

Effect of sintering procedures in development of LiCoO_2 -cathodes for the molten carbonate fuel cell

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Abstract

LiCoO_2 -powder was synthesized from carbonate precursors by calcination in air. Greentapes were tape-cast using a non-aqueous slurry and $10\ \mu\text{m}$ plastic spheres as pore formers. Sintering was carried out in air at $850\text{--}950^\circ\text{C}$ and in argon/air at $500/750^\circ\text{C}$. The two sintering procedures led to very different sub-micron morphologies, with the primary particles being much smaller in the latter case. The electrochemical performance at 650°C , in terms of overpotential at $160\ \text{mA}/\text{cm}^2$, for the air- and argon/air-sintered electrodes was 57 and 81 mV, respectively. The potential drop due to contact resistance between electrode and current collector was estimated to be 100 and 70 mV, respectively. The electrode materials were characterized by scanning electron microscopy (SEM), Hg-porosimetry, the BET-method (N_2 -adsorption), X-ray diffractometry (XRD), flame atomic absorption spectrometry (F-AAS), carbon analysis and a van der Pauw conductivity measurement set-up. © 2000 Elsevier Science S.A. All rights reserved.

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1. Introduction

The molten carbonate fuel cell (MCFC) has a great potential for high efficiency conversion of chemical energy to electrical energy. One of the main technical obstacles for commercialization is the slow dissolution of the NiO-cathode and the nickel precipitation within the matrix causing short-circuiting of the cell. In recent years, a large effort has therefore been directed towards the development of alternative cathode materials. One of the most promising materials seems to be LiCoO_2 [1]. LiCoO_2 has a lower, still acceptable electrical conductivity (about $1\ \text{S}/\text{cm}$) and a lower dissolution rate than NiO. Furthermore, the precipitation mechanism of dissolved Co is different; therefore Co is not so easily short-circuiting the cell. The major work on LiCoO_2 as cathode material for the MCFC has been performed by researchers at ECN and the Technical university of Delft in the Netherlands [1–5], and to some extent also by groups in Sweden, Germany, Italy, Japan, and the US [6–12]. It has been shown that the

pore size distribution has a large influence on performance of the MCFC-cathode [7]. Not only for LiCoO_2 but also for the conventional lithiated NiO, efforts have recently been made to improve the pore structure [13]. The general approach is to increase the amount of solid–liquid–gas interface (where the reactions take place) by decreasing the primary particle size. At the same time, it is important to have large pores ($> 1\ \mu\text{m}$) for gas transport and to maintain a sufficiently high electrical conductivity.

Since 1991, work on LiCoO_2 has been going on at the Royal Institute of Technology, Sweden. Emphasis has mainly been put on how to tailor an optimum pore structure. The development work has been based on LiCoO_2 synthesized from carbonate precursors [14,15]. Recently, also the Pechini method has been used for synthesis of LiCoO_2 [16]. In the course of the LiCoO_2 -cathode development project, it has been found that the electrochemical testing by far is the most expensive, complicated and time-consuming characterization step. Therefore, it is important to determine the materials characteristics before the electrochemical testing in order to be sure that the electrodes have a satisfactory pore size distribution and a sufficiently high electrical conductivity.

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In this paper, electrodes coming from the same greentape but sintered according to different procedures are presented. The objective is to demonstrate the influence of sintering procedure on the pore structure and how this affects the electrochemical performance.

2. Experimental

2.1. Powder synthesis

LiCoO₂-powder was synthesized by calcination of carbonate precursors in air. In order to decrease the particle size, Li₂CO₃ (99%, Johnson & Matthey) was preground for 1 h in a planetary mill, before being mixed with the CoCO₃-powder (Tech. grade, Johnson & Matthey) and ground for another 30 min. The calcination (batch size: 160 g) in air was conducted in two steps: first heating at 1°C/min. to a plateau at 400°C for 5 h. After cooling, the powder was ground in the planetary mill for 30 min and then subjected to a second heating at 2°C/min to 700°C and held for 5 h. The initial composition with respect to Li/Co-ratio was 1.14. In order to clean the calcined LiCoO₂-powder from unreacted Li₂CO₃, it was leached and decanted six times in H₂O. Finally, it was dried in air at about 100°C for 24 h.

