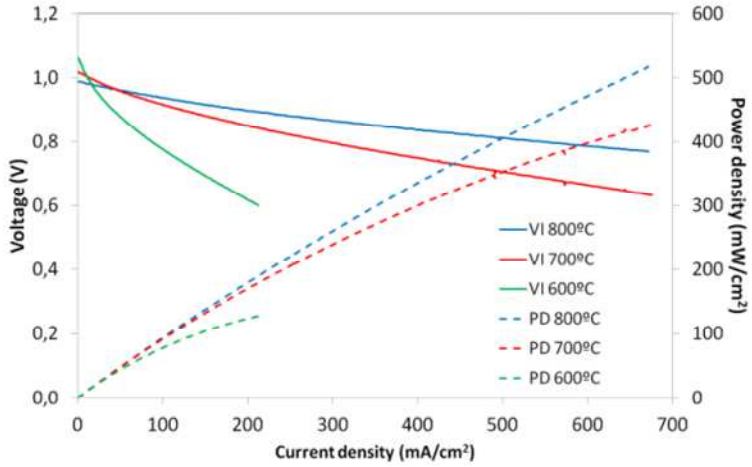


Figure 5. i-V curves at 600, 700 and 800°C of tubular metal supported cells using standard anode and cathode materials and RAMSES metallic support and electrolyte.

Electrochemical measurements were carried out on cells manufactured with HÖGANÄS presintered metal supports in order to compare them with free powder tubes (Figures 5 and 6). Figure 7 shows the i-V curves for cells made with HÖGANÄS tubes, measured at 600, 700 and 800ºC. ASR values between 1-1.2 Ω cm^2 were achieved at 700ºC, which were higher than the 0.40 Ω cm^2 value obtained with the free powder tubes. However, it is important to remark that cells with HÖGANÄS tubes and 8YSZ B exhibited a better surface quality and improved repeatability during sintering. Additionally, OCV was more stable and closer to the theoretical value in the case of HÖGANÄS tubes.

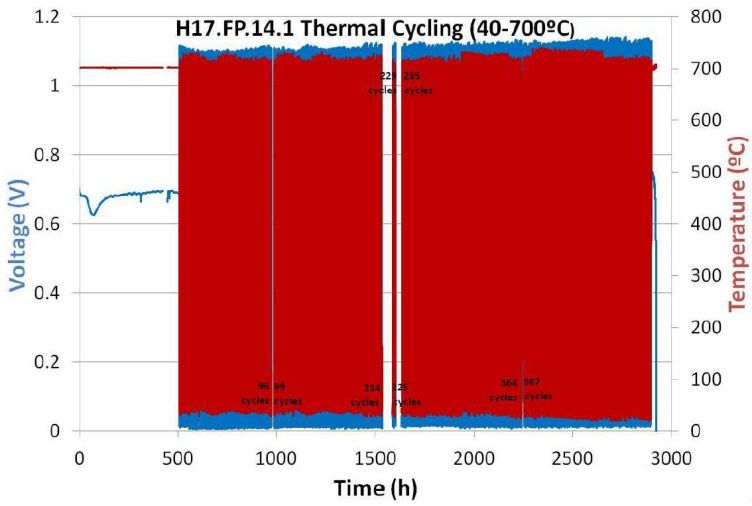


Figure 6. Evolution of voltage and temperature over 500 thermal cycles on a tubular MSC previously submitted to a stationary durability test under current density of 0.22 A.cm^{-2} during 500 h.

