

Oxygen Transport Membranes and their Role in CO2 Capture and Syngas Production

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Abstract

Membrane technology for gas separation has seen remarkable improvements in the last 20 years, particularly in the area of air separation for a cost-effective production of highly pure oxygen gas. It is rapidly paving way for alternate route to orthodox separation processes like cryogenic distillation. Solid-state electrochemical cells based on oxygen-ion conduction permit high temperature selective transport of O_2 in the form of ionic flux. Hence these systems can act as filters for molecular oxygen either for generation or separation of oxygen gas. The solar thermochemical conversion of CO_2 and H_2O into syngas is usually carried out at a high temperature of above 1500°C in repeated heating–cooling cycles with the help of durable metal oxide catalysts. Oxygen Transport Membranes (OTMs) are high density ceramic membranes which display mixed conductivity of oxygen ions and electrons and a two-phase mixed metal oxide OTM could thermo-chemically convert CO_2 and H_2O to syngas in a single step with an H_2 /CO ratio of 2:1; thus offering an alternative route for syngas production. OTMs also propose a favourable technology for oxy-fuel and CO_2 capture processes for gas and coal based power plants. Latest progresses in the field of ceramic membrane for oxygen separation from air at high temperatures vis-à-vis numerous materials and the prospect of ceramic-based membranes for the same are reviewed

Keywords: Synthesis gas; H_2O splitting; CO_2 decomposition; CO_2 capture; High temperature ceramic membranes

Abbreviations: OTM: Oxygen Transport Membrane; MIEC: Mixed Ionic Electronic Conducting; ASU: Air Separation Unit

Introduction

Oxygen is one of the most widely used chemical, ranked among the top five being produced in the world [1]. O_2 separation from the air is quite a large industry, with more than 100 million tons per year of production capability [2]. This market is expected to expand considerably due to the necessity of O_2 as feedstock for all sizable clean energy technologies [3]. Oxy-fuel combustion process and oxygen-blown gasification for conversion of natural gas and coal to syngas for subsequent electricity generation, and production of transportation fuels and other chemicals are but a few examples of such technologies [4].

Cryogenic and non-cryogenic distillations are the two orthodox technologies frequently used for the air separation. The second involves air separation at atmospheric temperatures using adsorbents based on molecular sieves through either Pressure Swing Adsorption (PSA) or membrane separation process. Lately, another class of air separation has evolved built around specific ceramic membranes that can effectively separate O_2 from air at higher temperatures. This new method is based on high density ceramic membranes which perform separation at temperatures between 800°C and 900°C. Mixed Ionic Electronic Conducting (MIEC) membranes, Ion Transport Membranes (ITM), and Oxygen Transport Membranes (OTM) are the acronyms used for these membranes.

A solid electrolyte membrane based on an oxygen-ion or mixed oxygen-ion and electronic conducting material is permeable only to oxygen (as ionic flux) at high temperatures, but not to other gases. Therefore, these membranes can be employed for making highly pure O_2 , enriching O_2 in atmospheric air, removing O_2 from a gas stream or an enclosure, and controlling O_2 in the gas stream at a preordained level. An oxygen-ion conducting ceramic membrane operating as a fuel cell can also be used for making syngas by the partial oxidation of

methane [5-7]. Methanol, produced from syngas, is considered to be a multipurpose fuel for use in fuel cells for transport, portable power and remote area stationary applications. OTMs also propose a favourable technology for oxy-fuel and $\rm CO_2$ capture processes for gas and coal based power plants.

Operating principle

Lately, ardent research efforts have been performed for advancement of O₂ separating ceramic-based membranes at hightemperature operations. These systems can be characterized as (i) pure oxygen conducting and (ii) mixed ionic-electronic conducting membranes. Pure O2 conducting membranes comprise of solid electrolytes (Figure 1 (a)) which are provided with electrodes for electron conduction. O₂ can be conveyed from one side of the membrane to another for storage as pure gas with the application of a voltage across the solid electrolyte cell consisting of an ion-conducting (O2-) electrolyte membrane coated with electrodes on both sides. These membranes are typically driven by either voltage or current and provide much better dominion over the level of O₂ produced or removed from a gas stream. The O₂ flux is directly related to the current flowing through the membrane and maximum attainable oxygen flux is determined by the applied voltage and electrical resistance, which ensuingly depends on the operating temperature, stability of the material, conductance of the ceramic membrane and the choice of electrode materials.