2.2. Tape casting

The obtained sub-micron sized LiCoO₂-powder [13] was, after planetary milling for 10 min to reduce the size of the agglomerates, mixed in a non-aqueous slurry and tape-cast. The composition of the slurry is presented in Table 1.

For preparation of the slurry, the LiCoO₂-powder was initially ball-milled with about half its weight of solvent for 3 h. The other slurry components were then added in the order: deflocculant (KD1, ICI Europa, Kortenberg, Belgium), plasticizer (S160, Monsanto Europe, Brussels, Belgium), poreformer (Orgasol 10 μm, Elf Atochem, Paris, France) and binder (BS18, Wecker-Chemie, Munich, Germany). The slurry was allowed to homogenize for 15–60 min by ball-milling before every new addition. The binder was added dissolved in some of the solvent. The slurry was, after appropriate homogenisation by ball-milling for

20 h and de-airing, tape-cast on a plastic carrier film. A batch tape-casting unit (CERLIM, Limoges, France) was used. After drying at room temperature the thickness of the greentape was 0.27 mm.

3. Sintering procedures

The binder burnout and sintering were conducted in a programmable tube furnace with possibility to control the atmosphere (Lindberg, model 54479, 58475P). Circular electrodes were cut out from the greentapes (Ø = 22 mm) and subjected to two different sintering procedures.

(1) Binder burnout and sintering in air at 850–950°C for 6 h. The temperature cycle was a heating rate of 1°C/min up to 600°C with three plateaus for 4 h at 250°C, 500°C and 600°C. From 600°C, the heating rate was 5°C/min. After sintering at 950°C, the electrodes were not completely flat. Before the electrochemical testing, they were therefore flattened by placing them between two alumina plates (weight ~ 15 g) and heat treating them at 900°C for 6 h.

(2) Binder removal and heat treatment in argon, following a heating rate of 1°C/min up to 500°C with a plateau at 250°C for 4 h. After another plateau at 500°C for 7 h in argon, air was introduced and the electrode was heated 1°C/min to 750°C, where it was sintered for 6 h.

To investigate the effect of the argon heat treatment, one electrode was also heat treated according to (2) up to 500°C for 7 h and then cooled in argon without changing to air.

3.1. Electrode characterization

Mass loss and shrinkage of the electrodes were determined by measuring weight and dimensions before and after sintering. The porosity was calculated from the dimensions and weight after sintering by assuming the electrode material to have the density of LiCoO₂ (i.e. 5.05 g/cm³). The pore size distribution was characterized by Hg-porosimetry (Micromeritics, Pore Sizer 9310). The specific surface area was determined by the N₂-adsorption method (BET) (Micromeritics, Flowsorb II 2300). The electrical conductivity at 650°C was measured using a van der Pauw set-up, which is described in Ref. [15]. The conductivity was first determined in air, and later cathode gas (30% CO₂, 70% air) was introduced. The electrode was allowed to stabilize in air and cathode gas for 5 and 20 h, respectively, before the measurement was conducted.

The Li/Co-ratio of the sintered electrodes was determined by dissolving 100 mg of sample in 20 ml concentrated HCl and analyzing Li and Co by flame atomic absorption spectrometry (F-AAS) (Perkin Elmer, 1100 B). The carbon content of the electrodes was determined by the so-called LECO-method, i.e. heating the sample to 2000°C in O₂ and analyzing the released CO₂ (Strölein, CS-mat 600). The electrodes were also investigated by

Table 1
Composition of tape-casting slurry

Function	Material	Trade name	Weight percent
Electrode material	LiCoO ₂	–	47.3
Solvent	Ethanol	–	24.7
Solvent	Toluene	–	11.6
Deflocculant	KD1	KD1	0.7
Plasticizer	D.B.P.	S 160	3.5
Poreformer	Polyamide	Orgasol 10 μm	7.9
Binder	PVB	BS18	4.3

scanning electron microscopy (SEM) (JEOL, JSM-840), equipped with an X-ray emission detector (XES) (Link, AN-10000), and by X-ray powder diffractometry (XRD) (Philips, PW3710, using Cu K_{α} -radiation and a monochromator).

