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The direct carbonate fuel cell technology: advances in multi-fuel processing and internal reforming

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Abstract

Carbonate fuel cells are new generation of power plants that provide electricity at very high efficiency and low environmental emissions. The direct carbonate fuel cell (DFC^{TM}) technology is fast approaching its commercial entry. DFC cell operates on a variety of hydrocarbon fuels such as natural gas, biogas, coal gas, diesel and LPG. Conventionally, an external reformer is used to supply hydrogen-rich gas to fuel cell. FuelCell energy's DFC^{TM} technology has adopted an internal reforming approach that eliminates the need for an external reformer, resulting in increased efficiency, compactness, better thermal management and lower cost. Fuel processing operation in DFC^{TM} technology varies based on the nature of fuel. However, the fuel thus supplied to the fuel cell has a two-step refinement approach involving impurities (sulfur) removal followed by pre-reforming desulfurized fuel to knock out higher hydrocarbons. Methane-rich gas obtained from pre-reformer is thus fed into fuel cell for internal reforming to produce hydrogen in DFC stacks, thereby producing DC power. At each stage of this operation, catalysts are used to facilitate production of desirable fuel cell fuel. FCE has developed stable catalyst system at each one of these steps. FCE's experience with internal reforming (indirect internal reforming and direct internal reforming catalysts) and fuel processing catalysts is discussed here.

Keywords: Carbonate fuel cell; Fuel processing; Internal reforming

1. Introduction

Fuel cells electrochemically convert chemical energy of a fuel directly to electricity, surpassing energy conversion efficiencies of conventional thermal energy conversion devices, while with unmatched low environmental emissions. Hydrogen is the preferred fuel of the present generation fuel cells because of its high electrochemical conversion rate compared to other fossil fuels. Therefore, hydrocarbon fuels need to be reformed to produce hydrogen for fuel cell use. Furthermore, in spite of its high-energy conversion efficiency, approximately 40–50% of the chemical value of hydrogen, depending on fuel cell type and design, still evolves as heat. This byproduct heat needs to be removed efficiently for isothermal operation as well as for system and/or a bottoming cycle use.

Steam reforming of hydrocarbon in an external reformer, operated normally at temperatures of 750–850 °C which is significantly higher than the carbonate fuel cell operating temperature of ~650 °C, is a well-established industrial process for hydrogen production. The steam reforming reaction is highly endothermic. The heat required for the natural gas reforming is approximately 20% of the lower heating value of the product hydrogen. Furthermore, while the product of the reforming reaction (hydrogen) is a reactant in the fuel cell, the fuel cell reaction product

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(water) is a reactant in the reforming reaction. Therefore, the thermal and chemical features of the fuel cell and reforming reactions are uniquely complementary if efficient integration of both reactions can be achieved.

The carbonate fuel cell system operates at high enough temperatures to allow practical reforming reaction kinetics in the presence of internal reforming catalyst for natural gas fuel directly inside the fuel cell [1]. In direct fuel cell (direct carbonate fuel, DFC) concept, the fuel cell and reforming reactions are carried out in close proximity to allow rapid thermal and mass exchanges. The DFC concept eliminates the external reformer and associated heat exchange equipment, resulting in the most simple and efficient power plant system. The single cycle smaller DFC power plants project to a system efficiency of 50–60% on a lower heating value (LHV) basis and the DFC-based combined cycle larger power plants project to >70% (LHV) efficiency operation.

DFC technology is unique in that it can operate directly on various hydrocarbon fuels without the use of an external reformer. However, fuel processing steps such as clean-up and preconversion are still required to produce a methane-rich fuel free from impurities such as sulfur, chlorine, particulates, higher hydrocarbons and others that may effect stability of fuel cell components and internal reforming catalysts. Fuel processing steps required may vary according to the types of hydrocarbon fuel used. In this paper, we present fuel processing options for natural gas and diesel fuel, and internal reforming technology status.