For oxygen enrichment in air, the oxygen produced can be mixed

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with air to achieve desired levels. Figure 2 describes the operating principle of a membrane system for removing or controlling oxygen levels in a gas stream. In the control loop feedback mode, for accurate control of oxygen in the gas stream, a signal is sent to the microprocessor to increase or decrease the electric current flowing through the oxygen separation device by the oxygen sensor placed downstream.

Second category belongs to the membrane which is a mixed conductor e.g., a two-phase mixture of oxygen-ion and electronic conducting materials or a single-phase material with mutual oxygen-ion and electronic conducting ability. The oxygen in the air combines with the electrons in the membrane to harvest negatively charged oxygenions. No electrodes or external circuits are required for this system to function and the driving force for oxygen-ion passage is provided by the temperature and pressure differentials. The pressure difference across the membrane may be provided by either a high pressure on the air side or a lower oxygen pressure in the compartment where O₂ is produced. The behaviour of electronic conductivity as an internal short circuit gives rise to the permeation of $\mathrm{O}^{2\text{-}}$ ions from higher to lower $\mathrm{O}_{\text{-}}$ partial pressure side, at the same time maintaining the overall charge neutrality by an offsetting flux of electrons (Figure 1 (b)). Such systems have promising prospects for large scale O₂ generation. The differential of partial pressure across the membrane can also be provided by maintaining air on one side and natural gas on the other side of the membrane. Partial oxidation reactors, using mixed ionic/electronic conductors, are ideal for the manufacture of syngas as a feedstock for liquid fuel production. Such a system would utilize the energy produced internally to be thermally and electrically self-sustaining or for cogeneration of both syngas and electricity. The overall efficiency of these systems can be optimized by the choice of materials, the operating temperature, fabrication techniques, the engineering of the device and the overall design.

Transport mechanism in OTMs

 $\rm O_2$ permeation through a nonporous MIEC material is restricted by bulk diffusion limitations, surface exchange resistance, or both with

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bulk diffusion being the controlling step in case of adequately thick membrane. Figure 3 depicts the mechanism for O_2 permeation through an MIEC membrane where the permeation process is distributed into three distinct zones: (i) an interfacial zone on air or the high partial pressure side; (ii) a central bulk zone; and (iii) an interfacial zone on sweep gas or the low oxygen partial pressure side. Membrane thickness and material are the two factors on which the performance of O_2 permeation flux is dependent. The permeation conditions like temperature, pressure and flow rate of sweep gas also play a vital role.

Ceramic membrane materials

Zirconia doped with different levels of Y_2O_3 possesses good oxygenion conductivity and hence is excellent material for the fabrication of ceramic membranes. Commercial powders of various compositions can easily be sintered to near theoretical density at temperatures in the 1400-1500°C range. These materials remain mainly oxygen-ion conductors over wider ranges of oxygen partial pressures and temperatures and are quite safe to use in a variety of applications [8]. Replacing Y_2O_3 with Sc_2O_3 has the effect of increasing the ionic conductivity. The sintered bodies of such materials can be manually handled with relative ease as the mechanical properties of most doped zirconia materials are adequate at both room temperature and at the cell operating temperatures [8]. These materials can be easily fabricated into various shapes (tubes, flat plates, etc.) and sizes depending on the design of the oxygen separation device. However, there is a limit on the thickness of the material, which can be produced for self-supporting membranes. A benefit of the flat





plate designs is high oxygen production or removal capacity per unit volume of the membrane but they require high temperature seals and exotic materials for interconnecting various cells and thus have low thermal cycling capability. The tubular designs require minimal sealing and the thermal cycling capability is expected to be significantly higher compared with the flat plate design. With the use of zirconia-based membranes, oxygen flux density of upto 1.5 cm³ min⁻¹ cm⁻² electrode/ electrolyte contact area can be easily achieved at temperatures around 750°C.