3.2. Electrochemical testing

The air-950°C- and argon/air-sintered electrodes were electrochemically tested in a laboratory fuel cell set-up of approximately 3 cm². The cell and some of its state-of-the-art components (i.e. anode, matrix, and electrolyte) were purchased from ECN in the Netherlands. Fig. 1 is a schematic drawing of the cell.

The cathode current collector was a stainless steel plate, perforated with holes of $\varnothing = 1.5$ mm. The electrolyte used was the eutectic mixture of 62 mol% Li_2CO_3 and 38 mol% K_2CO_3 . An oxygen reference electrode (Au wire, gas: 33/67% O_2/CO_2) was connected to the electrolyte by a salt bridge having the same salt composition as the rest of the cell. Tests were performed with standard gases, i.e. 80/20% H_2/CO_2 humidified at 60°C and 15/30/55% $O_2/CO_2/N_2$, for anode and cathode, respectively.

The electrochemical performance was recorded at 650°C and 700°C after optimization of electrolyte filling (i.e. obtaining optimal performance). Two methods were used for determination of overpotential.

(1) Potential measured directly between a probe on the current collector plate and the reference electrode. The overpotential was obtained by subtracting the open circuit potential from the measured potential at different steady-state current densities. As can be understood from Fig. 1, this measurement includes not only the cathode overpotential, but also the iR -drop due to the contact resistance between the cathode and its current collector and about half the iR -drop of the matrix.

(2) The electrode overpotentials were also obtained by using a current interrupt technique, which eliminates all iR -contributions to the overpotential. The overpotential was recorded 25 μ s after interruption of the current by using a potentiostat with a built-in current interrupt unit (Solartron Interface, SI 1286). It has been shown by

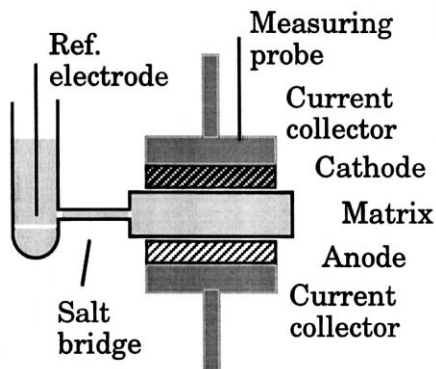


Fig. 1. Schematic drawing of the electrochemical testing cell.

Table 2

Effect of sintering procedure on some basic electrode characteristics

Sintering condition	Mass loss (%)	Shrinkage, $\Delta l/l$ (%)	Porosity (%)	BET (m ² /g)
Air, 850°C	26.3	6.8	58	–
Air, 900°C	26.4	9.3	57	0.92
Air, 950°C	28.0	14.5	53	0.45
Argon, 500°C	26.3	10.8	–	–
Argon/air 500/750°C	26.3	10.5	56	0.68

Lagergren et al. [17] that due to backward currents occurring in the electrode, an underestimation of the overpotential (η) is made when using this method. Therefore, the results have been corrected using the expression:

$$\eta = \eta_{c.i.} + \frac{L}{\sigma + \kappa} j \quad (1)$$

where $\eta_{c.i.}$ is the measured overpotential, L is the electrode thickness (~ 0.24 mm), j is the current density and σ and κ are the effective conductivities of the electrode and the electrolyte, respectively. It was demonstrated that in these systems $\sigma \gg \kappa$ [18], thus κ may be neglected. The electrochemical testing was conducted both at 650°C and 700°C, while the electrical conductivity was measured only at 650°C. σ at 700°C was therefore calculated by multiplying σ at 650°C with a correction factor of 1.22, estimated from the work of Tennakoon et al. [16].