2. Fuel processing requirements for DFC operation

Natural gas and diesel fuel contains impurities that may have negative effects on the internal reforming catalyst performance. The internal reforming catalysts are extremely active and have high selectivity for hydrogen, but they are also very sensitive to certain impurities. To assure long-term stability of DFC plant, it is important to ensure that the process fuel is free from impurities such as sulfur. Tables 1 and 2 represent sulfur levels in the natural gas (mainly odorant) in US distributed system as well as JP-8 diesel fuel. Furthermore, both fuels contain higher hydrocarbons that may promote carbon formation on the internal reforming catalyst. Thus, DFC operation needs to convert all higher hydrocarbons to methane-rich fuel before entering cells. The major fuel processing steps for natural gas and diesel fuel in DFC power plants are presented in Fig. 1. Both natural gas and diesel

Table 1
Odorant composition in US pipeline natural gas

Natural gas odorant blend	All mercaptan blend	Mercaptan/alkyl sulfide blend	Thiophene/mercaptan blend	Thiophene (99.9%)
Natural gas odorant market share, %	40–55	40–55	5	<1
Composition breakdown	100% mercaptan	Sulfide content is usually 20–50%, but can be 7–90% in limited areas	Thiophene content is usually 30–50%, but can be −70% in limited area	100% thiophene

Composition breakdown	Concentration, ppmv
Hydrogen sulfide	0.4
Carbonyl sulfide	1.0
Total mercaptans (methyl mercaptan; ethyl mercaptan; isopropyl mercaptan; *n*-propyl mercaptan; *tertiary* butyl mercaptan)	6–8
Dimethyl sulfide	1–2
Tetrahydro thiophene	1–2
Carbon disulfide	<1

Table 2
Properties of logistic fuels JP-8, DF-2 and NATO F-76

Property	JP-8	DF-2	NATO F-76
Molecular formula (average)	$C_{11.5}H_{22.8}$	$C_{14.6}H_{26.3}$	$C_{14.8}H_{26.9}$
Molecular weight	161	202	205
H/C ratio (molar)	1.98	1.80	1.82
Sulfur mass, %	0.3 (max)	0.5 (max)	1.0 (max)
Specific gravity	0.809	0.849	0.852
Net heating value, Btu/lb	18,570	18,442	18,358
Net heating value, Btu/gal	125,038	130,340	130,235

fuel require sulfur clean-up followed by adiabatic pre-reforming to convert higher hydrocarbons to methane. A discussion on each step is presented below.

2.1. Sulfur clean-up

Sulfur compounds are major poisons of nickel catalysts used in DFC internal reforming since nickel has a strong tendency to chemisorb sulfur. The sulfur poisoning causes rapid loss of reforming activity and life. Sulfur chemisorbs on active nickel, forming nickel sulfide of poor reforming activity (as shown in Reactions (1)–(3)).

$$\mathrm{Ni} + \mathrm{H_2S} \rightarrow \mathrm{NiS} + \mathrm{H_2} \qquad (1)$$

$$\mathrm{Ni} + \mathrm{COS} \rightarrow \mathrm{NiS} + \mathrm{CO} \qquad (2)$$

$$\mathrm{Ni} + \mathrm{RSH} \rightarrow \mathrm{NiS} + \mathrm{RH} \qquad (3)$$

The sensitivity of the catalyst to sulfur poisoning increases as the reforming temperature is lowered. There is a threshold limit for given conditions, below which the poisoning effect is not apparent. With very low levels of sulfur in the natural gas, significant loss of activity is only apparent after quite extended time of operation. It is reported that 0.2 mg sulfur/m^2 of catalyst surface may completely deactivate the reforming catalyst [2]. Thus, for a stable long-term operation of DFC, it is recommended to prevent sulfur from entering the DFC cells by utilizing an efficient desulfurization system.

2.1.1. Natural gas desulfurization

Desulfurization of natural gas depends on the amount of sulfur as well as the nature of the sulfur compounds. Natural gas consisted of H_2S, COS and mercaptans added as odorants. Organic sulfur such as mercaptan is difficult to remove. Table 1 shows the composition of mercaptans used in US natural gas distribution.

Low temperature desulfurization of natural gas using absorbents by activated carbon is an attractive and economical process. Currently, widely used in petroleum industries. The capacity of adsorbents can be further enhanced by impregnation of copper or potassium hydroxide on activated carbon. For continuous operation, two or more absorbent beds are designed so that one is on-line, while the other is being regenerated/replaced (Fig. 2). Testing at FCE with natural gas fuel containing 2–12 ppmv sulfur has demonstrated the capability to achieve <0.1 ppmv sulfur level.

