High Temperature Ion Conducting Ceramics

by T. A. Ramanarayanan, S. C. Singhal, and E. D. Wachsman

Research in materials today has progressed from materials exhibiting structural functionality to those exhibiting electronic functionality. The study of solid-state ionics ushers in a new era of chemically functional materials. This chemical functionality arises out of the defect equilibria of these materials, and results in the ability to transport chemical species and actively participate in chemical reactions at their surface. These chemically functional materials provide a promise for the future. They allow the harnessing of our natural hydrocarbon energy resources through highly efficient and non-polluting methods.

Numerous solid-state materials conduct cations (*e.g.*, Li^+ and Na^+) and anions (*e.g.*, F^- and O^{2-}) and are therefore chemically functional. Among these, activities in the ECS High Temperature Materials Division tend to focus on oxide ion conducting ceramics. These materials are used in, and are being investigated for, a wide variety of technological applications. Their required functionality goes far beyond mechanical strength and toughness, to include such properties as ionic and electronic conductivity, thermochemical stability, and catalytic activity. These latter properties arise out of their defect chemistry and resultant wide range in oxygen stoichiometry. Depending on their defect chemistry and environment, these materials can exhibit exclusively ionic (electrolyte) or electronic conduction, or mixed (both ionic and electronic) conduction.

Ionic conduction involves hopping from an occupied to a vacant lattice (or interstitial) site, as shown in Fig. 1a. At high temperatures, the ionic conductivity of these solid-state materials is comparable to that of aqueous solutions at ambient conditions. For example, at 1000°C the ionic conductivity of yttria-stabilized zirconia (YSZ) is 0.1 $S{\cdot}cm^{-1}$, and in fact, there are numerous oxides that have significantly higher conductivity at lower temperatures (albeit with a stability trade off). The high conductivity of these materials is attributable to the high concentration of mobile defects: oxygen vacancies ($V_o^{\bullet\bullet}$), oxygen interstitials (O_i''), electrons (e'), and electron-holes (h•). Each of these defects can be treated as a chemical species and their concentration determined from thermodynamics. By evaluating the heterogeneous equilibria between gaseous oxygen and the solid state,

$$O_o^x = 1/2O_2 + V_o^{\bullet\bullet} + 2e' \qquad (1)$$

combined with the internal chemical equilibria for ionic defect and electron-hole pair formation and the concentration of aliovalent dopants (such as Y^{3+} on a Zr^{4+} site - $Y_{Zr'}$), one can calculate the concentration of all species as a function of the external oxygen partial pressure, P_{O2}. This solution for the defect concentrations is shown graphically in a typical electrolyte defect equilibrium diagram, Fig. 1b. (For a review of defect equilibria, the reader is referred to References 1-2 and the numerous references therein.) This diagram shows three regions, low, intermediate, and high P_{O2}, in which a pair of defects dominate the neutrality relationship due to their relative high concentration. Thus the same material can exhibit respectively, n-type, ionic, and p-type conductivity depending on P_{O2}. Each of the mobile species is then transported through the material in response to an applied chemical (due to a ΔP_{O2}) or electrical potential.

Oxide materials such as YSZ, rare earth doped ceria, rare earth doped bismuth oxide, and doped lanthanum gallates have been widely investigated as oxide ion conductors. Of these materials, YSZ has been most successfully employed. The yttrium oxide dopant serves dual roles: it stabilizes the high temperature cubic phase in zirconia and also generates oxygen vacancies through the following defect reaction:

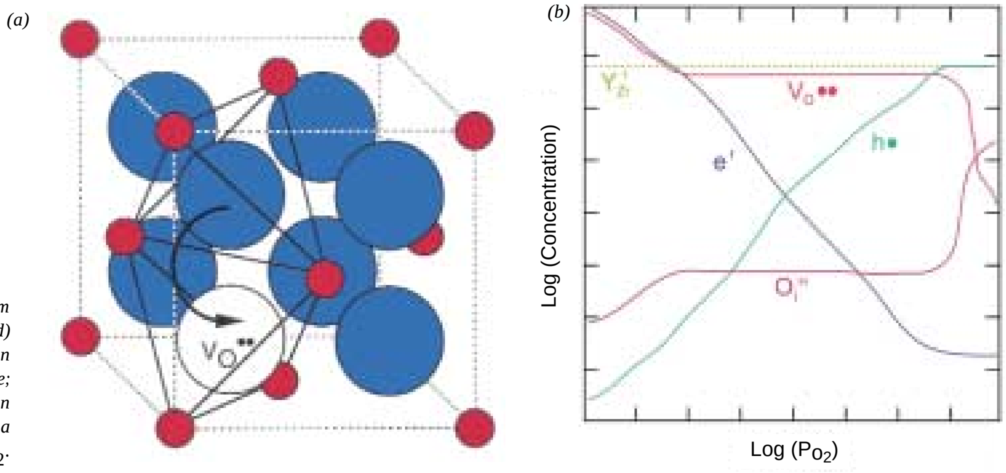


FIG. 1. (a) Mechanism of transport from an occupied (tetrahedrally coordinated) anion lattice site to a vacant anion site in a fluorite oxide; (b) Concentration of defects in yttria-stabilized zirconia as a function of P_{O2}.

$$Y_2O_3 = 2Y_{Zr}' + 3\,O_o^{x} + V_o^{\bullet\bullet} \qquad (2)$$

thus creating more of the conducting species ($V_o^{\bullet\bullet}$).

The high oxide ion conductivity over wide ranges of temperature and oxygen pressure in these materials has led to their use as electrolytes in a variety of electrochemical applications. High temperature solid oxide fuel cells (SOFCs) for electric power generation are now nearing commercialization. Oxygen sensors are widely used in combustion control, especially in automobiles, atmosphere control in furnaces, and as monitors of oxygen concentration in molten metals. Other applications include electrochemical pumps for control of oxygen potential, steam and CO_2 electrolyzers, and high temperature reactors for chemicals production from hydrocarbons. Some of these applications are discussed below.

Ceramic Fuel Cells for Power Generation

High temperature ceramic fuel cells, more commonly known as solid oxide fuel cells or SOFCs, offer a clean, low-pollution technology to electrochemically generate electricity at high efficiencies; their efficiencies are not limited by the Carnot cycle of a heat engine. These fuel cells provide many advantages over traditional energy conversion systems including high efficiency, reliability, modularity, fuel adaptability, and very low levels of NO_x and SO_x emissions. Quiet, vibration-free operation of SOFCs also eliminates noise usually associated with conventional power generation systems. Furthermore, because of their high temperature of operation (~800-1000°C), natural gas fuel can be reformed within the cell stack eliminating the need for an expensive, external reformer system.

An SOFC essentially consists of two porous electrodes separated by a dense, oxide ion conducting electrolyte. The operating principle of such a cell is illustrated in Fig. 2. Oxygen supplied at the cathode (air electrode) reacts with incoming electrons from the external circuit to form oxide ions, which migrate to the anode (fuel electrode) through the oxide ion conducting electrolyte. At the anode, oxide ions combine with H_2 (and/or CO) in the fuel to form H_2O (and/or CO_2), liberating electrons. Electrons (electricity) flow from the anode through the external circuit to the cathode. To keep the cell resistance low, the electrolyte (typically 10 mol% YSZ) is fabricated in the form of a thin film. The thermal expansion of 10 mol% YSZ is about 10 x 10^{-6}/°C; materials for all other cell components are chosen to have thermal expansion near this value to minimize thermal stresses.