The difference in overpotential obtained by the two methods was used to estimate the contact resistance between the cathode and the current collector.

4. Results and discussion

4.1. Electrode morphology

In Table 2, the effect of sintering procedure on mass loss, shrinkage, porosity, and specific surface area (BET)

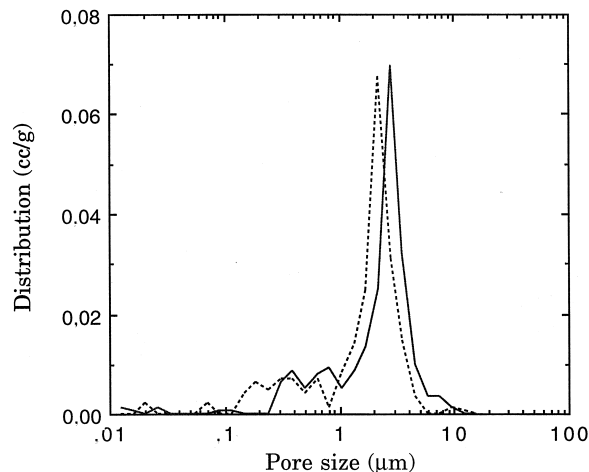


Fig. 2. Pore size distribution of $LiCoO_2$ -electrodes, (–) sintered in air at 950°C, (---) sintered in argon/air at 500/750°C.

are presented. Increasing the sintering temperature in air results in an expected increase of shrinkage, decrease of porosity and specific surface area. Also, the mass loss increases with temperature, indicating that lithium or lithium-compounds are evaporated at higher temperatures (see Section 4.3). The data shows that sintering in argon/air at 500/750°C is more efficient than sintering in air at 900°C. Fig. 2 presents the pore size distribution of the air-950°C, and the argon/air-sintered electrodes, the two electrodes that were chosen for electrochemical testing. The data reveal that the pore size distribution is displaced towards smaller pores in the argon/air-sintered electrode. The smallest pores, representing any significant volume, can for the air-950°C electrode be found at $\sim 0.3 \mu\text{m}$, while for the argon/air electrode they reach down to $0.1 \mu\text{m}$.

The difference in pore structure can also be seen in the SEM-micrographs of Figs. 3 and 4. For the air-950°C electrode (Fig. 3), the primary particle size is in the range $0.5\text{--}5 \mu\text{m}$. For the argon/air electrode (Fig. 4a), the surface of the primary particles is covered with flakes/plates, probably consisting of Li_2CO_3 (see Section 4.3 below). The size of the primary particles is therefore difficult to distinguish. However, when looking at an electrochemically tested (and leached) argon/air electrode (Fig. 4b), the primary particles are in the range $0.2\text{--}0.5 \mu\text{m}$, with a few larger particles incorporated in the electrode. These larger particles, examined by electron microscopy in back-scatter mode and XES-analysis, were found to have the same chemical composition as the smaller ones. We therefore conclude that they are LiCoO_2 -particles.

4.2. The effect of argon

It has been demonstrated that the organic materials of the greentape essentially burn off below 500°C, when sintering in air [7]. Due to the oxidizing conditions, the LiCoO_2 -particles remain unaffected until the temperature is raised above 700°C when sintering begins. When heat-

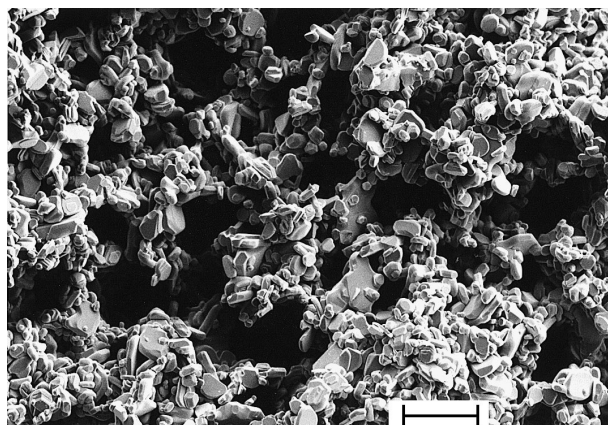


Fig. 3. SEM-micrograph of a LiCoO_2 -electrode sintered in air at 950°C (bar length = $10 \mu\text{m}$).