2.1.2. Fuel processing requirements for NATO F-76 marine distillate fuel for DFC application

Under an earlier DOD-sponsored project, FCE tested fuel processing systems for fuel cell applications using DF-2 and JP-8. Table 2 compares the properties of JP-8, DF-2 and NATO F-76. The marine distillate NATO F-76 is slightly heavier than DF-2 in terms of molecular weight and specific gravity. It also has the highest maximum sulfur level. The

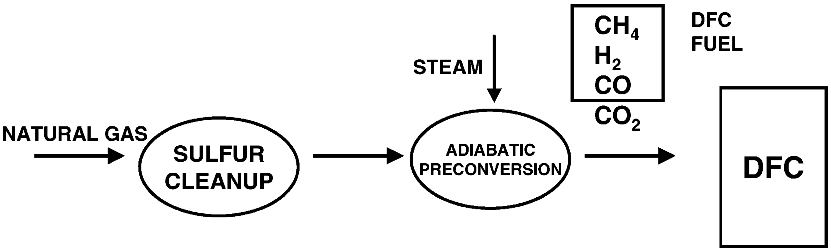

Fig. 1. Fuel processing approach for DFC.

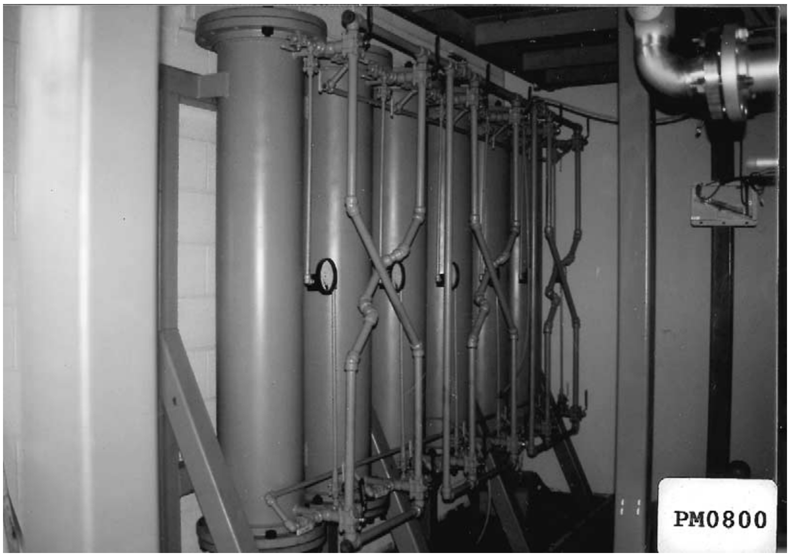

Fig. 2. FCE's desulfurization facility for 250 kW capacity natural gas clean-up.

approach to processing NATO F-76 marine distillate fuel is based on desulfurization followed by adiabatic prereforming to a methane-rich gas which can be utilized directly by the DFC. This approach minimizes changes to the DFC power plant being developed by FCE for commercial applications.

As discussed above, the carbonate fuel cell stack operates at temperatures sufficiently high for internal steam reforming, it is capable of utilizing methane directly to produce the needed anode hydrogen fuel. This means that the fuel processor need only convert the NATO F-76 fuel to methane rather than hydrogen. This feature provides the carbonate fuel cell system with a major advantage over lower temperature fuel cell systems because the conversion of NATO F-76 to methane can be carried out adiabatically thereby maximizing the efficiency of the fuel processor.

Both prereforming catalysts and fuel cells require sulfur levels in fuels to be below 0.1 ppmv. Although worldwide standards are gradually lowering sulfur levels in transportation fuels, it will still be necessary to have sulfur removal steps in fuel cell power plants. Deep desulfurization can be achieved by hydrodesulfurization (HDS). Testing at FCE with NATO F-76 distillate fuel containing 3800 ppmv sulfur has demonstrated the capability to achieve a 0.1 ppmv sulfur level with this process.

2.2. Prereforming

2.2.1. Natural gas prereforming

Natural gas may contain higher hydrocarbons ranging from 1 to 4% depending on the source and region. These higher hydrocarbons may promote coking on the reforming catalyst and subsequent activity loss. Prereforming is an important fuel processing step after desulfurization as shown in Fig. 1, offering the following benefits:

- Prereforming of natural gas at lower temperatures would knockout higher hydrocarbons to prevent coking on downstream reforming catalyst.
- Prereforming of natural gas can also provide some hydrogen to keep internal reforming catalyst in a reducing environment.
- Preconverter also serves as sulfur guard (if any sulfur breakthrough exiting from the desulfurizer bed) to prevent internal reforming catalyst from sulfur poisoning.