Materials like doped CeO_2 , and lanthanum gallates (LaGaO₃) are suitable for low temperature operation. In the CeO_2 system, the highest ionic conductivity has been observed for Sm_2O_3 and Gd_2O_3 dopants [8]. The only problem with these materials would be their reported low mechanical strength of around 150 MPa at room temperature, which would make the fabrication and handling of devices somewhat cumbersome [8].

Bi2O3 doped with metal oxides such as Y_2O_3 , Gd_2O_3 , Er_2O_3 , Dy_2O_3 , Nb_2O_5 , Ta_2O_5 , etc., becomes an oxygen-ion conductor with significantly higher conductivity than that of doped ZrO_2 or CeO_2 [8]. Another class of materials is from the BiMVOx family. These materials have high oxygen-ion conductivity [8] but most bismuth oxide based materials have extremely poor strength and can be easily damaged or eroded in low oxygen partial pressures and therefore are likely to have limited applications. For example, membrane devices that are constructed using these materials may be suitable for oxygen generation from air but are unlikely to be suitable for oxygen removal from low oxygen partial pressure gases. Such materials nevertheless can be fabricated into corrugated or honeycomb structures and are ideal for oxygen production in large quantities at lower operating temperatures.

Several mixed conducting materials having high oxygen-ion conductivity have been identified. Figure 4 shows the ideal structure of such materials, also known as perovskites. The most prominent materials are $SrC_{o}O_{3-\delta}$ doped at the A-site (for e.g., with La, Gd, Nd, Sm, Pr, etc.) and at the B-site (e.g., with Fe, Cr and Ti), $La_{1-x} Sr_x (Ga_{1-y}, M_y)$ (M=Fe,Co) $O_{3\pm\delta}$ and $Sr_4Fe_{6-x} Co_xO_{13\pm\delta}$ [9-11]. In these materials, O_2 permeation flux density relies on membrane thickness, bulk diffusion rates, temperature, gradient of O_2 partial pressure across the membrane and surface O_2 exchange rates. For air on one side and He on the other side of the membrane, O_2 permeation flux density of upto 1 cm³ min⁻¹ cm⁻² at 900°C and 2.5 cm³ min⁻¹ cm⁻² at 1000°C has been reported [9-11].



Applications

Oxygen separation has diverse applications including uses of O, for glass making, patients with chronic lung diseases, food processing, semiconductor manufacturing industry and the metallurgical industry (e.g., copper and steel production), combustion processes where higher efficiency and lower emissions are critical as with the for hydrocarbon fuels, cutting and welding, biological water purification, the petrochemical industry for converting natural gas to liquid fuels, clean waste incineration, aeration in aquariums and fish farming-oxygenation of fish-breeding tanks. The oxygen pumping devices based on oxygen-ion conducting ceramics can also act as filters for O₂, and therefore could be utilized for O₂ generation in outer space, or used in hermetically sealed transport carriers, or armoured vehicles to supply oxygen, which is free of toxic substances. Such oxygen separation devices can also be used to accurately control oxygen concentrations in a gas stream (by pumping oxygen in or out) when operated in a controlled loop feedback mode. Similarly, an oxygen-ion conducting membrane system can be used to supply nitrogen from air, or for gas purification, for example, removal of oxygen from an inert gas such as argon containing substantially higher amounts of oxygen. Other uses of oxygen-ion conducting membrane reactors include production of hydrogen by electrolysis of steam, NOx reduction, and production of chemicals.

The existing orthodox oxygen separation methods can be classified into cryogenic and non-cryogenic methods. In the cryogenic separation, air is purified, cooled to liquid form followed by subsequent distillation of O_2 from liquid air. Such plants are huge and expensive. On the other hand, pressure or vacuum swing adsorption methodologies are mainly employed in non-cryogenic methods for oxygen supply in which, molecular sieves are used to remove nitrogen from atmospheric air and O_2 is concentrated to 95-99%. The technology utilizing oxygenion conducting materials (ceramic membrane) has distinct advantages in that the physical (ceramic) barrier produces oxygen of extremely high purity by preventing any sort of contamination from entering the affluent gas stream. Moreover, similar devices can also be used for purification of gases containing oxygen, oxygen removal from gas streams and enclosures to extremely low levels and to control oxygen level in gas streams.