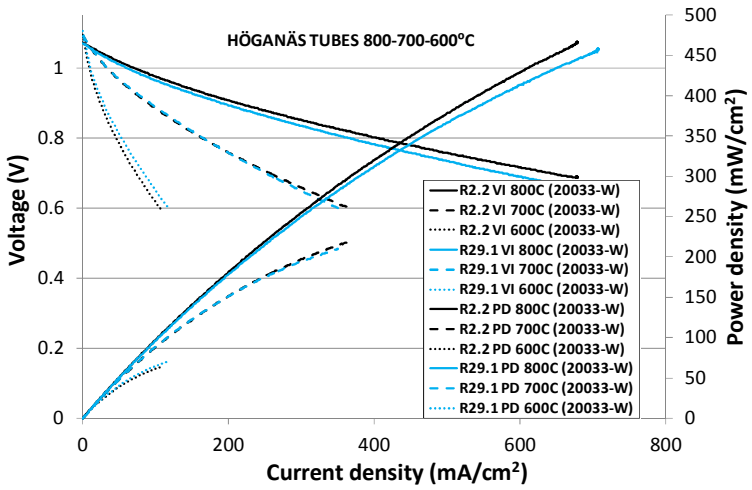


Figure 7. V curves at 600, 700 and 800°C of tubular metal supported cells using standard anode and cathode materials and RAMSES metallic support (manufactured by HÖGANÄS) and electrolyte.

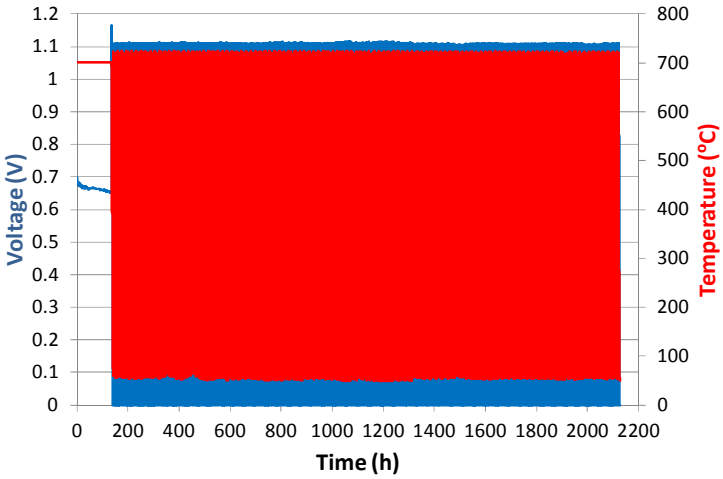


Figure 8. Evolution of voltage and temperature over 356 thermal cycles on a HÖGANÄS tubular MSC previously submitted to a stationary durability test under current during 140 h.

Figure 8 depicts voltage and temperature signal over 356 thermal cycles carried out on one of the aforementioned cells. It can be seen that open circuit voltage (around 1.11 V) remains unaffected. Test is still ongoing despite the end of the project thanks to the good behavior of the cell.

Concerning planar cells, the results of the electrochemical test are given in Fig. 6a. The open circuit voltage observed for this cell was below the expected Nernst potential, suggesting some defects in the electrolyte, which lead to leakage. The current density observed for this cell reached a maximum of 210 mA/cm² at 0.7 V and 800°C. From the EIS analyses the main limitation on the electrochemical performances of the cell was associated to the anode, probably due to extensive Ni coarsening and/or Cr-Ni interdiffusion (Figure 9).

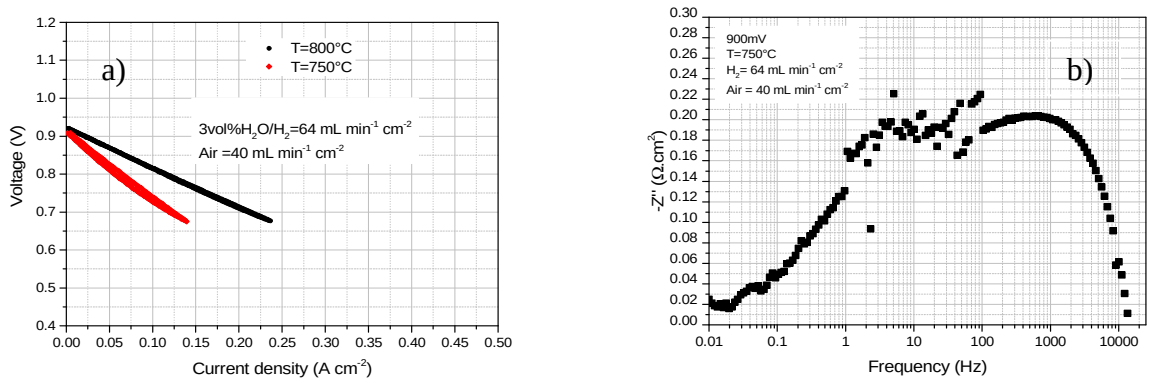


Figure 9. Polarization curves for planar MSC at 800°C and 700°C; (b) Imaginary part of the Bode plot related to the planar MSC at 750°C and at constant voltage 900 mV.