The natural gas–steam mix is fed to the adiabatic converter operating at 400–500 °C. The prereforming of the natural gas helps to increase the capacity of the internal reforming catalyst. Excess steam requirement for reforming is also reduced which results in energy savings. The prereformer converts C_2 and C_3

hydrocarbons to methane. The higher hydrocarbons can reform easily at lower temperatures than methane. The overall prereforming reaction is endothermic and the temperature declines toward the exit end of the bed. This also favors the exothermic shift reaction and methanation reactions. Since both of these reactions convert carbon monoxide, a typical prereformer will produce an outlet gas containing mainly hydrogen and methane.

Reforming reaction in internal reforming utilizes heat generated by DFC cell reaction and thus helps in stack thermal management. It is therefore recommended not to reform much of methane to hydrogen during prereforming, but only sufficient to provide necessary hydrogen for protecting the reformer catalyst from oxidation. On the other hand, prereforming needs to convert 100% of higher hydrocarbons to methane to avoid carbon deposition. Testing at FCE for over 11,000 h of operation indicates a stable performance of the preconverter providing methane-rich gas fuel for DFC. The gas composition of the fuel gas produced is close to the expected equilibrium.

2.2.2. *NATO F-76 distillate fuel prereforming*

Adiabatic prereforming of desulfurized NATO F-76 is being used to convert the naval distillate to a methane-rich gas useable by the fuel cell. As shown by the reactions below, the endothermic reforming reaction is driven by the exothermic shift and methanation reactions, thereby eliminating the need to provide heat to the reactor.

(1) Reforming : $C_nH_m + nH_2O \rightarrow nCO + (n + m/2)H_2$
(2) Shift : $CO + H_2O \rightarrow CO_2 + H_2$, $-H^{\circ}_{298} = 41.2\,\text{kJ mol}^{-1}$
(3) Methanation : $CO + 3H_2 \rightarrow CH_4 + H_2O$, $-H^{\circ}_{298} = 206.2\,\text{kJ mol}^{-1}$

Testing at FCE for over 1400 h of operation indicates that a complete conversion of the naval distillate is achieved with no detectable higher hydrocarbons above C_1. The gas composition of the fuel gas produced is close to the expected equilibrium as shown in Table 3. A subscale carbonate fuel cell stack built with 9 ft^2 cells has operated on pre-reformed NATO F-76 for over 1000 h with stable performance.

Table 3
Prereformer exit gas composition measured values are close to equilibrium

	Calculated equilibrium (vol.%)	Measured (vol.%)
H_2	24.3	23.4
CH_4	51.7	51.8
CO_2	23.4	24.2
CO	0.6	0.6
Total	100	100

3. Internal reforming

While the internal reforming fuel cells help to bring about a simple overall system design desired for cost and reliability, the fuel cell anode needs to be integrated with the internal reforming catalyst. In the conventional DFC approach called direct internal reforming (DIR), the catalyst is located in the anode compartment where it gets exposed to the electrolyte-containing environment. The internal reforming catalyst is required to remain sufficiently active throughout the life in the presence of the carbonate vapors. An alternate approach named indirect internal reforming (IIR) where the reforming catalyst is placed in between cell groups and not exposed to carbonate vapor has also been developed to achieve much longer catalyst life while retaining thermal management benefits. A hybrid system that incorporates both the DIR and the IIR schemes is being adopted by FCE and other DFC developers.

In the DIR assembly described in US Patent No. 3,488,226, reforming catalyst is placed inside the anode compartment of each fuel cell and therefore is directly in the cell environment. The DIR scheme results in high conversion and high cell efficiencies as compared to external reforming systems. However, the DIR cells experience a large temperature gradient due to the extremely fast reforming reaction rates at the fuel inlet edge and almost no reforming reaction at the fuel exit edge. In a DIR cell, the need to reform the fuel at the entrance to ensure high cell performance conflicts with heat balance. In practical DIR designs, excessive reforming reaction at the entrance is suppressed to achieve heat balance at the cost of some performance penalty. An approach for the distribution of the reforming load throughout the cell area has been investigated using a novel graded catalytic system.

Fig. 3. A 10 kW IIR plate.

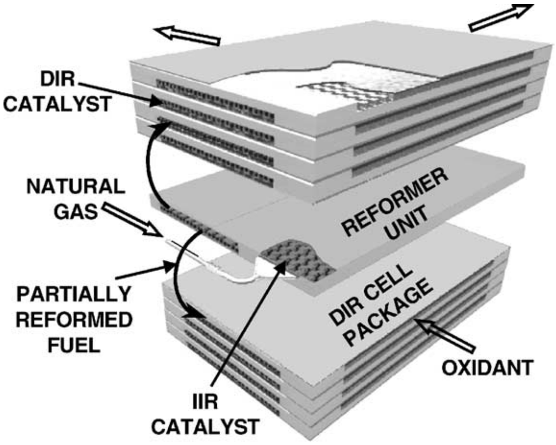

Fig. 4. IIR–DIR stack concept.

