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Review of Refractory Materials for Alkali Metal Thermal-to-Electric Conversion Cells

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Refractory alloys are being considered as structural materials in multitube, vapor-anode alkali metal thermal-to-electric conversion (AMTEC) cells for future use in radioisotope space electric power systems. In these power systems, the AMTEC cells would operate at a heat source temperature of ~ 1150 K and a radiator temperature of ~ 550 K, for a 7–15 year mission lifetime. In addition to high strength, low density, and low brittle-to-ductile transition temperature, suitable materials must be compatible with the sodium working fluid and have low thermal expansion and low vapor pressure (<133 nPa). Refractory metals and alloys are reviewed for their potential use as structural materials in vapor-anode AMTEC cells. Results indicate that Nb–1Zr (niobium–1% zirconium) is a suitable choice, particularly for the hot-end structures of the cell (>900 K). C-103 (niobium–10% hafnium–1% titanium–0.5% zirconium) is also suitable, particularly for the cell's colder structure because of its higher strength and lower thermal conductivity. However, the compatibility of these niobium alloys with sodium at typical operating temperatures and in the presence of minute amounts of oxygen (>5–10 ppm) for up to 15 years needs further evaluation. Despite the limited availability of rhenium, Mo–Re alloys, with a rhenium content of 14–45%, are also good choices as structural materials in vapor anode AMTEC cells. However, their relatively higher density and thermal conductivity could lower the cell's performance and increase its specific mass.

Introduction

VAPOR-ANODE, multitube, alkali metal thermal-to-electric conversion (AMTEC) cells have been under development for more than 10 years. They have the potential for use in radioisotope power systems (RPSs) for future space exploration missions.[1] This technology, which also shows promise for terrestrial electric power generation in remote areas, offers several benefits over other static energy conversion devices. AMTEC cells operate at a moderate heat-source temperature (1150–1200 K) and at a relatively high condenser temperature (~600 K). These cells have demonstrated conversion efficiencies as high as 15% (Refs. 2–4), with up to 30% conversion efficiency being theoretically possible. In comparison, at the same hot- and cold-side temperatures, the state-of-the art SiGe/GaP thermoelectrics have typical conversion efficiencies of 5–7%. Therefore, replacing the SiGe/GaP thermoelectrics with AMTEC cells in an RPS can more than halve the amount of $^{238}PuO_2$ needed, in the form of standard general purpose heat source (GPHS) modules (or bricks), to meet a mission's electric power requirement.

Numerous multitube, vapor-anode AMTEC cells of the Pluto-Express (PX) series have been tested in vacuum from a few months to more than 2 years without failure. Test results have demonstrated conversion efficiencies of up to 15% and electric power output of 3–5 W per cell at a load voltage of 2.5–3 V (Refs. 2–4). The PX series AMTEC cells used sodium as the working fluid and operated at a hot-side temperature of 1100–1173 K and a cold side, or heat rejection temperature of 550–630 K (Refs. 5 and 6). These all stainless-steel structure cells have shown a rapid initial drop off in performance, followed by gradual degradation that amounted, in some cells, to more than 50%. A schematic of a typical PX-type cell is provided as Fig. 1.

This decrease in cell performance has been partially attributed to the degradation of the cathode electrodes of the beta″-alumina solid electrolyte (BASE) tubes [composition $Na_2O(Li_2O)_{0.176}(Al_2O_3)_{6.21}$]. Similar degradation with operation time has been reported in single effect electrode tests performed at the Jet Propulsion Laboratory (JPL).[7] Other potential causes for the degradation in PX-cell performance include potential contamination of the cathode electrodes and the BASE by volatile alloying elements, such as manganese and chromium, from the stainless-steel structure. Posttest examination of some of the PX series cells that failed early in vacuum testing have revealed massive material transport and deposition. Large globules of materials have been detected on the casing of the liquid sodium return artery. Similar material deposition has also been noted on the inside of the cell wall near the condenser. So far, a detailed evaluation of the composition of these deposits has not been performed, nor have potential material transport mechanisms been investigated.