OTMs for syngas production

Syngas is an antecedent to liquid hydrocarbon fuels like methanol which is used in electrolyte membrane fuel cells. Syngas also serves as a rudimentary chemical for a huge number of applications in metallurgical and petrochemical industries and it also be used as a fuel to generate electricity. The orthodox processes for converting natural gas to syngas are based on partial oxidation or steam reforming or both. The main cost related partial oxidation is that of the O_2 plant. The endothermic nature of the reaction makes steam-reforming of the natural gas an energy intensive process. The oxygen-ion conducting ceramic membrane technology has distinct advantages over conventional oxygen generating technologies. The process combines O_2 separation from air with the partial oxidation of methane into a single reactor. Studies have indicated that such membrane reactors could be considerably smaller in size and 40% less expensive for O_2 generation/ removal than the existing technologies [5-7].

Converting water and carbon dioxide into energy intensive commodities by solar powered unnatural photosynthesis has become more attractive in past few decades, by virtue of its significant part in extenuation of energy deficiency and global warming [12-17]. Syngas can be produced from splitting of H₂O and CO₂ by means of solar thermochemical loop [18], having fast reaction kinetics and operating at higher temperature using the solar spectrum for thermal energy. A dual-step thermochemical cycle using metal oxide redox reactions [19] - where O₂ is separated from CO₂ or H₂O and stored in oxygen-short metal oxides like ceria or perovskites during the oxidation step [20] and is subsequently discharged amidst next stage at elevated temperature, resulting into the attainment of hydrogen/ carbon monoxide and O₂, in that order. Nonetheless, two-step thermochemical cycles require a very high temperature of about 1500°C during the endothermic step for reducing the metal oxides, necessitating solar reactors and metal oxides to be robust and durable throughout the recurring heating-cooling cycles. One likely solution to reduce the working temperature, is to eliminate the O₂ produced from the equilibrium-limited breakdown of CO₂ and H₂O with the help of a dense OTM having mixed oxygen ion and electron conductivity [12]. A recent study into thermal breakdown of CO₂ and H₂O for producing syngas in OTM reactor encompassed of concurrent splitting of CO₂ and H₂O in a single process leading to substantial lowering of energy loss as well as equipment investment in syngas production.

On site removal of O₂ produced from thermal CO₂ and H₂O splitting with an OTM, can overcome the thermodynamic equilibrium limit of the two breakdown reactions as depicted in Figure 5. As long as an O₂ partial pressure differential exists between the feed and permeate sides, the O2 generated during co-splitting of CO2 and H2O will be continuously removed. A very small partial pressure of O₂ on the permeate side can be established with the help of (i) a vacuum pumps, (ii) an inert sweep gas, or (iii) an O₂ consuming reaction such as selective methane oxidation. Since splitting of both CO2 and H2O are extremely endothermic, with heat demand exceeding the heat liberated during exothermic partial oxidation of methane, the required energy can originate from the sun, using an ordinary solar oven. Thus, syngas production at an accelerated rate could be anticipated in OTM reactor even at a lower temperature of less than 1000°C.

The H₂/CO ratio in syngas is a key factor for the downstream process, and it could be attuned by altering feed gas CO₂/H₂O ratio, as depicted in Figure 6. In typical situations, the change of Gibbs' free energy (ΔG) for carbon dioxide splitting is greater than water. Yet, the values of ΔG for mentioned reactions come closer to each other with increasing the temperature [21-23] and ΔG value for H₂O dissociation surpasses that of CO₂ splitting at temperature of around 830°C, which specifies that the equilibrium constant of carbon dioxide splitting is greater than that of



Figure 5: Simultaneous splitting of H2O and CO2 using OTM for Syngas production [21].

water splitting at elevated temperatures. Hence, to get a gas mixture of H_2/CO with a ratio of two, a feed of H_2O/CO_2 with a ratio of above two is required. It is worth mentioning that syngas is acquired uninterruptedly from carbon dioxide and water splitting at 930°C in an OTM reactor, and the working temperature is far lesser than ordinary thermochemical loop cycles.