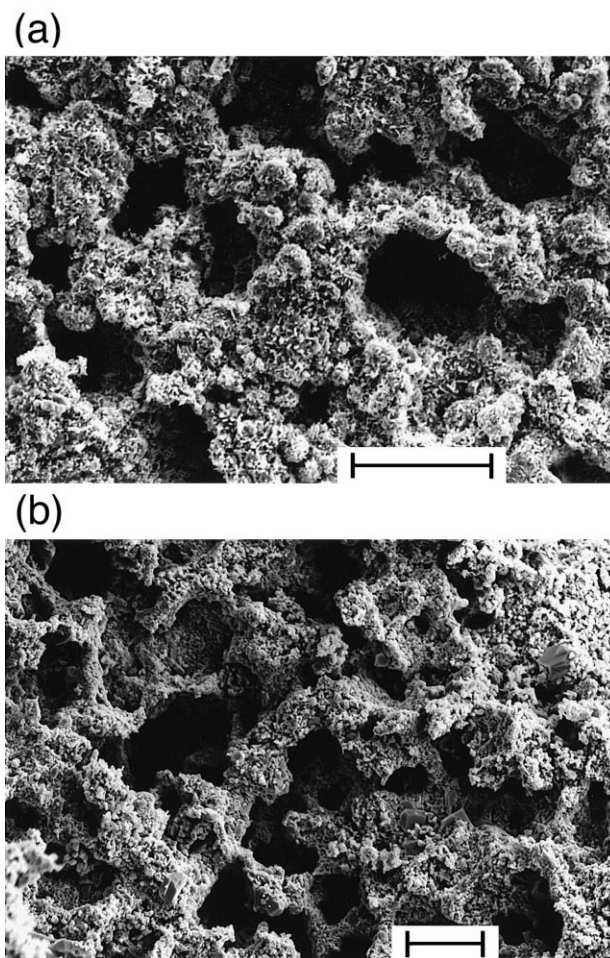


Fig. 4. SEM-micrograph of a LiCoO_2 -electrode sintered in argon/air at 500/750°C (bar length = $10 \mu\text{m}$), (a, top) before electrochemical testing, (b, bottom) after electrochemical testing (and leached from electrolyte).

ing the greentape in argon, it is expected that, due to the organic materials, highly reducing conditions are created inside the tape. Fig. 5 shows a SEM-micrograph of electrode material after heat treatment at 500°C for 7 h and cooling in argon. XRD-analysis on this material revealed

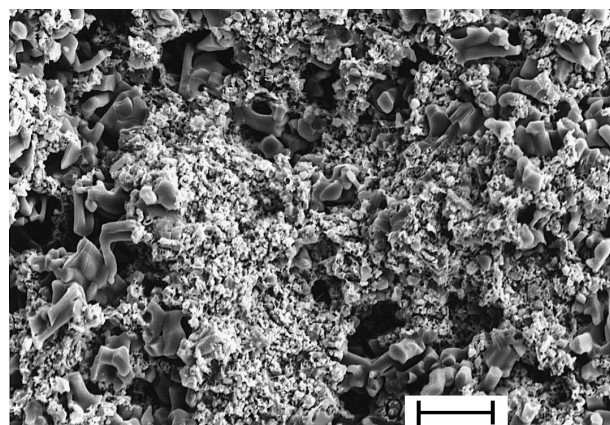


Fig. 5. SEM-micrograph of electrode material after heat treatment at 500°C and cooling in argon (bar length = $10 \mu\text{m}$).

that the LiCoO_2 had been reduced to metallic Co and Li_2CO_3 . From electron microscopy in back-scatter mode and XES-analysis, the large particles of Fig. 5 were identified as Li_2CO_3 and the small particles as Co. When comparing Fig. 5 with Fig. 4 it seems as if large Li_2CO_3 -particles have been formed mainly in the cavities left from the poreformer and that the skeleton of small Co-particles prevents the electrode pore structure from collapsing.