As a replacement for stainless steel, El-Genk et al.[6] have investigated the performance of PX-type cells with a nickel hot-end structure and Haynes-25 at the cell cold end. Although the predicted cell performance was up to 30% higher than a similar stainless-steel PX-type cell, several lifetime issues were raised. One of these issues is that the vapor pressure of nickel at the typical hot-side temperature of 1200 K is higher than 10^{-9} torr (133 nPa). A vapor pressure of 10^{-9} torr has been recommended by JPL as a practical design limit to ensure minimal mass loss and transport within the cell during its operation lifetime of 7–15 years.[8] Although this vapor pressure limit may eventually be proven to be quite conservative, it is nonetheless a recommended rule of thumb until actual data on volatilization and transport become available.

In addition to the potential degradation of the electrodes and the BASE, other important lifetime issues are those related to the compatibility of the structural material with the sodium working fluid. These include corrosion of the cell wall and gradual loss of oxygen from the BASE tubes and the BASE ceramic/metal braze (Fig. 1). Volatile alloying elements or impurities in the cell wall could be released over time and deposited at other parts of the cell, such as on the cathode electrode or in the condenser wick structure. Such material transport and deposition could reduce the permeability, the average pore size (or porosity), and the wetability of the porous structures in the condenser, the liquid sodium return artery, and the evaporator. The resulting increase in internal pressure and polarization losses in the cathode electrode would degrade cell performance over time.

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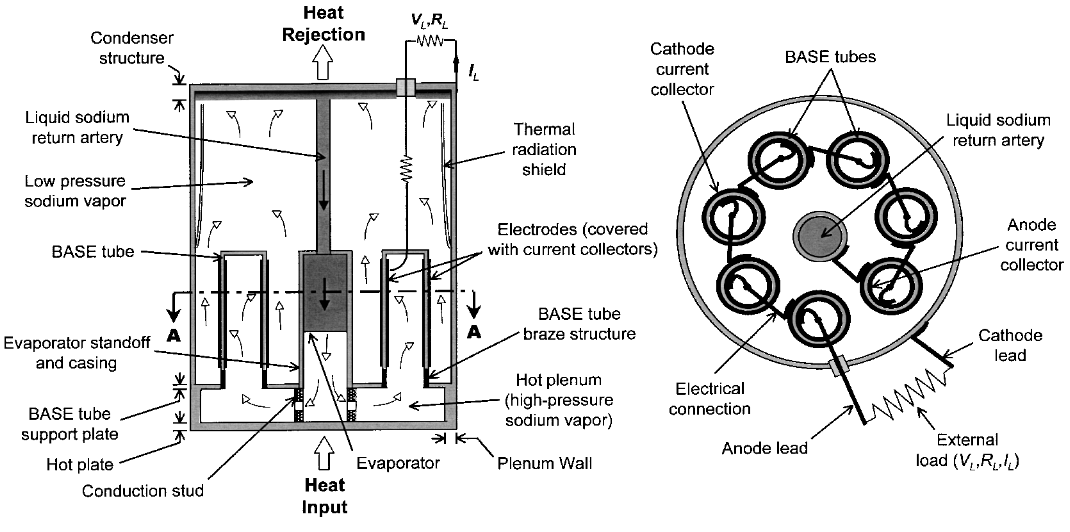

Side View

Plan View (Section A-A)

Fig. 1 Cutaway and plan views of a PX-type multitube, vapor-anode AMTEC cell.

Several adverse contaminant/BASE reactions have been documented.[9,10] Volatile alloying elements in stainless steel could react with the BASE to form a metal oxide and alumina (Al_2O_3). For example, chromium reacts with BASE to form a thin surface film of Cr_2O_3 and Al_2O_3 (Ref. 9) impairing the passage of sodium ions into the BASE solid electrolyte and, hence, reducing the cell performance. Manganese has also been shown to react with BASE material to form $MnAl_2O_3$ and probably Al_2O_3 (Ref. 10). These reaction products are also likely to increase the ionic resistance of the BASE, impairing the cell performance. In addition to its high vapor pressure, potential reaction of nickel with the BASE material is also a concern, which is worthy of future investigation.