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Expeditious exclusion of O2 produced in water/ CO2 breakdown is essential in order to augment the production rate of syngas. The driving force for O₂ conveyance is the pressure differential across the membrane, which can either be embedded by a very high vacuum or by using a reactive gas on the permeate side or by using a sweep gas. When methane is used as the sweep gas, it not only devours the permeated O₂ but also produces supplementary syngas $[CH_4+\frac{1}{2}O_2 \rightarrow CO+2H_2]$ with a H₂/CO ratio of two. The incessant O₂ conveyance can be upheld if permeate side $\mathrm{O_2}$ concentration is considerably lower than feed side.

OTMs for CO₂ Capture

As per the estimates of Intergovernmental Panel on Climate Change (IPCC), Carbon Capture and Storage (CCS) potential would be somewhere between 10-55% of the total carbon extenuation effort until the year 2100. CO, capture from power plants, particularly coal based power generation is the main focus of researchers these days. One of the most vital notions in this regard is the Integrated Gasification Combined Cycle (IGCC) for coal based power plants with CO, capture. Substantial energy penalty associated with CO₂ capture is the major snag which leads to abridged economic feasibility. Air Separation Unit (ASU), shift reactors, CO₂ capture unit and gas turbine that uses fuel rich in H₂ gas are the key sources of energy penalty in IGCC plants.

Typical energy consumption of modern cryogenic ASU's lies between 145-160 kWh/t O₂ [24-26]. OTMs which have the capability of separating O₂ from air with a 100% selectivity - a favourable substitute to O₂ production by cryogenic distillation, are likely to be used in oxyfuel and pre-combustion CO₂ capture processes for gas and coal power plants [27] where partial pressure differential between the O₂ rich feed side and the permeate side of the membrane is the main driving force for mass transfer. The feed stream is kept at a higher pressure than the permeate side and partial pressure of permeating O₂ is kept as low as possible by either reducing the permeate O₂ concentration or the permeate stream pressure. Figure 7 represents the two commonly utilized membrane operating methods.

Figure 7 (a) depicts an operating method where sweep gas is used and O₂ free stream is sent to the low pressure permeate side to reduce the concentration (and thus partial pressure) of O₂ on the permeate side; consequently, aggravating the driving force. The major benefit of using this method is that the feed and permeate side pressures could be set to virtually equal values, thus plummeting the pressure differential across the membrane which resultantly improves membrane's mechanical strength. Hence, the O₂ generated is at a higher pressure which ultimately reduces the compression work by the gasifier. Sweep gas at a temperature close to the operating temperature of the membrane should be used. In case of excessive heat in the sweep, preheating of feed stream with the sweep gas is performed to bring its temperature down to the membrane operating temperature.

Figure 7 (b) depicts the operating method which does not involve the use of sweep gas where permeate side pressure is set at vacuum or atmospheric settings so as to curtail O₂ partial pressure on permeate side [28]. This method involves no energy penalty with regards to steam extraction from the bottoming cycle for sweep gas. Nonetheless, low

pressure of produced O_2 leads to an energy penalty in the shape of compression of O_2 to the gasifier conditions. Hefty pressure differential between the permeate and feed side is yet another drawback of this method necessitating fabrication of such a membrane in which

mechanical failure could be avoided by meeting certain prerequisite. Preliminary scrutiny of the stated methods demonstrates that the one without a sweep gas yields promising cycle efficiency.

Figure 8 shows a two stage combustor with the OTM placed







between the two stages used to augment the process efficiency with 100% air integration. With respect of O_2 production requirement, only part of the air enters the OTM from the compressor exhaust. The overall plant performance shows overall plant efficiency of 37.3% with a CO₂ capture rate of 90.9%. This is because (i) preheating of OTM feed to 900°C does not require any extra fuel, and (ii) efficiency is not affected by the degree of air separation. A small capacity cryogenic ASU is also required for this process, which along with the GT hampers the flexibility and operability of the process.

Critical issues in OTMs and the way forward

Aside from substantial developments in the past decades, numerous concerns are yet to be addressed in the area of mixed conducting ceramic membranes. Significant concerns associated with high temperature O_2 separation from air using ceramic-based inorganic membranes for are highlighted in Figure 9. By altering various factors like membrane thickness, material, microstructure and membrane operating temperature, the O_2 permeation fluxes could be significantly enhanced. Membrane stability in mixed conducting ceramic membranes is one of the major issues because most membrane materials having immense ionic conductivity have poor chemical and/or mechanical stability and vice versa. Most ceramic membranes of perovskite-type are usually constructed from alkaline earth material having high O_2 permeation flux properties but on the same time having long-lasting problems with stability, because of their affinity for reactions with gas species such as sulfur dioxide, carbon dioxide or vapors of water [13].