This approach has been verified in an 8 kW DIR-only stack test at Elkraft Power Company's Kyndby (Denmark) site in 1996. Such attempts to decelerate the reforming reaction at the inlet, of course, incur some performance penalty due to the low average hydrogen partial pressure that hinders the fuel cell reaction.

In the alternate IIR, described in US Patent No. 4,877,693, a reforming plate (or unit) is placed in between fuel cell groups in a stack. The photograph of the reformer unit is presented in Fig. 3. A group of cells typically might comprise of 5–15 cells. The reforming units (RUs) are in thermal communication with the fuel cells but without the hydrogen and steam synergy benefits between the reforming and the anode electrochemical reactions. The reformation achieved is dependent on steam-to-carbon ratio and operating temperature. The IIR design can be viewed as a fuel cell-integrated flatbed reformer. The IIR catalyst is not exposed to the cell environment, and remains very stable in activity with life. Therefore, the extent of the reforming reaction and its distribution can be engineered to provide the most desired heat balance to achieve relatively uniform temperature distribution. The cost of the RU needs to be sufficiently low for commercial use. The cost of the FCE's IIR components is projected to be $<10\%$ of the cell packages of a stack.

The reforming reaction in the IIR section is independent of the anode reaction. Also, the IIR catalyst is not exposed to the cell environment, and is observed to remain very stable in activity with life. Therefore, the extent of the reforming reaction and its distribution can be engineered to provide the most desired heat balance to achieve relatively uniform temperature distribution. In a DIR cell, the need to reform the fuel at the entrance to ensure high cell performance conflicts with heat balance. In practical DIR designs, excessive reforming reaction at the entrance is suppressed to achieve heat balance at the cost of some performance penalty.

A hybrid assembly incorporating both the direct and IIR combines the benefits of the direct and indirect arrangements. Fig. 4 shows the hybrid arrangement in which the reforming catalyst is placed in the anode compartment of each fuel cell and an RU is placed in between fuel cell groups. This IIR–DIR approach is preferred from performance, life, and thermal management considerations. The IIR–DIR hybrid design provides an additional degree of freedom in controlling the temperature than the DIR-only design, because the extent and the location of the reforming in the RU can be adjusted by design. Therefore, significantly improved temperature profile can be achieved for the IIR–DIR design. FCE results point out that such a hybrid stack operates with a much narrower temperature band. As a result, for a set maximum temperature (fuel cell life is a function of the maximum operating temperature), the hybrid design will result in the highest average cell temperature, hence, a much higher electrochemical performance. The

MELCO researchers also arrived at same conclusion [3,4].

4. Catalysts

The IIR catalysts have shown negligible deactivation in fuel cell use because they are generally designed for much higher operating temperature. On the other hand, the stability of the DIR catalyst is strongly affected by the fuel cell environment, as investigated by FCE [1], BG [5], Mitsubishi [6], CNR-TAE Institute (Italy) [7] and other laboratories [8–10]. Stability of the DIR catalyst varies significantly with experimental conditions and the type of alkali carbonate used in fuel cell. The major causes for the deactivations of the DIR catalyst are: (1) electrolyte contamination, (2) catalyst sintering, and (3) irreversible catalyst poisoning due to the presence of sulfur impurity in the feed gas. As mentioned earlier, sulfur can be removed selectively from the natural gas by using effective adsorbents. Conventional high temperature sintering for the reforming catalyst is not considered an issue due to the somewhat low carbonate fuel cell operating temperature (~650 °C). This leaves electrolyte to be the major contributor for DIR catalyst deactivation, which is transported to the catalyst as vapor or by creepage on the metal hardware surface.