A number of potential reactions of the contaminants and alloying elements in stainless steel with the BASE electrodes are noted by Ryan et al.[11] For example, manganese and chromium react with and degrade the titanium nitride (TiN) electrodes.[7] Nickel in contact with tungsten/rhodium (W/Rh) electrodes has been shown to cause rhodium diffusion toward the nickel and to increase sintering and void formation, reducing the performance of the electrodes.[11] Manganese in contact with W/Rh electrodes accelerates grain growth, which also decreases the performance of the electrodes.[7]

A concern with the PX-type stainless-steel and nickel/Haynes-25 cells[5,6] is the accelerated corrosion of the cell structure by the sodium working fluid. Alkali metal corrosion of stainless-steel and superalloy materials typically includes preferential dissolution of nickel and chromium.[12] Therefore, alloys with a greater fraction of nickel tend to exhibit a higher corrosion rate in liquid sodium. As shown in Fig. 2, increasing the nickel content of an alloy increases its corrosion rate in sodium, but decreases the accelerating effect of oxygen. To ensure long operation lifetime, recent attention has been focused on the use of refractory metals and alloys as structural materials in PX-type AMTEC cells. Advanced Modular Power Systems, Inc. (AMPS) has replaced stainless steel with Nb–1Zr (niobium–1% zirconium).[13] Incoloy 909, a low thermal-conductivity superalloy with thermal expansion similar to Nb–1Zr, was considered for the colder part of the cell wall to reduce parasitic heat conduction to the condenser. Preliminary tests performed at the Mound facility[14] revealed significant difficulties in welding Incoloy and Nb–1Zr and, as a result, the first generation refractory cells have used only Nb–1Zr (Refs. 15 and 16). The reported cell has more than twice the mass of a PX-type stainless-steel cell of the same design (>350 g) and a projected efficiency of 15.7% and electric power output of 6.1 W, at a load voltage of 3.5 V. King and El-Genk[17] have investigated the performance of a Nb–1Zr/C103, PX-type cell that weighed only 164 g and had a projected efficiency of 18% and electric power output of 8 W at a load voltage of 3.5 V. Other refractory alloys were also considered for use as AMTEC structural materials, but to date, only Nb–1Zr cells have been designed, fabricated, and are currently being tested at AMPS. A concern has recently been raised related to potential oxygen pick up and embrittlement of Nb–1Zr during the integration of refractory cells to GPHS bricks, if the assembly is done in the inert atmosphere assembly chamber at Mound.[18]

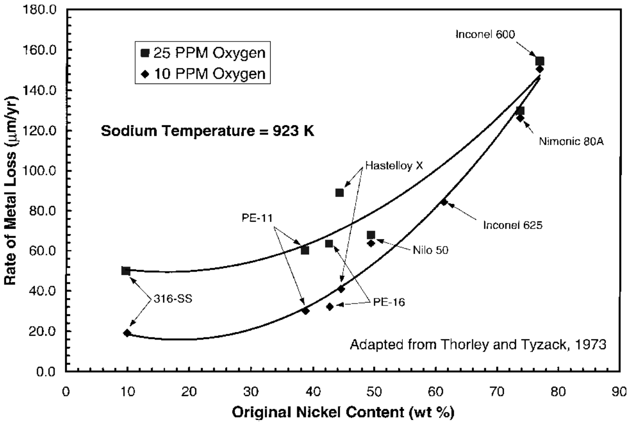

Fig. 2 Effect of nickel content on the corrosion of several alloys in liquid sodium (Adapted from Thorley and Tyzack).

Modeling of PX-type, multitube, vapor anode AMTEC cells (Fig. 1) has shown that the performance of the cells depends strongly on the amounts of heat lost by conduction to the cell condenser and by radiation to the cell wall.[19,20] To reduce these parasitic heat losses, a cell structural material that has a low thermal conductivity and low emissivity is desirable, particularly in the colder part of the cell wall near the condenser (Fig. 1). Because the thermal conductivity of the refractory materials of interest is much higher than that of stainless steel, conduction losses in refractory cells could be significant. The emissivities of the refractory metals and alloys of interest are typically lower than for nickel and stainless steel, partially offsetting the effect of the higher thermal conductivity of these materials on the parasitic heat losses in the cell. In addition, to minimize potential material transport and induced thermal stresses, the cell structural material should have low vapor pressure and small thermal expansion at the cell's typical operating temperatures.

Although structural materials that ideally meet the operation requirements for vapor-anode AMTEC cells might not exist, there is a need to examine the existing database for refractory metals and alloys and to identify suitable candidate materials. Refractory metals have been extensively studied for space nuclear reactor power applications during the system(s) for Nuclear-Auxiliary Power (SNAP) and Space Power-100 kwe (SP-100) programs in the 1960s and 1980s, respectively.[21–23] Some of the reactor concepts in these programs used liquid-metal coolants.[24] PWC-11 (niobium-1% zirconium-0.5% carbon), developed in these programs, was selected as a structural material in the SP-100 nuclear reactor power system, which was cooled with liquid lithium.[25] Other reactor coolants considered in these programs include liquid sodium, potassium, and Na–K alloy. Na–K and potassium were also considered as working fluids in ground tests of alkali metal Rankine cycles for potential use in space nuclear reactor power applications.[26] Because of the lack of an ongoing effort to develop suitable alloys for use in vapor-anode AMTEC cells in space power applications and to investigate systematically various corrosion and material degradation mechanisms, the selections of AMTEC structural materials have been based solely on information reported in the literature.