Inspection techniques

The test bench (see Figure 10) is designed in order to accommodate tubular and planar cells of different diameters and sizes. The sample is mounted on a fixture that allows tubular cells to rotate around their axis while the planar cells are rotated around a diameter.
The diameters or sizes are measured using a Mitutoyo optical laser micrometer which is also used to detect the position and the length of the fuel cell. These information are then used for the dimensional test, to centre the cell in front of the camera and to put it under the nozzles of the penetrant liquid and the compressed air used to quickly dry the surface after wetting. Figure 11 (left) shows an example of dimensional check of a tubular cell. Deviations from ideal cylindrical shape are accentuated subtracting the nominal diameter to each diameter actually measured (see Fig. 11, right).
Surface integrity test is performed by means of image analysis. The presence of dark spots due to the penetration of the fluid under the surface is detected by a machine vision system which is composed of a TDI 8192x256 pixels line scan camera and a linear LED illuminator. The camera is connected to the pc through the Camera Link interface and the acquisition is managed by the FPGA processor in the PCIe Camera Link acquisition board. This setup allows capturing of the whole surface in just one image and makes the test very fast (< 10s).
Figure 12 shows the comparison of results of surface analysis algorithm when testing a good cell (Figure 12a) and a cell with defect (Figure 12b), where dark spots due to the penetration of the liquid inside the electrolyte are detected and highlighted with red rectangles.

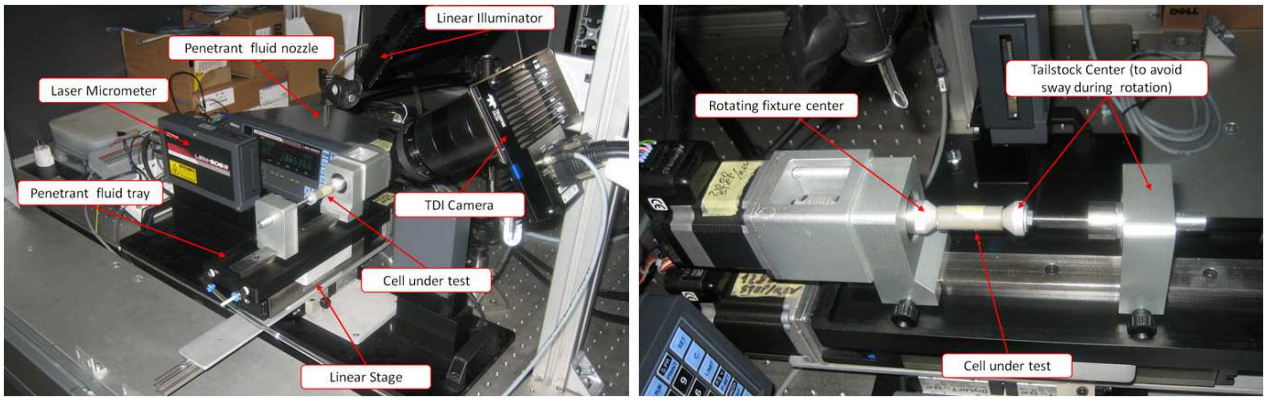
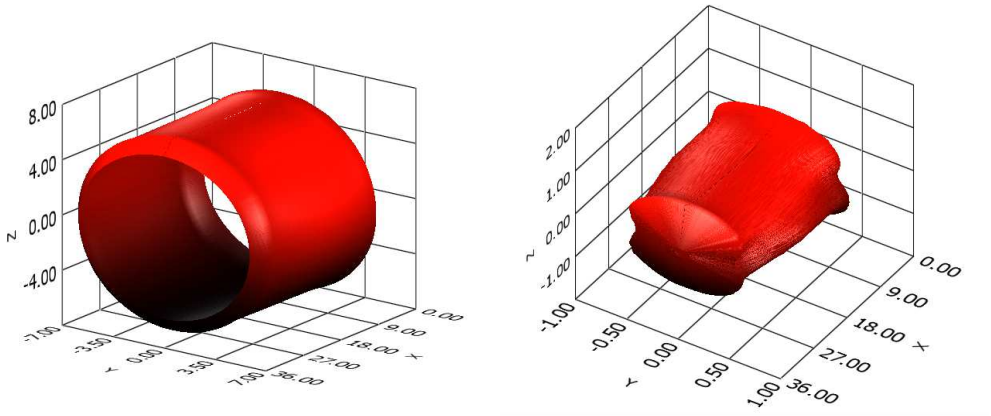


