# Advances in Electrode Materials for AMTEC

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**Abstract.** An improved electrode for the Alkali Metal Thermal to Electric Converter (AMTEC) has been made and tested. The electrode is made from a slurry of metal and $TiO_2$ powders which is applied to the electrolyte and fired to sinter the electrode. During the first 48-72 hours of operation in a SETC, the electrode takes up Na from low pressure sodium vapor to make a metal-Na-Ti-O compound. This compound is electronically conducting and ionically conducting to sodium; electronic conduction is also provided by the metal in the electrode. With a mixed conducting electrode made from robust, low vapor pressure materials, the promise for improved performance and lifetime is high.

## INTRODUCTION

The Alkali Metal Thermal to Electric Converter (AMTEC) is a direct energy conversion device capable of near-Carnot efficiencies; in current versions of the device, the alkali metal used as the working fluid is sodium. The device is a sodium concentration cell which uses a ceramic, polycrystalline $\beta''$-alumina solid electrolyte (BASE) tube as a separator between a high pressure region containing liquid sodium at 900 - 1200 K and a low pressure region containing a condenser at 500 - 700 K. Sodium metal is oxidized at the liquid sodium/BASE interface and sodium ions are conducted through the BASE to the low pressure side of the BASE. Electrons travel through an external load to recombine with sodium ions at the interface between BASE and a thin film, porous metal cathode on the low pressure side of the BASE. Sodium vapor travels through the porous cathode, leaves the electrode as vapor and is collected on a cold condenser, from which it can be recirculated to the hot, high pressure side of the BASE (Weber, 1974; Cole, 1983).

NASA's interest in AMTEC is as the power source for outer planet exploratory spacecraft. For an AMTEC device to be feasible for space applications, it must have an operating lifetime of 7 - 15 years. Tests done in AMTEC research in this and other laboratories have shown that, on the time scales studied (up to 8000 hours), the cathode is the component most likely to influence device performance and limit operating lifetime. The anode will also degrade, but its performance is not as critical as that of the cathode. The ideal AMTEC cathode is one which has three primary characteristics:

1. It allows ready transport of sodium atoms from the electrode-electrolyte interface through the electrodes to the low pressure vapor space, for vaporization and transport to the condenser;
2. It offers good electronic conductivity for recombination of electrons and sodium ions; and
3. It is robust and refractory, leading to a lifetime which will allow continuous operation with minimal degradation in performance for seven (*e.g.* mission to Europa) to fifteen years (*e.g.* mission to Pluto).

Electrode materials tested at JPL in AMTEC and Sodium Exposure Test Cell (SETC) experiments have included refractory metal and metal alloy electrodes, including molybdenum, tungsten, rhodium-tungsten and platinum-tungsten, and titanium nitride electrodes (Wheeler, *et al.*, 1988; Williams, *et al.*, 1989; Williams, *et al.*, 1990b). Other researchers have studied metals, titanium nitride, titanium carbide, niobium nitride and niobium carbide as electrode materials and found them to be promising electrodes (McBride, 1987; Asakami, 1990; Fang, 1992; Nagata, 1993). JPL electrode studies have determined that rhodium-tungsten films co-sputtered approximately 0.7 - 1.0 μm thick are among the best performing electrodes, exhibiting power densities of ~0.5 $W/cm^2$ (Williams, *et al.*, 1989, 1990a). Current collectors made of molybdenum screen are overlaid on the electrode and tied on with molybdenum wires. The current collector must not impede sodium vapor flow as sodium leaves the electrode to condense on the cold surface; current collection networks have included wires spiraled over bus bars around the electrode (Weber, 1974), networks

made of individual wires or of metal meshes or screen (Wheeler, *et al.*, 1988; Williams, *et al.*, 1990a), and coarse felt or sponge (Tanaka, *et al.*, 1992).

AMTEC electrode studies have typically focused on three performance parameters, the sheet resistance in the electrode, the exchange current as measured by the interfacial impedance, and the resistance to sodium flow through the electrode and away from the interface. These parameters are reported as the sheet resistance $R_{sh}$ ($\Omega$/square) measured before and after an experiment, the temperature independent exchange current B with units ($A\cdot T^{1/2}/m\cdot Pa^{-1}$), and G (dimensionless) the morphology factor which expresses resistance to sodium transport (Williams, *et al.*, 1990a,b). In experiments at JPL, these parameters are measured in Sodium Exposure Test Cells (SETC) (Ryan, *et al.*, 1993, 1998), as well as in power-producing AMTEC cells.