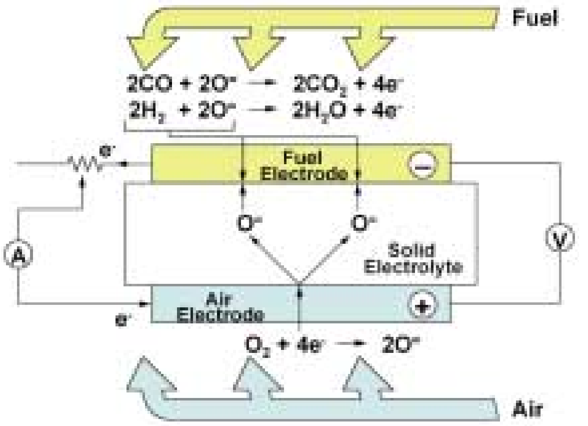
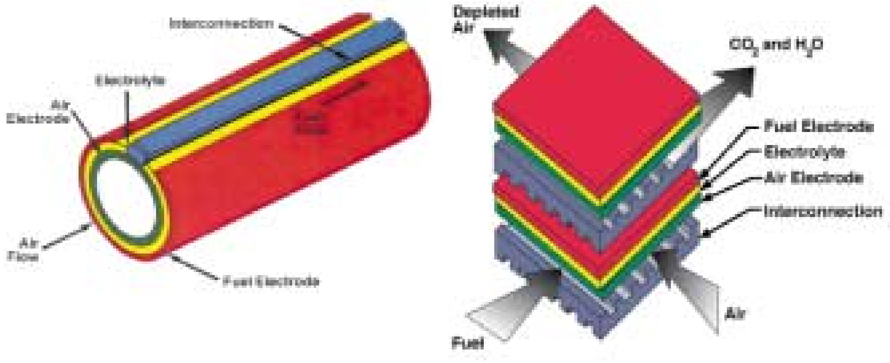


Fig. 2. (right) Operating principle of a solid oxide fuel cell.

Fig. 3. (below) (a) Tubular SOFC design; (b) Planar SOFC design.



Lanthanum manganite suitably doped with alkaline and rare earth elements is generally used for the cathode. Lanthanum manganite is a p-type perovskite oxide and shows reversible oxidation-reduction behavior. The material can have oxygen excess or deficiency depending upon the ambient oxygen partial pressure and temperature. The electronic conductivity of lanthanum manganite is due to hopping of an electron hole between the +3 and +4 valence states of Mn. This conductivity is enhanced by doping with a divalent ion such as calcium or strontium.

The reducing conditions on the fuel side of an SOFC permit the use of a metal such as nickel for the anode. However, the thermal expansion of nickel, 13.3 x 10^{-6}/°C, is considerably larger than that of YSZ. Nickel can also sinter at the cell operating temperature resulting in a decrease in the fuel electrode porosity. These problems are circumvented by forming a skeleton of YSZ around the nickel particles. The YSZ skeleton prevents sintering of the nickel particles, decreases the fuel electrode thermal expansion coefficient bringing it closer to that of the electrolyte, and provides better adhesion of the fuel electrode with the electrolyte.

For the interconnection (the bipolar material), generally doped lanthanum chromite is used. Lanthanum chromite is a p-type conductor; its conductivity is due to small polaron hopping from room temperature to 1400°C at oxygen pressures as low as 10^{-16} kPa. The conductivity is enhanced as lower valence ions (*e.g.*, Ca, Mg, Sr, etc.) are substituted on either the La^{3+} or the Cr^{3+} sites. Recently, high temperature metallic alloys have also been employed as the interconnection material, particularly for cells intended for operation at lower temperatures (~ 800°C).

SOFCs of two different basic geometries are being developed; tubular and planar;[3-6] these are illustrated in Fig. 3. The materials for cell components in these two designs are very similar in nature. In the tubular design, pioneered by the Siemens Westinghouse Power Corporation, the cell components are deposited in the form of thin layers on a cathode tube.[5] The YSZ electrolyte is fabricated in the form of a 40 µm thick dense layer by an electrochemical vapor deposition process (EVD).[7] Deposition of the YSZ electrolyte film by a more cost-effective non-EVD technique, such as plasma spraying or colloidal/electrophoretic deposition followed by sintering, is also being investigated to reduce cell cost.