Figure 10. Test bench for tubular half cell dimensions and surface inspection



(a) (b)

Figure 11. Dimensional control of a tubular cell (a) and deviations from ideal cylindrical shape (b)

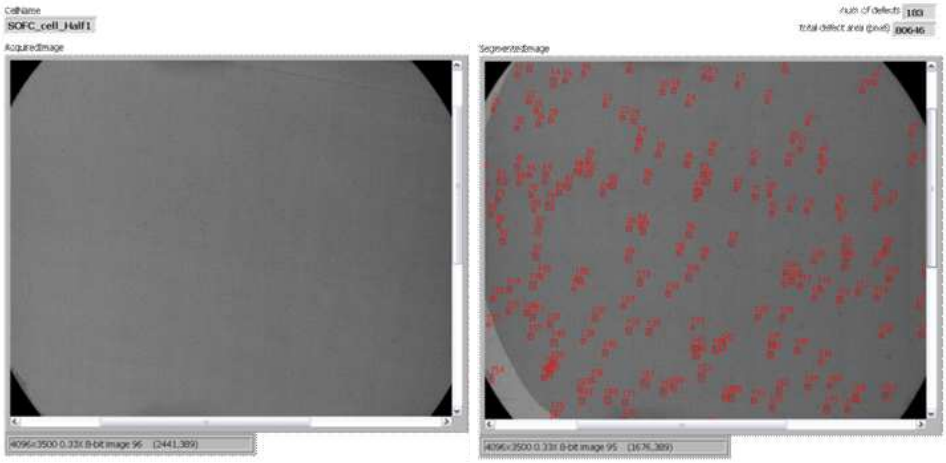


Figure 12. Results of the surface inspection of a good cell (a) and of a cell with several defects (b)

Results obtained with this technique have been compared to measurements performed using a permeation test consisting in the measurement of the oxygen leak rate measurement through the electrolyte in an inert compartment thanks to a mass spectrometer. Figure 13 shows a very good agreement between the inspection technique and the permeation measurements, showing that the inspection technique can also be a kind of quantitative tool.

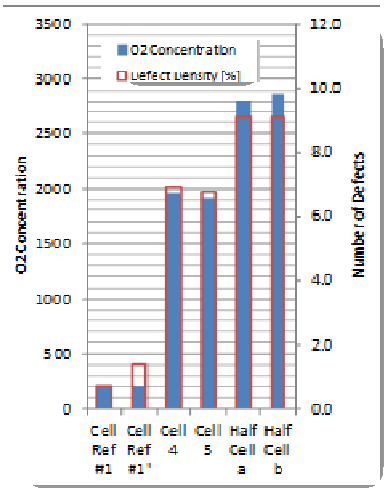


Figure 13. Comparison of the number of defects observed with the inspection technique and the oxygen concentration detected with a permeation test.

1.5. Conclusions

A strategy of piece by piece validation has been used to optimize all metal supported cell components.

A metal substrate suitable for metal supported cell has been developed, able to achieve the targets set in terms of porosity, sinterability and oxidation resistance, being at the same time cost-effective. Both pre-coating on presintered metallic substrates and post-coatings on complete cells have been investigated. Both increase the oxidation resistance of the support, the second option overcoming the issues due to subsequent ceramic layers manufacturing steps.