The requirement of ready transport of sodium atoms through the electrode must be balanced with the requirement of efficient electronic conduction. Thickening the electrode to enhance electronic conductivity may result in greatly impeding transport through the electrode if transport is accomplished by sodium vapor flow or sodium diffusion through the electrode. An approach which will enhance conductivity while maintaining the ability for transport, or enhance transport while retaining conductivity, is desirable. In previous work in this laboratory, maximizing both conductivity and transport in a metal electrode has been addressed by making a thin film, porous electrode which includes a thicker, underlying grid for current collection (Ryan, *et al.*, 1995).

Because the site of electron/sodium ion recombination is apparently at the BASE/electrode interface, the ability of the electrode material to allow sodium atoms to pass through the electrode is critical to AMTEC performance. The primary mechanism for sodium transport is free molecular flow through pores in the electrode in mature molybdenum electrodes (Williams, *et al.*, 1986b), and an activated process such as grain boundary diffusion in rhodium tungsten and platinum tungsten electrodes (Williams, *et al.*, 1992). In either case, electrode thickness will affect the resistance to sodium transport.

The performance of an electrode can be improved if a material which is an ionic conductor to the alkali metal is incorporated in the electronically conducting electrode. A sodium ion conductor present in the cathode will transport sodium ions through the electrode, allowing the recombination of electrons and sodium ions to take place throughout the electrode. This process will reduce both electronic resistive losses and sodium transport losses in the electrode. The performance effect of the presence of an ionic conductor in the electrode is an increase in the magnitude of the exchange current and a decrease in the resistance to sodium transfer through the electrode; *i.e.* an increase in B by facilitating ion-electron recombination and removal of sodium ions from the interface, and a decrease in G by providing an additional mechanism for sodium transport through the electrode. This effect has been noted with the sodium ion conductors sodium molybdate and sodium tungstate early in life for electrodes containing molybdenum and tungsten, respectively (Williams, *et al.*, 1989). Sodium molybdate and sodium tungstate cannot be used to enhance electrode performance because the melting points of these compounds are within the range of AMTEC operating temperature, and the compounds evaporate in a few hours to days.

Pre- and post test analyses of titanium nitride electrodes have shown that in some TiN preparations, titanium-oxygen compounds are present. $TiO_2$ was identified in unoperated slurry electrodes through X-Ray Diffraction (XRD). These electrodes were as much as 20 $\mu$m thick, yet had better performance than might be expected for such a thick electrode. In an AMTEC or SETC experiment, the electrode is exposed to low pressure sodium vapor (~ 10 Pa) at 775 - 900 °C, and any $TiO_2$ in the electrode may react with Na to become a sodium-titanium-oxygen compound. Recent work in this and other laboratories has shown that $TiO_2$ will react with gaseous sodium at high temperature to form Na-Ti-O compounds which are both electronically and ionically conducting (Watanabe & Sekikawa, 1982; Colaitis, 1992; Ryan *et al.*, 1999). As it is possible that $TiO_2$ can be added to a metallic electrode to add ionic conductivity to the electrode, the conductive behavior of $TiO_2$ exposed to low pressure Na vapor and the performance of mixed metal and $TiO_2$ electrodes were studied.

# EXPERIMENTAL

A group of experiments was done to determine whether $TiO_2$ could be reacted with sodium vapor to form Na-Ti-O compounds under AMTEC operating conditions, to measure the conductivity of $TiO_2$ reacted in Na vapor, and to determine performance parameters of $TiO_2$ mixed with a metal used as an AMTEC electrode.

$TiO_2$ powder (anatase) and a piece of single crystal $TiO_2$ were loosely wrapped in Mo foil and placed in a stainless steel chamber with a Ti liner. The materials were heated to 850 °C for 100 hours. A pool of liquid sodium held at 300°C was at one end of the chamber, providing a sodium pressure of 3 Pa. On removal, the samples were analyzed by XRD and by gravimetric analysis.