It may thus be concluded that when sintered in argon/air, LiCoO_2 is first reduced in argon at 500°C and that LiCoO_2 is reobtained again when air is introduced at $500\text{--}750^\circ\text{C}$. Looking at Table 2, it also appears as if the shrinkage of the greentape is due to the argon heat treatment rather than due to the formation and sintering of LiCoO_2 in the oxidation step at $500\text{--}750^\circ\text{C}$. This is understandable since 750°C is a very low temperature for sintering of LiCoO_2 .

4.3. Composition and conductivity

From the analysis of carbon content, presented in Table 3, it was found that the argon/air electrode contained more carbon than the air- 950°C electrode (all the air-sintered electrodes contained $0.06\text{--}0.07$ wt.% of C).

This indicates the presence of unreacted Li_2CO_3 in the argon/air electrode. This finding is also supported by the electrical conductivity of the argon/air electrode being significantly higher in air than in cathode gas (30% CO_2 , 70% air) (Table 3). The same results but with a greater difference has been reported with electrodes containing unreacted Li_2CO_3 in larger quantities [7,15]. The phenomenon was explained by that Li_2CO_3 in air partially decomposes to Li_2O that interacts with LiCoO_2 and increases its conductivity. When changing to cathode gas Li_2CO_3 is formed again and consequently the conductivity is lowered.

From the XRD-spectra of Fig. 6, the sintered electrodes were found to mainly consist of LiCoO_2 . In the air- 950°C electrode (Fig. 6a), a small amount of Co_3O_4 was also detected. Although flakes/plates of possible Li_2CO_3 can be seen in the micrograph of Fig. 4a, Li_2CO_3 was not detected in the XRD-spectrum of the argon/air electrode (Fig. 6b). This could be due to the low scattering factor of Li_2CO_3 compared to that of LiCoO_2 . The presence of Co_3O_4 in the air- 950°C electrode indicates that lithium or

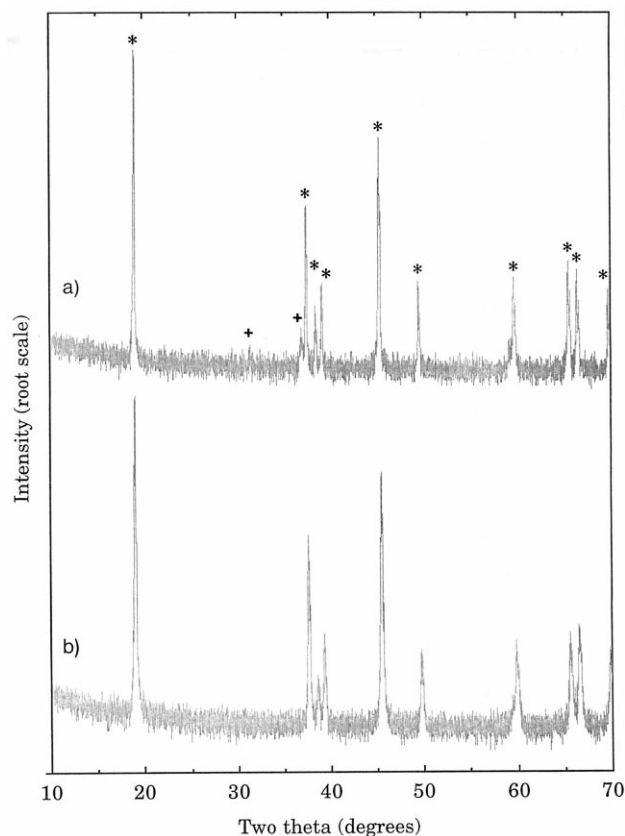


Fig. 6. XRD-spectra of sintered electrodes, (a) sintered in air at 950°C , (b) sintered in argon/air at $500/750^\circ\text{C}$ (* = LiCoO_2 , + = Co_3O_4).