A customized electrolyte powder has been developed, allowing a decrease of the sintering temperature of 100°C compared to the reference Tosoh 8YSZ powder.

A modified Ni-YSZ anode as well as a nickelate-based cathode were found to reach the performance targets set in the project, with polarization resistances of 0.37 Ω cm² and 0.20 Ω cm² at 600°C in H_2 and air, respectively.

Tubular metal supported cells including RAMSES metallic substrate and electrolyte materials have been successfully tested, leading to an ASR of 1.56 Ω.cm² at 600°C and 0.42 Ω.cm² at 700°C. Durability over 500 h has been demonstrated, and 500 thermal cycles have been successfully applied for a total operation time of almost 3000 h. Upscaled cells have been produced and tested in terms of performance, durability and thermal cycling.

First planar cells have been produced and electrochemically tested.

Finally a non-destructive inspection technique suitable for surface defect detection has been developed, with a setup specifically designed and constructed. This setup allows capturing of the whole surface in just one image and makes the test very fast (< 10s). It was demonstrated to allow an accurate detection of electrolyte defects, with a good correlation with permeation test results.

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3 Potential socio-economic impact and use

Fuel cell applications can contribute significantly to European public policy objectives for energy security, air quality, reduction of greenhouse gas emissions and industrial competitiveness. It is moreover worth noticing that SOFC technology has a strong European dimension with numerous SOFC components being proprietary to European companies and institutions. Within the EU supportive policy framework to stimulate research, development and deployment, **the 3 year RAMSES project worked to produce a robust, highly performing and durable cell for stack manufacturers addressing the CHP application**. As a result it has contributed to improve performance and durability and to lower the cost of tubular metal supported cells targeting this application, which should contribute to the market penetration. In addition it also developed some components (electrolyte, cathode, etc…) with improved performances directly transferrable to other types of cells (anode supported cells for example) already commercially available and considered for CHP applications at short term (2015 – 2020).

3.1. Energy security

As underlined by the European Hydrogen and Fuel Cell Technology Platform [2], today's society depends crucially on the uninterrupted availability of affordable fossil fuels which, in future, will be increasingly concentrated in a smaller number of countries – creating the potential for geopolitical and price instability.
Indeed, unless domestic energy can be more competitive, in the next 20 to 30 years around 70 % of the Union's energy requirements, compared to 50% today, will be met by imported products, some from regions threatened by insecurity. Oil and gas roughly represent half of the current European consumption with the following details [3]:

- Oil: the consumption (which represents 37% of the total EU energy consumption) has increased by 20% since 1994, and global oil demand is projected to grow by 1.6% per year.
- Gas: representing 24% of the EU total consumption, gas imports come from only three countries (Russia, Norway, and Algeria). Furthermore, on current trends, gas imports would increase to 80 % of the total EU consumption over the next 25 years.

Therefore, in order to be able to fulfil the worldwide demand for energy, predicted to grow at a rate of 1.8 % a year for the period 2000 – 2030 [4], a coherent energy strategy is required, addressing both energy supply and demand.

In this context a particular emphasis is also given to energy efficiency. The European Union has developed a set of regulations targeting cogeneration or CHP, in particular, Directive 2004/8/EC of the European Parliament and of the Council of 11 February 2004 on the promotion of cogeneration based on a useful heat demand in the internal energy market and amending Directive 92/42/EEC. In the medium to long term, these directives should serve as a means to create the necessary framework for high efficiency cogeneration, aimed at reducing emissions of CO_2 and other substances, to contribute to sustainable development. SOFC are a privileged technology to achieve such a goal with a total efficiency (electricity + heat) reaching 80%.

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[2] *Hydrogen Energy and Fuel Cells – A vision of our future, 2003*
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However, cost and durability/reliability are the two major impediments to SOFC widespread development and commercialisation: according to the European Hydrogen and Fuel Cell Technology Platform [5] and to the IPHE [6], fuel cell systems for stationary applications require 40 000 hours durability at a cost of less than $750/kW if they want to be competitive with current technologies.