The objectives of this paper are to review the literature on refractory metals and alloys and identify potential structural materials for use in PX-type, vapor-anode, multitube AMTEC cells that are likely to meet the operation and lifetime requirements of future space missions. The screening criteria used are based on either recommended rules of thumb or desirable properties. These properties are 1) low–moderate thermal conductivity; 2) low density, vapor pressure, surface emissivity, and ductile-to-brittle transition temperature; 3) high yield and rupture strengths along with high strength-to-density ratios; 4) low thermal expansion; and 5) good compatibility with sodium at typical operating temperatures and sodium vapor pressures. Because of the large variations in the reported data from different sources, particularly those on corrosion and solubility in liquid metals, and the sensitivity of the experimental results to the presence of impurities and variations in testing conditions, the present ranking is largely qualitative in nature. The next section presents a brief description of a PX-type AMTEC cell.

PX-Type AMTEC Cells

A typical PX-type AMTEC cell consists of two enclosed pressure cavities separated by 5–9 BASE tubes, the liquid sodium return artery and the evaporator standoff, and the BASE support plate (Fig. 1). In the inner cavity, the pressure of sodium vapor is $\sim$60 KPa, whereas in the outer cavity, the sodium vapor pressure is $\sim$20–60 Pa. The hot plate and the plenum wall enclose the inner cavity, whereas the condenser structure and the cell wall (see Fig. 1) enclose the outer cavity.

The BASE material has high ionic conductivity whereas its electron conductivity is very low. An electric potential develops across the BASE tubes as sodium ions flow through the BASE from the anode (high sodium pressure) to the cathode (low-pressure) side. This electric potential is directly proportional to the logarithm of the sodium pressure ratio across the BASE tubes.

The BASE tubes are connected electrically in series to increase the terminal voltage of the cell. In early PX-type cells, the cylindrical wall that encloses the outer cavity was made of stainless steel. Recent cells use either Nb–1Zr (Refs. 15 and 16) or Nb–1Zr and C-103 for the hot- and cold-cell structures, respectively.[17] The metal conduction studs (Fig. 1) enhance the heat transfer from the heat source (or the cell's hot plate) to the BASE tube support plate and in turn to the BASE tubes and the evaporator. The BASE braze structure readily conducts heat from support plate to the BASE tubes, but electrically insulates them from each other. During operation, the temperature of the far end of the BASE tubes is maintained at least 20 K above the evaporator's temperature to prevent sodium from condensing inside the BASE tubes and potentially shorting them.

The hot part of the cell wall, facing the BASE tubes, is typically $\sim$200 μm thick. Also, because its temperature is close that of the hot plate, it minimizes heat losses by thermal radiation from the BASE tubes to this part of the wall. The colder portion of the cell wall, facing the liquid sodium return artery above the BASE tubes, is only $\sim$100 mm thick to reduce parasitic heat losses by conduction to the condenser. To reduce the parasitic heat losses further, the cell condenser is remotely located, and a low surface-emissivity thermal-radiation shield is installed on the inside of the cell wall above the BASE tubes. The low surface emissivity (or high reflectivity) of the thermal-radiation shield and the liquid sodium film covering the condenser surface reduce heat losses by thermal radiation from the BASE tubes to the cell wall and the condenser, respectively. Additionally, in an RPS the cell would be thermally insulated on the outside using low-conductance multifoil and molded Min-K insulation[5,27,28] to reduce the parasitic heat losses further. The following section reviews and screens refractory metals for potential use as structural materials in vapor-anode AMTEC cells.