The interconnection is deposited in the form of an 85 µm thick, 9 mm wide strip along the cathode tube length by plasma spraying. The anode, 100-150 µm thick layer of nickel/YSZ, is deposited by first applying nickel powder slurry over the electrolyte and then growing YSZ around the nickel particles by the same EVD process as used for depositing the electrolyte. Deposition of a Ni-YSZ slurry over the electrolyte followed by sintering has also yielded anodes that are equivalent in performance to those fabricated by the EVD process; use of this non-EVD process results in a substantial reduction in the cost of manufacturing SOFCs.

A large number of tubular SOFCs has been electrically tested for up to about 70,000 hours. These cells perform satisfactorily for extended periods of time under a variety of operating conditions with less than 0.1% per 1,000 hour performance degradation. To construct an electric generator, individual cells are connected both in parallel and in series to form a semi-rigid bundle that becomes the basic building block of the generator. A three-in-parallel by eight-in-series cell bundle is shown in Fig. 4.[6] Siemens Westinghouse has designed, built, and operated successively larger SOFC power generation systems since 1984. A 100 kW atmospheric SOFC power generation system, shown in Fig. 5, operated very successfully in The Netherlands for a (contract) period of two years without any measurable performance degradation. The system provided 110 kW ac to the Dutch grid at an efficiency of 47%. In addition, it produced approximately 85 kW of hot water for the local district heating system. Such atmospheric SOFC systems are ideally suited for distributed power generation and cogeneration.

SOFC operation at elevated pressures yields a higher cell voltage at any current density due to increased Nernst potential and reduced cathode polarization, and thereby permits higher stack efficiency and greater power output. With pressurized operation, SOFCs can be successfully used as replacements for combustors in gas turbines; such pressurized SOFC-gas turbine hybrid power systems are calculated to reach efficiencies approaching 70%. Siemens Westinghouse, under the sponsorship of Southern California Edison, recently fabricated a 250 kW class pressurized SOFC-gas turbine hybrid power system; this system combines a 200 kW SOFC module operating at 355 kPa pressure and a 50 kW microturbine. The system is installed at the National Fuel Cell Research Center on the University of California at Irvine campus, and is presently undergoing proof-of-concept testing.

Even though the tubular SOFCs have progressed the most, their electrical resistance is high, specific power output (W/cm^2) and volumetric power density (W/cm^3) low,[5] and manufacturing costs high. The low power density (0.25 to 0.30 W/cm^2) makes tubular SOFCs suitable only for stationary power generation and not very attractive for mobile applications. Planar, particularly anode-supported, SOFCs, in contrast, are capable of achieving very high power densities of up to about 2 W/cm^2.[8] In the planar design, the cell components are configured as thin, flat plates. The interconnection having ribs on both sides forms gas flow channels and serves as a bipolar gas separator contacting the anode and the cathode of adjoining cells. The dense electrolyte and interconnection are fabricated by tape casting, powder sintering or CVD, whereas the porous electrodes are applied by slurry methods, screen printing, or by plasma spraying.

Sizeable cost reductions are possible with the planar design through a concept called "mass customization" that is being pursued in the recently initiated U.S. Department of Energy's Solid-State Energy Conversion Alliance (SECA). This concept involves the development of a 3 to 10 kW size core SOFC module, that can be mass produced and then combined for different size applications in stationary power generation, transportation, and military market sectors, thus eliminating the need to produce custom-designed and inherently more expensive fuel cell stacks to meet a specific power rating.

FIG. 4. Photograph of a three-in-parallel and eight-in-series cell bundle.

FIG. 5. Siemens Westinghouse 100 kW SOFC system installed in The Netherlands.