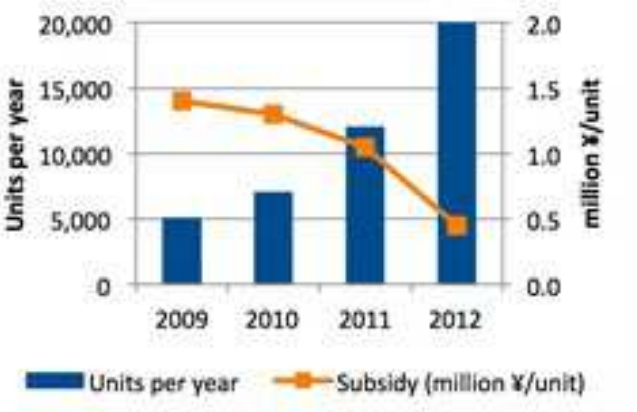
The RAMSES cell is tailored to fulfil the durability requirements, i.e. 40,000 hours for stationary applications and, in addition to withstand a hundred thermal cycles, constituting a competitive component for stack integration in CHP applications. Results achieved in RAMSES on tubular cells, with no degradation after 500h of stationary operation followed by 500 full thermal cycles for a total test duration of 3000h represents a step towards this target, the tolerance to thermal cycles even representing, as far we know, one of the best results worldwide.

The RAMSES cells have demonstrated to be cost-efficient, with tubular cell 37% cheaper than reference tubular cell thanks to improved performance; and planar cell 14% cheaper than reference anode supported cell thanks to the replacement of an expansive ceramic layer by a cheap metal layer, and in addition produced by low cost processes.

3.2. Competitiveness vs. USA & Japan

Driven by the unrelenting rise in petroleum and fossil fuel prices, rising concerns over environmental pollution, and the need for clean energy generation technologies, the world SOFCs market is projected to reach $443 million by the year 2010. [7]

World SOFCs market is dominated by Japan and other environment driven countries in North America, and Western Europe. Japan already started the commercialization of microCHP products (Ene-Farm 700W), with 37000 units installed whose 3000 SOFC, and a growth being confirmed despite the decrrase of incentives (see figure below).



North America spearheads the technology's commercialization, with the region housing a large number of active players and boasting an exhaustive list of products in the pipeline and under final stages of testing and demonstration. As stated by the recent report published by Global Industry Analysts, Inc., the Japanese market is forecast to grow at a CAGR of 8.81% over the period 2011 through 2015, while North America and Europe, together, are expected to post combined revenues to the tune of $393.02 million by the year 2012.

[5] *European Hydrogen and Fuel Cell Technology Platform – Deployment strategy, 2005*
[6] *International Partnership for the Hydrogen Economy, Implementation Scoping Paper, 2006*
[7] *Global Industry Analysts, Inc., Solid_Oxide_Fuel_Cells_SOFCs_Market_Report, 2008*

Longer-term estimations even evaluate the SOFC market to 2.6 Billion$ by 2014 [8]. Being a major player on this field will therefore offer a unique opportunity to aim for the world's largest commodity market, e.g. the electricity market, recently entering a phase of real paradigm change.

In Japan, the last years have seen developments for SOFC government supported activity. For the first time ever, SOFC technology has been included in Japanese funding documents. In addition, the Japanese government has published a highly detailed roadmap with well defined targets for SOFC up to 2025. Although the focus for SOFC remains heavily on research and development activities rather than commercial targets, it is anticipated that the document will strengthen future SOFC development and commercialisation in Japan [9].
At the same time, the Solid State Energy Conversion Alliance (SECA), established in the USA with the aim of bringing together US government, industry and the scientific community in order to promote the develoment of the SOFC technology for use in stationary and transportation applications is very active. Phase I of SECA targets include a minimum of 50000 units and the development of a modular, solid state fuel cell that can be mass produced for different uses by 2010. According to the " Office of Management and Budget" the SECA program has exceeded its 2005 performance targets, and it is on track to meet its goal economically competitive technology by 2010."