Refractory Metals

The term refractory metals refers to the 12 metallic elements with melting points equal to or greater than that of chromium.[29] These elements are shown as a subset of the periodic table in Figs. 3a and 3b (Ref. 30). The area of the buttons in Figs. 3 is proportional to the density (Fig. 3a) and the melting point of the elements (Fig. 3b). The melting point of technetium (2430 K) technically qualifies it as a refractory metal; however, it is a manufactured, radioactive element and, hence, has been omitted from consideration. Of the 12 refractory metals, only the 6 elements in groups 5 and 6 [vanadium (V), niobium (Nb), tantalum (Ta), chromium (Cr), molybdenum (Mo), and tungsten (W)] are usually considered as alloy base metals. However, because of its high volatility, chromium has also been excluded. Five of the remaining elements [hafnium (Hf), ruthenium (Ru), osmium (Os), rhodium (Rh), and iridium (Ir)] are sufficiently rare that they are usually used as alloying additives, coatings, or catalysts in specialized applications. Although rhenium (Re) is relatively rare, it has been used for a variety of special applications in recent years.[31] It is extremely heavy and hard to work and, thus, is considered only as an alloying additive.

Historically, element number 41 (niobium), along with the naturally occurring niobium/tantalum alloy, has been referred to as

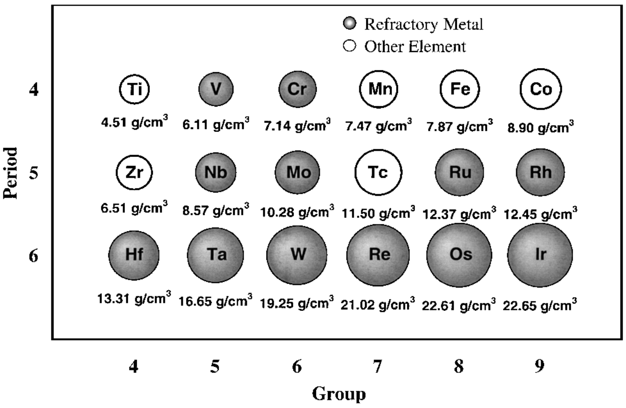

Fig. 3a Refractory metal densities (data from Winter[30]).

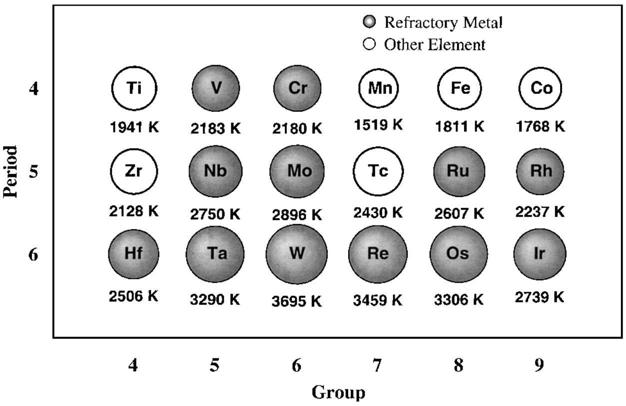

Fig. 3b Refractory metal melting points (data from Winter[30]).

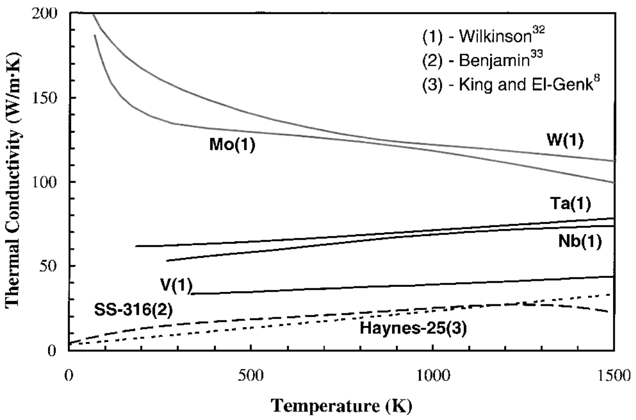

Fig. 4 Thermal conductivity of the refractory metals.