To test the performance of mixed conducting electrodes for AMTEC applications, samples were made by mixing Mo and $TiO_2$ (anatase) powder 50 wt% each, or 54.5 at% Mo, with an organic cement in a slurry and applying it to a tubular section of $\beta$"-alumina solid electrolyte to make four electrodes. The samples were fired in vacuum for 2 hours at 1000 °C to remove the cement and sinter the electrode. Anatase transforms to the rutile structure at 915 °C; thus, after firing, the sintered $TiO_2$ was in the rutile phase. The electrodes formed from this process were 5 μm thick. Mo was chosen as the metal for the mixed electrode as the performance of Mo electrodes is well understood (Williams, *et al.*, 1986a,c).

The electrodes were contacted with Mo screen tied on with Mo wire and Mo wire leads, and operated in a SETC chamber in which the stainless steel chamber was lined with Mo foil to protect the BASE and electrodes from volatile elements in stainless steel (Ryan, *et al.*, 1994b). The SETC has been described in detail elsewhere (Ryan, *et al.*, 1993, 1998). Briefly, it is an evacuated chamber heated in a tube furnace. Inside, contacted electrodes on sections of BASE tube are mounted so they are electrically isolated from the chamber and the leads can be accessed from outside the chamber. Sodium pressure or, more precisely volume fraction of sodium, can be varied by controlling the temperature of a pool of liquid sodium at the cool end of the chamber, and electrode temperature controlled by the furnace hot zone. Two and four electrode measurements can be taken on the samples.

A sample to measure the conductivity of $TiO_2$ exposed to sodium vapor was made as a 5 μm thick coating of $TiO_2$ on an α-alumina tube. The coating was made by applying a slurry of $TiO_2$ powder (anatase) mixed with an organic cement to α-alumina. The sample was then fired in vacuum for 2 hours at 1000°C to remove the cement and sinter the $TiO_2$. The electrode was then contacted with four Mo wires. As noted above, the sintered $TiO_2$ was in the rutile structure. The four wires acted as electrodes to measure impedance to be used to calculate the conductivity of the Na-$TiO_2$ formed by exposure to Na vapor. The sample was mounted in the same chamber as the electrode experiment described above.

Four RhW electrodes sputtered on a section of BASE tube were operated in the SETC chamber in which the $TiO_2$ experiments were done. The iV and impedance characteristics of RhW electrodes are well understood (Williams, *et al.*, 1989); the electrodes were included for the purpose of direct performance comparison with the experimental electrodes.

The experiment was operated for 1500 hours at 700 - 850 °C in a sodium vapor atmosphere. Sodium pressure was varied from 0.01 to 10 Pa. Current-voltage curves and electrochemical impedance spectroscopy were taken at regular intervals on all sample sets using Solartron instruments. As described in earlier papers (Williams, *et al.*, 1990a,b; Ryan, *et al.*, 1998), these measurements are used to characterize electrodes by deriving B from the interfacial impedance and G from the limiting current.

# RESULTS AND DISCUSSION

$TiO_2$ powder which was exposed to low pressure sodium vapor at AMTEC operating temperatures was identified as $Na_{0.5}TiO_2$ by gravimetric analysis and XRD. The single crystal of $TiO_2$ which was included in the chamber degraded to powder during the Na exposure and was not analyzed (Ryan, *et al.*, 1999). Based on this early result, the experiments to measure conductivity and electrode performance were assembled. The conductivity ( σ, units S/cm) of the Na-Ti-O compound formed by exposing $TiO_2$ to sodium vapor at 600 - 850 °C was measured and found to be dependent on the sodium pressure as well as on the temperature. The rutile crystal structure is octahedral, and

the octahedra form chains in the bulk. Channels between the chains may intercalate atoms under appropriate conditions; it is likely that intercalation and deintercalation account for the reversible dependence of $Na_xTiO_2$ conductivity on sodium pressure. Na-Ti-O compounds are known to be electronically conducting (Colaitis, 1992); it was not possible in this work to separate ionic from electronic conductivity.