Since O_2 permeation flux has an inverse relation with the membrane thickness, present focus of the manufacturers is on making tens of micrometers thick membranes. Common membrane designs comprise of asymmetric structures where thin, nonporous membranes are supported on a porous substrate providing the desired mechanical strength to the membrane. The porous substrate may be constructed from the same material as of membrane and apparently asymmetric membranes with thin nonporous layer and a porous support (Figure 10) could defeat these problems [14]. In most of the cases, porous support having a pore size lesser than skin layer thickness is preferred instead of the one having adequate mechanical strength. The support layer must have a chemical and thermal expansion similar to those of the skin layer and it must endure the membrane working temperature for a prolonged period of time without undergoing any structural or







chemical changes. α -Al₂O₃ is the widely chosen support material due to its low price, ample supply, and its effortless processing into consistent and extremely porous substrates however, it has poor expansion coefficient in comparison to most of O₂ permeable mixed-conducting perovskites which dictates its impracticality to be used as a support material.

Membrane surface alterations with an advanced surface exchange rate layer appears to be a rather attractive alternative due of its positive influence on surface kinetic reaction rate, thereby augmenting the O_2 permeation flux. Application of catalytic materials (e.g., Argentum) on membrane surface also increases the surface exchange reaction rate, thereby improving the O_2 permeation flux.

Meeting the requirements desired mechanical strength and stability of the membrane along with an appreciable O_2 permeation flux in single-phase membrane material is rather a difficult task. That is where the composite materials comprising of a dual-phase prepared from O_2 ionic and electronic conducting phases in the form of mixed conducting membrane materials come into play. Creation of a dual-phase MIEC membrane (Figure 11) with the dispersion of insoluble metallic or another ceramic phase having admirable electron conducting properties into copiously ionic conducting phase is an alluring preference vis-àvis O_2 separation is concerned. Aside from optimistic future prospects, further assessments of various amalgamations of materials other than metals are required to enhance the performance dual phase membranes as perovskite metal membranes are susceptible to defect formation which could compromise the purity of obtainable O_2 .

When compared to seasoned cryogenic air separation, separation based on ceramic membranes is quite a new technology as far as the production of O_2 from air is concerned for applications such as power generation and fuel production through the processes like oxy-fuel combustion, coal gasification and syngas production. Conspicuously, membrane materials are the core of membrane-based separation at high temperatures and they should be fully stable, robust, and skulk resistant having extraordinary O_2 flux properties. Consequently, improvements are anticipated in membrane performance for oxidizing and reducing environments necessitating development of rational projects under realworld operating conditions so as to obtain incredible O_2 production capacities for industrial consumption.

Membrane surface area has been under constant scrutiny because it increases with decrease in O_2 differential partial pressure; thus increasing the recovery rate of O_2 . Numerous abstract preferences

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including hollow fiber membranes [15], monolithic tubes [29-33], planar arrangements [34] and honeycomb structures [33-38] are available to fulfil the obligatory rudiments.

Conclusions

The commercial opportunities offered by ceramic membrane technologies are substantial. No single material or technology will fulfill such a diverse range of applications. The commercialization of these membranes has a bright future with regards to improved O_2 separation from air. The amalgamation of an OTM reactor with catalytic thermolysis and solar energy offers a ground-breaking itinerary for transforming water and CO_2 into syngas. An efficiency improvement of 0.7% with OTM integrated IGCC plant has some eloquence over the reference IGCC plant with cryogenic ASU with regards to future prospects. Other extremely significant issues like membrane module, geometry, and sealing must be addressed in order to enhance the performance of these membranes

Far-reaching research and development efforts are still required for commercial availability of MIEC membranes. Promising potential of ceramic-based membrane technology especially in the areas of fuel production, cleaner environment and sustainable energy delivery, anticipate their widespread future usage for separation of O, from air.

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