lithium compounds has evaporated during sintering. This finding is supported by the Li/Co-ratio of Table 3, obtained from F-AAS-analysis, being only 0.87 for the air- 950°C electrode. For the argon/air electrode, sintered at a much lower temperature, it was 1.00. F-AAS-analysis was also conducted on the air- 850°C electrode giving a Li/Co-ratio of 0.99. Similar results with respect to lithium evaporation at elevated sintering temperatures have been found by Tennakoon et al. [16].

The electrical conductivity, σ , of both electrodes is presented in Table 3. Fontes et al. [19] have recently calculated that a conductivity at theoretical density (σ_0) of 0.8 S/cm should be sufficient for making high performance MCFC-electrodes. A rough estimate of σ_0 can be obtained from the expression [2]:

$$\sigma_0 = \sigma / (1 - P)^{1.75} \quad (2)$$

where P is the pore fraction. The calculation gives that σ_0 , in cathode gas at 650°C , is 0.9 and 1.3 S/cm for the air- 950°C and argon/air electrodes, respectively. Thus, the electrical conductivity of the electrodes is satisfactory.

4.4. Electrochemical performance and contact resistance

Fig. 7 presents polarisation curves obtained by current interrupt technique of the air- 950°C and argon/air electrodes at 650°C .

Table 3
Chemical composition and electrical conductivity of electrodes

Sintering condition	Carbon content (wt.%)	Li/Co-ratio from F-AAS	Electrical conductivity σ , at 650°C (S/cm)	
			Air	30% CO_2 , 70% air
Air, 950°C	0.06	0.87	0.25	0.24
Argon/air, $500/750^\circ\text{C}$	0.09	1.00	0.43	0.31

The shape of the curves is similar to data from an earlier study of LiCoO_2 -cathodes at optimum electrolyte fill [7]. The figure shows that the air-950°C electrode has the best electrochemical performance. This result is a bit surprising since the argon/air electrode has a significantly smaller primary particle size, which should yield more reaction sites. The explanation may be a higher mass transport resistance of the argon/air electrode in the liquid phase and/or in the gas phase. However, the difference in overpotential at 160 mA/cm^2 is small (Table 4), for 700°C it is less than 10 mV. The performance data of Table 4 obtained by current interrupt technique ($\eta_{\text{c.i.}}$) is competitive with state-of-the-art NiO-cathodes.

However, the overpotentials measured directly between a probe on the current collector and the reference electrode ($\eta_{\text{c.c.r.}}$) are less satisfactory and are higher than the loss of ~ 100 mV (650°C, 160 mA/cm^2) which is projected for the NiO-cathode in a commercial fuel cell [20]. The directly measured overpotential includes not only the electrode overpotential, but also the iR -drop due to ionic resistivity in the matrix and contact resistance between the cathode and the current collector. The difference ($\Delta\eta$) between the two measurement techniques of Table 4 may thus be considered as the iR -contribution to the cathode overpotential. The iR -drop at 160 mA/cm^2 of half the matrix (i.e. 0.5 mm) is ~ 10 mV [21]. At 650°C, this leaves a contact resistance drop of about 100 and 70 mV for the air-950°C and argon/air electrodes, respectively. The corresponding value for lithiated NiO is 25 mV [20]. The contact resistance seems to be rather sensitive to the degree of electrolyte filling, indicating that to some extent the electrical current over the current collector/electrode interface passes through the electrolyte or that the electrical conductivity of the interface depends on the presence