At the Europeean level, the creation of the Joint Technology initiative in 2007 has gathered major european stakeholders and allowed the construction of a Europeean technical road map for bridging the gap to SOFC marlet entry with 154-167 M€ to be invested over the period 2008-2013 on stationnary CHP applications [10].
In addition, it is worth noting that Germany is particularly actively promoting the SOFC development and commercialisation. On September 2008, the German Ministry for Transport, Construction and Urban Development (BMVBS), together with nine partners from industry, launched Germany's biggest practical test for fuel cell heating systems for domestic use under the project name Callux. This marks the start of a new era of decentralized power supply in Germany.
About 800 highly efficient heaters are nationwide in basements of private single-and multi-family houses and installed over a period of eight years for their practicality tested. The goal is to fuel heaters in 2015 at the market, a real alternative to conventional devices to create.
Callux comprises a consortium from all over Germany. The National Organization Hydrogen and Fuel Cell Technology (NOW) is the cooperation of heaters manufacturers (Baxi Innotech, Hexis, Vaillant, Viessmann), of energy suppliers (EnBW, E. ON, EWE, MVV, VNG) and of scientific bodies (Center for Solar Energy and Hydrogen Research Stuttgart) who coordinates.
The total volume of the project amounts to 86 M€ with a participation of the BMVBS around 40 M€.

The RAMSES project, because it addresses the development of a third generation SOFC cell tailored for CHP applications is thus fully in line with the Japanese, North American and European road maps[1] including the German orientation. It is moreover complementary with the Callus program by involving other European skills and contributing to strenghten the Europe competitiveness. :

- The enhancement interactions between basic research carried-out by RTD partners and applied development by a SME (SP), including know-how and people transfer;
- The support given to a high-tech SME (SP) to develop medium to long term research activities;

[8] *The Freedonia Group Inc., 2007*
[9] *Fuel Cell Today, "Opening doors to fuel cell commercialisation", January 2007*
[10] *Fuel cells and Hydrogen Joint Undertaking (FCH JU), Multi - Annual Implementation Plan 2008 - 2013*

- The implication of newcomer industry groups in the development of SOFCs such as (HÖGANÄS, BAIKOWSKI, AEA)
- The orientation of top level RTD performers towards market-driven technological objectives indicated by SP and Copreci;
- The formation of new skilled young researchers and engineers, the number of them being insufficient in Europe. Indeed, several post-doctoral fellowships were involved in the project;
- The effective transfer of outstanding R&D capabilities from academic partners (CNRS-BX), via applied R&D centres (CEA, SINTEF, IKL), to partners capable of developing market compatible products and to industrialise them (SP, Copreci);
- The build up of a critical mass of researchers working on a common topic.

It is also worth noting that one third of all the companies involved in the SOFC market are research and development entities and 42% are operating as commercial organisations. This indicates that the SOFC industry is not as advanced in terms of the commercialisation process as the PEMFC sector for instance where 75% of companies describe themselves as being commercial entities. As a consequence, there is still a great deal of development work occurring in the SOFC sector.
The RAMSES project, with its well-balanced consortium addresses that issue as the project allowed close collaboration of leading R&D centres with industrial partners putting a significant effort on R&D activities with the purpose of product commercialisation (see exploitation plans of SP and Copreci.

Commercialisation remains the top priority to be ultimately achieved. The RAMSES cell MSC concept that was developed in the RAMSES project will contribute to overcome the challenges (cost, reliability, performance) essential for full commercialisation.