Electrolyte creepage towards the catalyst has been resolved by advanced cell design at FCE and others utilizing a non-wetting metal surface like Ni. Hence, current DIR catalyst deactivation is mainly by vapor phase transport of electrolyte onto the catalyst. This process results in poisoning of the active sites as well as structural deterioration. Deactivation mechanism by carbonate vapor was elaborated earlier by Katikaneni and co-workers [1], Matsumura and Hirai [6], Berger et al. [10–12], Clarke and Smith [13], and Cavallaro et al. [14]. Electrolyte pickup by the catalyst results in decreasing active metal and BET surface areas. Active nickel sites were further decreased significantly as a result of possible accelerated sintering of the catalyst in the presence of electrolyte. In addition, metal crystallite size and average pore size also increased linearly, indicating sintering of the active nickel metal and support. The average nickel crystallite size has been measured by several investigators using different techniques such as XRD, H_2/CO chemisorption and TEM. FCE results using these techniques indicate that the crystallite size increased by 5–6 folds, resulting in deactivation of the catalyst. In addition, pore filling/plugging of the catalyst support and coverage of the active nickel sites by electrolyte vapor may further contribute to the overall deactivation. Significant success has been achieved in reducing these undesired effects by developing low-cost advanced catalysts.

Alkali resistant supports such as MgO, calcium aluminate, and α-alumina were used earlier [15] for improved DIR catalyst stability. Among these, MgO supported Ni catalysts have shown the best activity and stability [6]. However, the MgO support still undergo sintering in the presence of electrolyte, contributing to catalyst deactivation. Thus, researchers concentrated on novel catalyst formulation to mitigate sintering of the catalyst to improve stability.

In Europe, a project team consisting of ECN, British Gas and CNR worked on developing of DIR catalyst. CNR has concentrated on the application of Ni/MgO system [14] and British Gas on alumina-based nickel catalysts [5]. Several Japanese companies are also involved in the developing of stable DIR catalyst. Mitsubishi Electric Company is using Ni/MgO-based system. On the other hand, Sanyo and Tonen have carried out extensive work on the application of Ru/ZrO_2 catalysts for LPG internal reforming. Ru- and Rh-based catalyst exhibited better activity and stability than nickel catalysts [16]. However, the catalyst cost may be too high to be economical.

Hence, the recent interest at FuelCell Energy [1], British Gas [5,17], CNR and other laboratories was to develop a suitable nickel-based catalyst formulation with suitable alkali resistant supports and promoters/or stabilizers. Out-of-cell accelerated tests have been developed at FCE and other laboratories to evaluate catalyst performance and stability (1,10,17). These tests produce a more severe alkali environment than an actual cell test. Based on the short time accelerated test results, catalyst stability and end-of-life activity in actual cell environment can be projected. FCE has identified a more stable and active Ni-based catalyst. Life testing of this advanced catalyst under accelerated decay conditions in out-of-cell as well as in-cell has been completed. The advanced DIR catalyst provided ~5

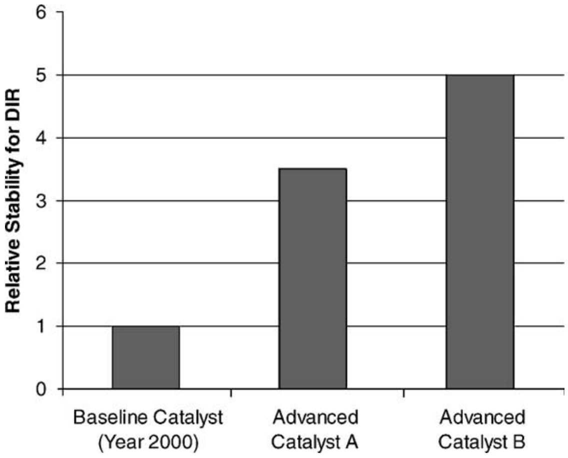

Fig. 5. Improvement of DIR catalyst stability.

times longer life over the baseline catalyst. The performance of the FCE's advanced catalysts is presented in Fig. 5.

5. Conclusions

The DFC power plants are inherently simple, compact, and cost-effective compared with external reforming type fuel cells. A fuel cell power plant based on FuelCell Energy's DFC^{TM} technology promises significant fuel saving advantages over power generation alternatives due to its high efficiency. Fuel clean-up and preprocessing requirements for natural gas and diesel fuels have been designed and verified in stacks. Processing of naval distillate NATO F-76 can be achieved efficiently for the internally reforming carbonate fuel cell, thereby making this fuel compatible with the DFC^{TM} for marine fuel cell power applications. The IIR–DIR hybrid design has been tested in stacks and systems. The DFC power plant technology has been demonstrated by operating using both natural gas and diesel fuels. Fuel processing catalysts and internal reforming (IIR and DIR) catalysts have been identified and developed for the commercial design DFC power plant.

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