Even though the polymer electrolyte membrane (PEM) fuel cell is generally regarded as the fuel cell of choice for transportation applications, they require pure H_2, with no CO, as fuel to operate successfully. However, presently no H_2 infrastructure exists, and on-board reformer systems to produce H_2 from the existing fuel base (gasoline, diesel) are technically challenging, complex, and expensive. Furthermore, it is difficult to eliminate all the CO from the reformate stream. In contrast, SOFCs can use CO along with H_2 as fuel, and their higher operating temperature and availability of water on the anode side makes on-cell or in-stack reformation of hydrocarbon fuels feasible. Also, no noble metal catalysts are used, thus reducing the cost of the cells. The initial application of SOFCs in the transportation sector will be for on-board auxiliary power units (APUs). Such APUs, operating on existing fuel base (gasoline, diesel), will supply the ever-increasing electrical power demands of luxury automobiles, recreational vehicles, and heavy-duty trucks. Delphi Automotive Systems recently developed and tested a 5 kW APU using anode-supported planar SOFCs. The challenge in commercializing planar SOFCs offering high power densities requires successful development of seals for isolating oxidant from the fuel, development of low-cost oxidation-resistant metallic alloys for use as interconnection, ability for rapid start up and thermal cycling, and lower cost.

Related High Temperature Materials Division Symposia

International Symposium on Ionic and Mixed Conducting Ceramics

This continuing symposia series made its debut at the fall 1991 meeting of the Society in Phoenix, Arizona. During the past decade, it has provided a rich forum for the international research community to share and discuss the forefront activities that are ongoing in the exciting field of ionic and mixed conducting ceramics. Some of the specific themes include ionic transport in solid electrolytes, mixed conduction in ceramics, thermo- and chemo-mechanical properties of mixed conductors, hydrocarbon conversion by ceramic electrochemistry, electrocatalytic phenomena, electrode reactions involving ceramic cells, ceramics-based fuel cells and batteries, and thin film ceramic membranes.

The lead symposium organizer is T. A. Ramanarayanan, Exxon/Mobil Research and Engineering Company (e-mail: taraman@erenj.com). The next (fourth) symposium in this series will be held at the ECS/ISE Joint International Meeting in San Francisco, California, September 2-7, 2001.

International Symposium on Solid Oxide Fuel Cells (SOFCs)

This continuing symposia series provides an international forum for the presentation and discussion of developments related to solid oxide fuel cells based on zirconia or other oxide electrolytes. Topics addressed include materials for cell components. (*e.g.* electrolyte, electrodes, and interconnection); fabrication methods for complete cells and components; cell design, electrochemical performance and modeling; stacks and systems for residential and automotive applications; and field tests of SOFC demonstration systems.

This symposia series started in 1989 at the Society's fall meeting in Hollywood, Florida and has since become the leading symposium in the field of solid oxide fuel cells. The symposium is held every two years, rotated among the USA, Europe, and Japan, and co-sponsored by the SOFC Society of Japan.

The lead symposium organizer is S. C. Singhal, Pacific Northwest National Laboratory (email: singhal@pnl.gov). The next symposium in this series (SOFC-VIII) will be held at the Society's 203rd meeting in Paris, France, April 27-May 2, 2003.

International Symposium on Solid-State Ionic Devices

Solid-state electrochemical devices, such as batteries, fuel cells, membranes, and sensors, are becoming more and more important for our technologically driven lifestyles. The development of these devices involves common research themes such as ion transport, interfacial phenomena, and device design and performance. The intent of this symposium is to bring together researchers in all fields of solid-state ionics. This continuing symposia is a forum for current advances in solid-state ion conducting materials, regardless of the class of materials or whether the solid-state is amorphous or crystalline, and the design, fabrication, and performance of devices that utilize them.

Topics covered include modeling and characterization of defect equilibria, ionic and electronic transport, interfacial and electrocatalytic properties of ion conducting ceramics, novel synthesis and processing of thin films, membranes, and nanostructured materials or devices; the effect of nanostructures on ionic transport and catalytic activity; electrode kinetics, interfacial phenomena, and electrode microstructure pertaining to chemical sensors, fuel cells, gas separation membranes and reactors, solid-state battery and microbattery electrodes.