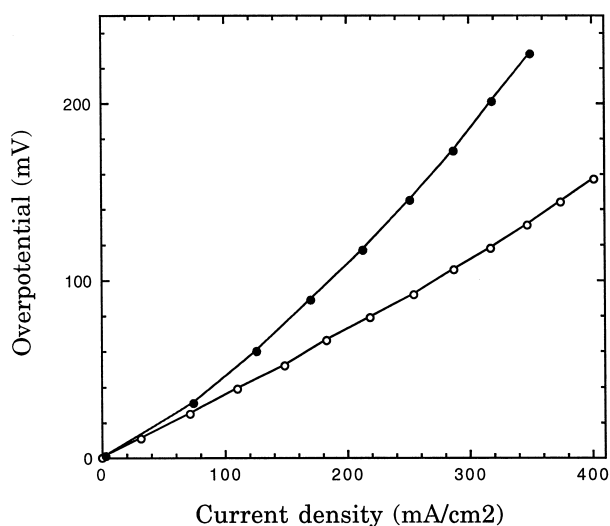


Fig. 7. Cathode polarisation (η) curves at 650°C obtained by current interrupt technique and corrected for underestimation of (—o—) LiCoO_2 -electrode sintered in air at 950°C, (—●—) LiCoO_2 -electrode sintered in argon/air at 500/750°C.

Table 4

Electrochemical overpotentials (η) at 160 mA/cm^2 measured directly between current collector and reference electrode (c.c.-r.) and by current interrupt technique (c.i.)

Sintering condition	η at 650°C (mV)			η at 700°C (mV)		
	$\eta_{\text{c.c.r.}}$	$\eta_{\text{c.i.}}$	$\Delta\eta$	$\eta_{\text{c.c.r.}}$	$\eta_{\text{c.i.}}$	$\Delta\eta$
Air, 950°C	167	57	110	129	41	88
Argon/air, 500/750°C	162	81	81	94	50	44

of electrolyte. Therefore, the difference in decrease of contact resistance, between the air-950°C and argon/air electrodes, when changing from 650°C to 700°C (Table 4), may be explained by different electrolyte filling levels at the measurement occasions (i.e. at optimal performance).

The lower contact resistance of the argon/air electrode may be understood by considering that it is less sintered than the air-950°C electrode. This gives it less mechanical integrity and therefore, a better current collector/electrode contact is probably created. A similar observation has been made by researchers at ECN [21]. The potential drop due to contact resistance is still about three times larger than that of lithiated NiO. This is unacceptable and future efforts should therefore be put into solving this problem.

4.5. The argon / air electrode and its prospects

This study has shown that sintering in argon/air at 500/700°C provides electrodes with much finer primary particles than sintering in air, while still retaining a sufficiently high electrical conductivity. The electrochemical performance, however, is not, as could be expected, better than that of the air-950°C electrode. The reason for this is not clear. Still, considering the pore size distribution, the argon/air sintering procedure seems propitious for making LiCoO_2 -cathodes with a very good electrochemical performance.

Moreover, the fact that the LiCoO_2 -material can be reduced to metallic Co and Li_2CO_3 , and oxidized back without having its pore structure deteriorated may enable in-situ formation of LiCoO_2 . It should be possible to take the reduced electrode, place it in a fuel cell and oxidize it in-situ. The advantage of this procedure is that the handling of the very fragile ceramic LiCoO_2 -material may be avoided. This problem becomes increasingly important as the electrode size increases. The matter should be further investigated.

5. Conclusions

High performance LiCoO_2 -cathodes have been fabricated by using a sub-micron LiCoO_2 -powder and 10 μm plastic sphere poreformers. Starting from the same green-tape, it was found that sintering in argon/air at 500/750°C

resulted in a different sub-micron porestructure, with much finer primary particles than sintering in air at 950°C. Still, sufficient sintering (i.e. shrinkage) and electrical conductivity were obtained in the argon/air electrodes. Sintering in argon/air was found to proceed over the reduction of LiCoO_2 to Li_2CO_3 and metallic cobalt in argon, before reobtaining LiCoO_2 again by changing to air. Both the air- and argon/air-sintered cathodes exhibit electrochemical performances competitive with lithiated NiO, but the high contact resistance of the LiCoO_2 -cathodes still remains to be solved.

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