Over the initial 48 hours of heating at 550 °C, the conductivity of $TiO_2$ on $\alpha$-alumina rose steadily as the $TiO_2$ was exposed to sodium vapor. When the sample temperature was raised to ~745°C, the conductivity changed linearly with the pressure above the Na pool. There is an irreversible component of the conductivity for the $Na_x$-$TiO_2$ compound(s). $TiO_2$ samples removed from the furnace became conductive; this conductivity is electronic and is probably caused by substoichiometric oxygen content in the rutile structure. At AMTEC operating temperatures and pressures, the conductivity is dependent on both the temperature of $TiO_2$ and the sodium pressure (the saturated vapor pressure of sodium at the sodium pool temperature). $TiO_2$ is a semiconductor and will increase conductivity with temperature and it will lose oxygen in a reducing atmosphere to make a highly doped n-type semiconductor. Both of these phenomena contribute to electronic conductivity. The ionic conductivity may be expected to increase as $TiO_2$ temperature increases but the volume fraction of sodium does not. Intercalation of Na into rutile channels will increase as the kinetic energy of Na atoms increases, in effect an increased local pressure of sodium while the volume fraction of sodium remains the same. Moreover, when pressure above the Na pool increases, the volume fraction of Na increases, and there are more collisions of Na with the $TiO_2$ coating. Figure 1 shows the dependence of conductivity on Na pressure at 745°C; Table 1 tabulates conductivity at ~745°C and ~845°C at varying Na pressures.
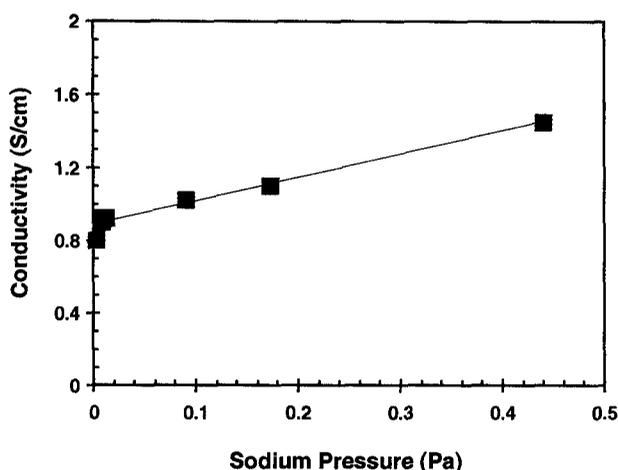


**FIGURE 1.** Dependence of conductivity of $Na_xTiO_2$ on Na Pressure. $Na_xTiO_2$ at ~745°C .

**TABLE 1.** Conductivity of $Na_xTiO_2$ at ~745 °C and ~845 °C as it changes with Na pressure. Ionic and electronic conductivity are not separated.

| T (°C) | $P_{Na}$ (Pa) | $\sigma$ (S/cm) | T (°C) | $P_{Na}$ (Pa) | $\sigma$ (S/cm) |
|---|---|---|---|---|---|
| 745 | .0031 | .80 | 841 | .022 | 1.50 |
| 743 | .0078 | .90 | 839 | .025 | 1.50 |
| 742 | .010 | .92 | 845 | .037 | 1.50 |
| 749 | .091 | 1.02 | 845 | .044 | 1.50 |
| 750 | .17 | 1.10 | 841 | .076 | 1.90 |
| 752 | .44 | 1.45 | | | |

Mixed conducting electrodes showed performance superior to that of electrodes made from Mo alone; both exchange current and sodium transport were improved in the mixed electrode. Performance improved dramatically in the first 20 hours of operation, as the $TiO_2$ in the electrode reacted with Na vapor to form the Na-Ti-O compound. Over the following 1500 hours of operation, the performance of the electrode was not found to change significantly.

The performance of thin Mo electrodes is well known. For a Mo electrode operated < 500 hours at 845 °C, G has been measured to be 20-30 (Williams, et al, 1986b, 1990a), while G for mixed $Mo/TiO_2$ electrodes operated in this experiment is similar to that of the RhW electrodes, 5-10. The mixed electrodes are substantially thicker (~5 μm) than thin film Mo electrodes (~1 μm). G would be expected to be larger on thicker electrodes as the transport path for sodium atoms from the interface to the outer surface of the electrode is longer and more tortuous; however, G is smaller in the mixed electrodes, indicating enhanced sodium transport.

Similarly, B for a thin film Mo electrode has been measured to be ~ 80 after a few days of operation. B for the mixed electrode was similar to that of the control RhW electrodes, ~ 100. B is a parameter which is derived from the charge transfer resistance for the electrode/electrolyte. Increased B indicates either larger reaction area or more efficient transfer of ions away from the interface.