The lead symposium organizer is E. D. Wachsman, University of Florida (email: ewach@mail.mse.ufl.edu). The next symposium in this series (SSID-III) will be held at the Society's 202nd meeting in Salt Lake City, Utah, October 20-25, 2002.

Conducting Ceramics for Production of Chemicals

An important application of conducting ceramics is in the production of high value chemicals using high temperature processes. Two types of ceramics are useful in this regard: mixed electron-ion conductors for permeation membrane reactors, and purely ionic conducting materials for solid electrolyte reactors. Ceramics in which oxide ions are the main ionically conducting species have received the widest interest.

Mixed Conducting Membrane Reactors—The idea that significant levels of electronic conduction can be deliberately introduced into predominantly ionically conducting systems goes back to early work by Engell and Vygen[9] on slags produced in steel making operations where they observed the existence of Fe in two valence states (+2 and +3) depending on the oxygen partial pressure, leading to electronic conduction in the slags. This idea has been used in introducing electronic conduction into the ZrO_2-Y_2O_3 matrix by dissolving an oxide whose cation can exist in multiple valence states; cerium oxide, titanium oxide, and terbium oxide have been investigated. Cales and Baumard[10] showed that in the ZrO_2-Y_2O_3-CeO_2 system, a maximum in electronic conduction occurred corresponding to oxygen partial pressures at which Ce^{3+} and Ce^{4+} ions would exist in roughly equal amounts. This is because the probability of electron jump between these two valence states is proportional to the product of their concentrations, which is maximized when the two concentrations are equal.

The above mixed conductors belong to the fluorite structure. Additionally, compositions belonging to the perovskite, pyrochlore, and brownmillerite classes have also been shown to exhibit mixed conduction. In addition to these single-phase mixed conducting systems, dual phase materials have been developed where one phase fulfils the ionic conduction requirements while the other provides the electronic transport path.

Hazbun[11] describes in U.S. Patent 4,827,071 a process for hydrocarbon conversion reactions involving a double-layered ceramic reactor. One layer is a compact mixed conductor made up of ZrO_2-Y_2O_3 with dissolved CeO_2 or TiO_2 while the other layer is porous ZrO_2-Y_2O_3 containing an appropriate catalyst. Oxygen from air is ionized to O^{2-} at the tube exterior, oxide ions pass through the mixed conductor and appropriate oxidative reactions occur at the interior tube surface. Hazbun describes the conversion of methane to ethane and ethylene in the temperature range 700-900°C, ethylene to ethylene oxide between 270 and 350°C, and propylene to propylene oxide between 300 and 400°C. British Petroleum (BP) has developed a syngas ($CO+H_2$) production technology called Electropox (Electrochemical Partial Oxidation). The heart of the process is a mixed conducting ceramic membrane that separates oxygen from air by oxygen ionization, and the oxide ions transport to the opposite surface of the membrane to trigger the partial oxidation reaction. Membrane materials of sufficient strength, chemical stability and performance have been developed. Based on bench-scale simulations, considerable savings are projected for this process when compared with conventional steam reforming or combined reforming. BP also has developed a cheap and reliable fabrication process that can be used to produce commercially viable membrane modules.[12,13]

Air Products & Chemicals and Praxair are similarly developing mixed conducting membrane technology to produce pure O_2 (and N_2) from air at a smaller size scale where cryogenic plants are not considered economical.