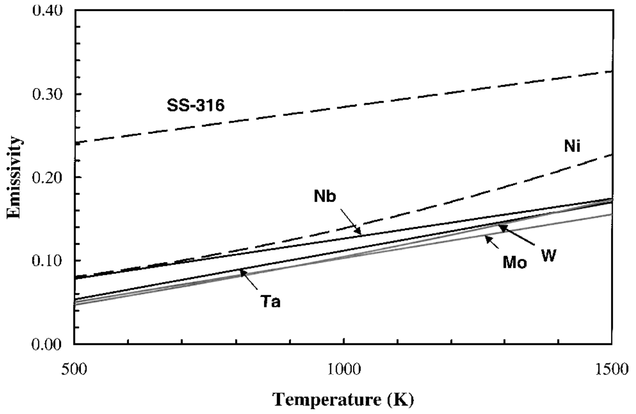

Fig. 5 Thermal-radiative emissivity of the refractory metals (data from Touloukian et al.[34]).

columbium (Cb). In accordance with the current International Union for Pure and Applied Chemistry (IUPAC) standards, element number 41 is designated niobium for the remainder of this work. The term refractory metal refers to the pure refractory elements, and the term refractory alloy indicates an alloy of the refractory metals.

Figure 4 presents the temperature dependent thermal conductivities of the candidate refractory metals.[8,32,33] Molybdenum and tungsten have the highest thermal conductivities, which pose challenges to reducing the parasitic conduction heat losses in the AMTEC cells. The thermal conductivity of niobium and tantalum are also relatively high. Of the refractory metals shown in Fig. 4, only vanadium has a thermal conductivity that is only slightly higher than that of the stainless steel used in the laboratory-tested PX-type AMTEC cells. Based on thermal-conductivity considerations alone, vanadium would be the first choice, followed by niobium and tantalum.

As shown in Fig. 5, all of the elements in groups 5 and 6 have similar surface emissivities that are lower than those of stainless steel and nickel.[34] The group 6 elements have slightly lower emissivities than the group 5 elements. Data on the emissivity of vanadium could not be found; it may fall between those of nickel and niobium. Based on the small variations in the reported emissivities, no clear distinction can be made between the candidate elements based on the emissivity.

Figure 6a compares the percent linear thermal expansion of the refractory metals of interest vs temperature.[34] The corresponding linear thermal-expansion coefficients are plotted in Fig. 6b. Tungsten and molybdenum have the lowest thermal expansion, followed by tantalum. Niobium and vanadium have higher thermal expansions, but still expand less than nickel and stainless steel. A low thermal expansion is a desirable property for an AMTEC structural material. However, because the refractory metals thermally expand less than stainless steel, they are all acceptable based on this criterion.

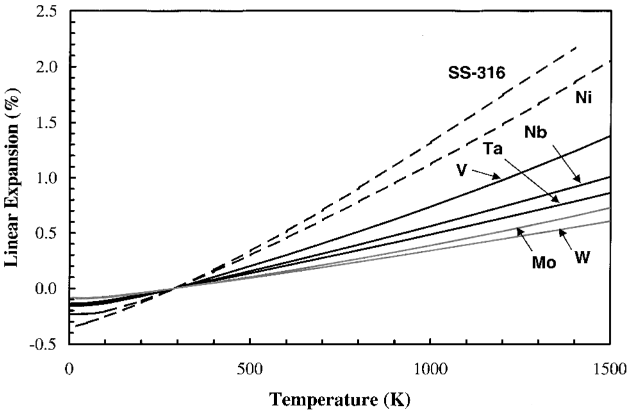

Fig. 6a Percent linear thermal expansion of the refractory metals (data from Touloukian et al.[34]).

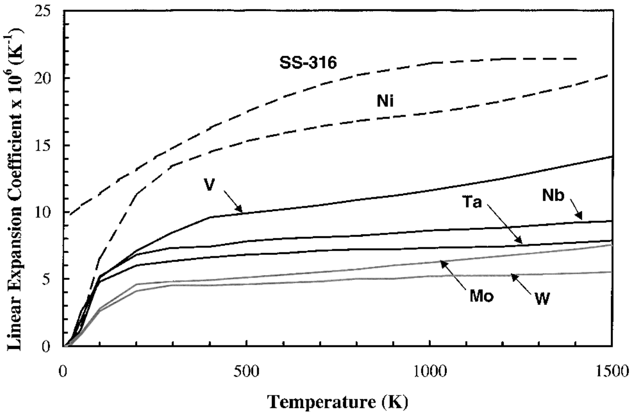

Fig. 6b Linear thermal-expansion coefficients of the refractory metals (data from Touloukian et al.[34]).