Figure 2 shows an iV curve of RhW and Mo/ TiO$_2$ electrodes taken after 909 hours at high temperature (T>750°C). The slope of the curves through the origin and the limiting currents for both electrode sets are similar. A thin film Mo electrode would have a curve with a smaller slope (lower exchange current) and a smaller limiting current (larger G) than is seen in this plot. The redox waves seen at 1.2 - 1.5 V in the mixed electrode indicate the intercalation and deintercalation of sodium atoms in the rutile channels of TiO$_2$. This feature has been noted in earlier experiments with TiN electrodes (Ryan et al., 1994a). At the time, the redox waves were attributed to reaction with oxygen which had leaked in to the cell. That is a likely scenario for the presence of the waves, but these experiments show that the reaction with oxygen was in forming Ti-O compounds which was followed by reaction with sodium. The absence of redox waves or anomalous behavior in the RhW electrodes confirms that there was not an oxygen leak into the cell.
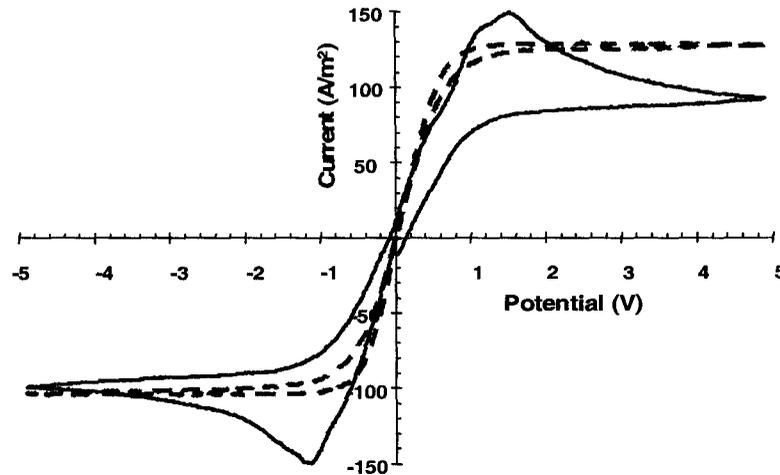


**FIGURE 2.** Current vs. Voltage curve of RhW (dashed line) and Mo/TiO$_2$ (solid line) electrodes. Electrode temperature - 841 °C; P$_{Na}$ = .022 Pa (liquid sodium at 192 °C); electrodes above 740°C 909 hours. Conductivity of Na$_x$TiO$_2$  σ = 1.5 S/cm.

## CONCLUSIONS

TiO$_2$ exposed to low pressure Na vapor at temperatures above 550 °C  will take up Na, resulting in a material Na$_x$TiO$_2$ which is both ionically and electronically conducting. The compound which was formed and remained after cooling and removing the sodium atmosphere was identified as Na$_5$TiO$_2$. TiO$_2$ will lose some oxygen in a reducing atmosphere such as Na vapor, increasing the electronic conductivity of the semiconducting TiO$_2$; however, the conductivity measured here is attributed to both electronic and ionic conduction, as evidenced by the enhanced sodium transport in Mo/TiO$_2$ electrodes on β"-alumina electrolyte. Conductivity of Na$_x$TiO$_2$ is a function of sodium pressure in the cell after the initial reaction; the unchanging portion of the conductivity is ~ 1 S/cm. Changes in conductivity induced by change in P$_{Na}$ are reversible. Na$_x$TiO$_2$ conductivity at a given temperature and P$_{Na}$ does not change over 1500 hours of operation above 800°C.

The performance of a thick film mixed conducting electrode made from Mo and TiO$_2$ is substantially better than that of a thin film Mo electrode. This improved performance is attributed to the action of the TiO$_2$, both as an ionic conductor to Na, improving sodium transport through the electrode (decreased G), and as an electronic conductor where electrode resistance is decreased. The Mo/Na$_x$TiO$_2$ electrode is robust and does not degrade in performance over 1500 hours of operation. Mo alone degrades owing to grain coalescence and decreased electrode area and conductivity.

Mixed conducting electrodes made from mixed metal and TiO$_2$ can be used to lengthen the life of an AMTEC electrode and to improve performance over TiN or other electrodes. Its performance is equivalent to that of RhW,

the best performing electrode material found. Metal mixed with $TiO_2$ can be applied as a paint or slurry and used as an inexpensive, robust, high performance AMTEC electrode. The $Mo/TiO_2$ ratio of 50 wt% for each compound was an arbitrary ratio selected for these experiments. Future work will include studies of both $Mo/TiO_2$ ratio to optimize performance and of other metals to replace Mo.

# ACKNOWLEDGMENTS

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