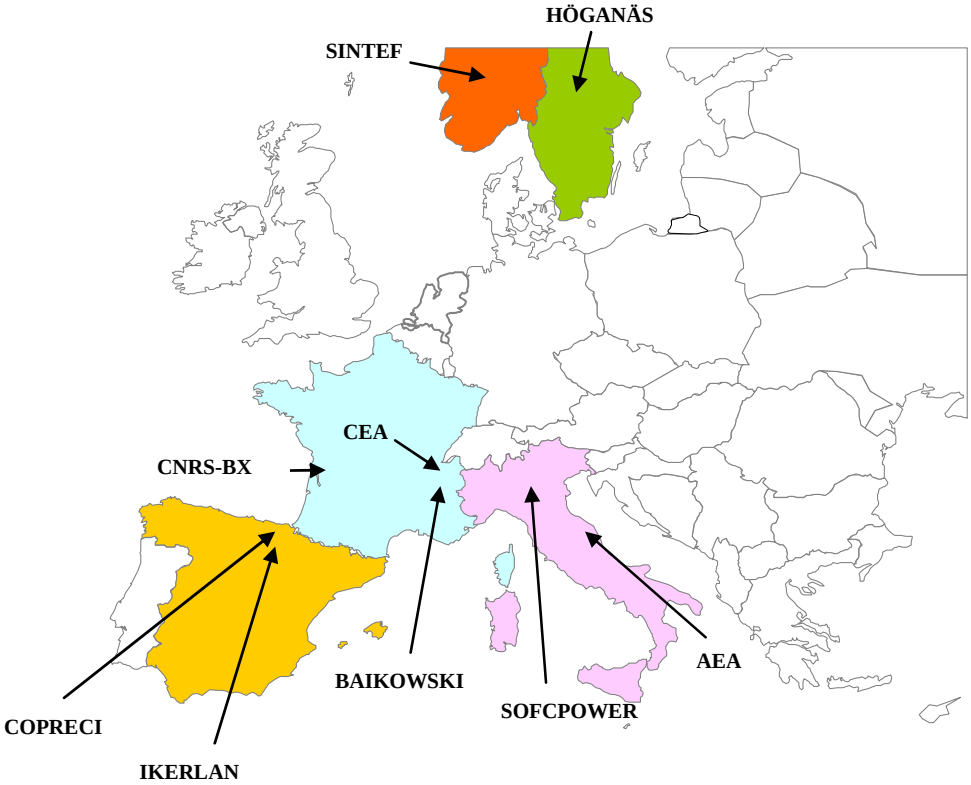
3.3. Contribution to Community environmental objectives

Energy systems of the future must be cleaner in order to offer a potential solution satisfying global energy requirements while reducing carbon dioxide and other greenhouse gas emissions.
A special attention was paid by the RAMSES consortium to the environmental impact of RAMSES cell with its metal support concept. A Life Cycle Analysis has been conducted to evaluate the impacts and environmental performances of this technology, as compared with the ceramic-based technology, and it was found that the impact was expected to be lower. The MSC concept increasing the percentage of metallic compounds within SOFCs, their recyclability is expected to be significantly improved. Additionally a major attention was given to designing manufacturing processes being not too energy consuming and having limited negative environmental impact. For these reasons co-sintering of the half-cell as well as in-situ sintering of the cathode was considered as much as possible to decrease the number and duration of high temperature energy-consuming sintering steps, and water-based “green” slurries were preferentially used for tape-casting.

3.4. The European approach of RAMSES and its contribution to the ERA (European Research Area)

Europe has the skills, resources and potential to become a leading player in the supply and deployment of SOFC technologies. Its diversity in terms of expertise and innovation offers enormous strength provided it can be harnessed and strategically structured.
The RAMSES project contributed to the strengthening of common European activities by bringing together unique European expertise. As shown in Figure below, the project gathered 9 multi-disciplinary organisations coming from 4 different member states (France, Italy, Spain and Sweden) and one associated country (Norway). This European consortium has been built in order to reach the

right critical mass capable of addressing the ambitious objectives presented in this proposal. One IPHE country (Canada) was part of the consortium since their experience in the field of MSCs was high.



4 List of the beneficiaries & Contacts

Partner number	Partner Name (Short name) / Contact	Industry/SME or Research	Country
1(coordinator)	**Commissariat à l'Energie Atomique et aux Energies Alternatives (CEA)**	Research	France
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2	**SOFCpower S.p.a. (SP)**	SME	Italy
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More details of the project can be found in the project website:
http://www.ramses-project.org/

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