Solid Electrolyte Reactors—Exclusive ionic conduction in solid electrolyte ceramics has led to their use in driving specific chemical reactions. Examples include high temperature steam electrolysis, upgrading of hydrocarbons, and pollution control (*e.g.*, NO_x reduction). Of particular interest in production of chemicals is a phenomenon termed NEMCA (Non-Faradaic Electrochemical Modification of Catalytic Activity) investigated by Vayenas and co-workers.[14] Enhancement of catalytic activity due to the formation of heterojunctions between dissimilar materials or the application of an applied potential can influence the mechanistic steps involved in heterogeneous chemical reactions. The effect can be best described by referring to the graph in Fig. 6, which shows in the inset the electrochemical cell arrangement. The reaction under investigation is the oxidation of ethylene on a Pt catalyst, the reacting mixture consisting of ethylene at a partial pressure of 0.36 kPa and oxygen at a partial pressure of 4.6 kPa. With no current passing through the electrochemical cell, the reaction rate at the working electrode is 1.5 x 10^{-8} mol O/sec. Upon passing 1 μA through the cell so that oxide ions are transported to the working electrode (Pt), the reaction rate gradually increases, plateauing at 40 x 10^{-8} mol O/sec. The rate enhancement factor, namely the ratio of the maximum rate with the applied current to the rate without any current flow is 26. The difference between the maximum reaction rate at I=1 μA and at I=0 is 38.5 x 10^{-8} mol O/s. If the increase in reaction rate is solely due to oxidation by the oxygen transported via the applied current, the increase would be I/2 F where F is the Faraday constant. The ratio of the actual rate increase to that expected from Faraday's law is 74000. This ratio is called the NEMCA factor; its large value clearly indicates that the reaction rate enhancement is non-Faradaic. When the applied current is cut off, the reaction rate gradually decays to the original value before the application of current. The understanding of this phenomenon is in its infancy but could be far reaching in its impact.

Ceramic Oxygen Generators for Life Support

Electrochemical cells based on zirconia or ceria electrolytes are being developed for a variety of life support systems because they can readily produce a pure oxygen gas stream from ambient air. Compact ceramic oxygen generators are being developed to provide a continuous supply of oxygen-enriched air for people with breathing disorders. Similarly, these devices can enrich the breathing oxygen concentration for high altitude aircrafts.

More recently this technology has sparked the interest of NASA for space exploration. Because of the distance involved, if we are to travel to other planets in the future, we need to utilize their resources (*in-situ* resource utilization, or ISRU) for life support and propellant production. As early as 20 years from now, NASA plans on sending a manned mission to Mars. A conceptual picture of the Mars Ascent Vehicle is shown in Fig. 7. In order for this to be feasible, this mission will require the utilization of the Mars CO_2 atmosphere to produce the propellant for the return trip and oxygen, both for breathing and the fuel oxidant. The technology envisioned to make this possible is based on a ceramic oxygen generator converting CO_2 to O_2 and CO.

High Temperature Sensors

The most prevalent application of ion conducting ceramics is the automotive oxygen sensor. Every automobile sold in the U.S. since about 1975 (and in most of the industrialized nations sometime thereafter) has had its air/fuel ratio, and thus its gas mileage and emissions, controlled by this simple zirconia-based Nernstian device. Advances to this technology have focused on expanding the sensor range (using an amperometric

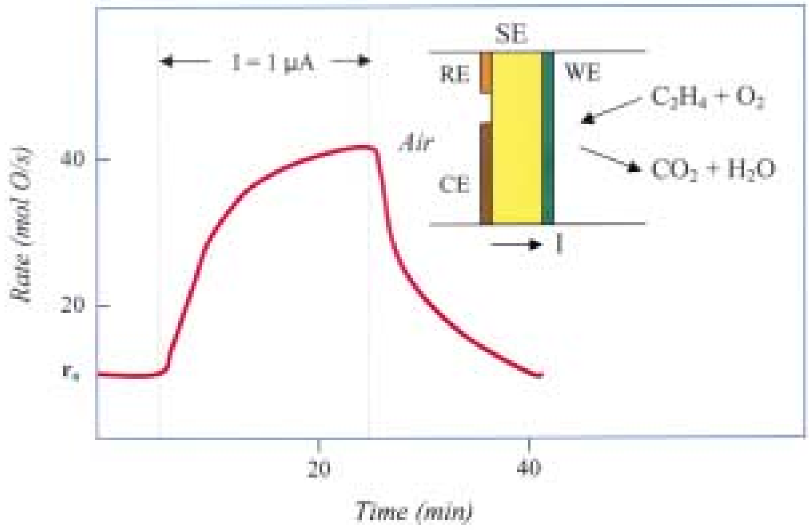


FIG. 6. NEMCA effect in ethylene oxidation. The designations, SE, RE, CE, and WE refer to the solid electrolyte, reference electrode, counter electrode and working electrode, respectively.

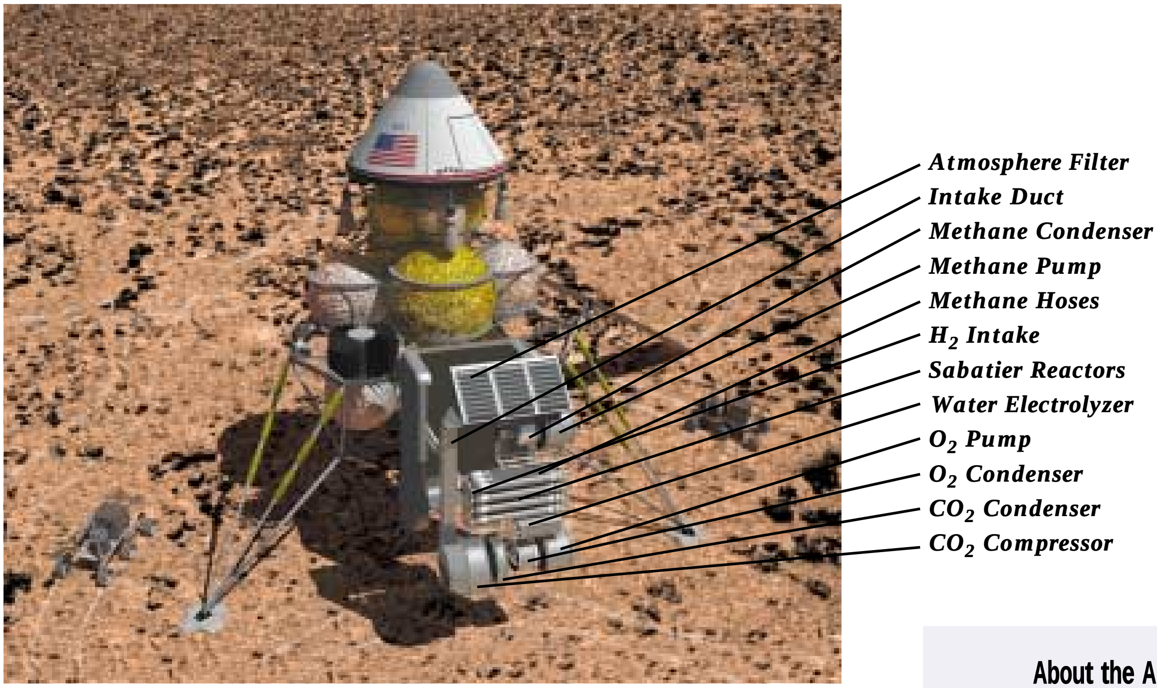


FIG. 7. *Mars Ascent Vehicle based on ISRU propellants.*

design) and enhancing the selectivity to specific gaseous species; the latter is critical for combustion control, in order to meet future environmental regulations. The issue is that ppm levels of pollutants such as NO_x and CO have to be monitored and controlled in concentrations of O_2, CO_2, and H_2O that are not only orders of magnitude higher than the species to be detected, but that also fluctuate dramatically under typical driving conditions. Modifications of the electrode materials and microstructures are being actively investigated to obtain the needed selective response.

Summary

Tremendous progress has been made in understanding the defect chemistry and electronic and ionic conduction in many high temperature ceramics over the last four decades. Such ceramics are now attracting increasing attention for a variety of applications, notable among these being fuel cells for power generation, production of chemicals, oxygen generation for life support systems, and high temperature electrochemical sensors. Many of these applications promise to become wide-spread commercial successes in the coming years as the cost is brought down through further technical advances and mass production. ■

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