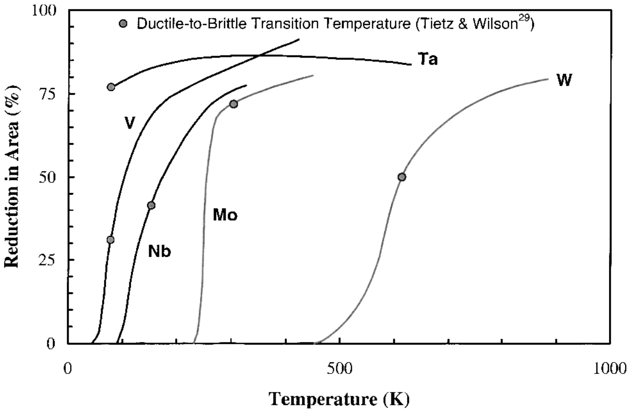

Fig. 7 Ductility of the refractory metals (adapted from Wilkinson[32]).

Although it is not strictly a structural property, the ductility of the refractory metals, particularly at room temperature, is an important consideration for their use as structural materials in vapor-anode AMTEC cells. Unfortunately, many of the refractory metals (particularly the group 6 elements) are not ductile at room temperature.[32] Although brittle materials could be manufactured and handled safely, a structural material that is ductile at all temperatures of interest will greatly simplify the fabrication and handling of the AMTEC cells. Figure 7 presents two measures of the ductility of the refractory metals, namely, the reduction in area at fracture and the ductile-to-brittle transition temperature. Note that the values of these parameters are very dependent on the purity of the element, the fabrication techniques used, and the handling of the material. All group 5 elements (V, Nb, and Ta) are ductile at all temperatures of interest. The ductile-to-brittle transition of molybdenum occurs at or near room temperature. Much work has

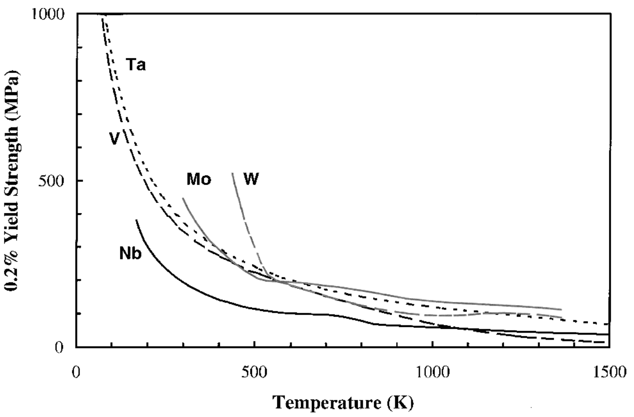

Fig. 8 Yield strength of the refractory metals (adapted from Tietz and Wilson[29]).

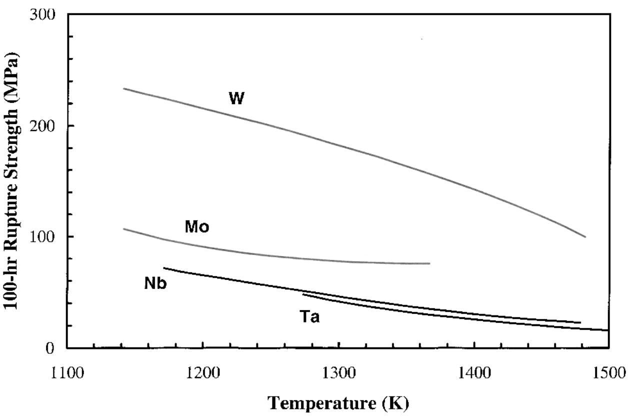

Fig. 10 Refractory metals 100-h rupture strength (adapted from Tietz and Wilson[29]).

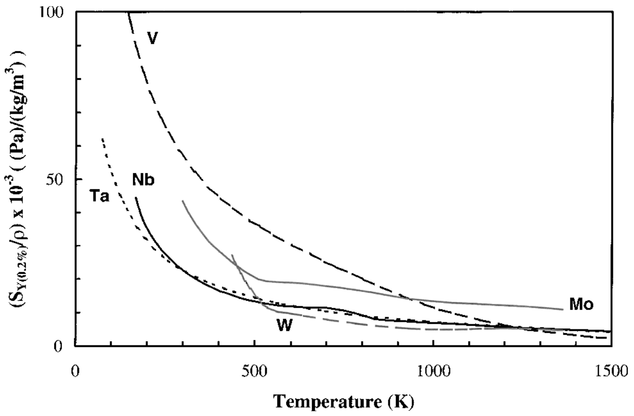

Fig. 9 Yield strength to density ratio of the refractory metals.

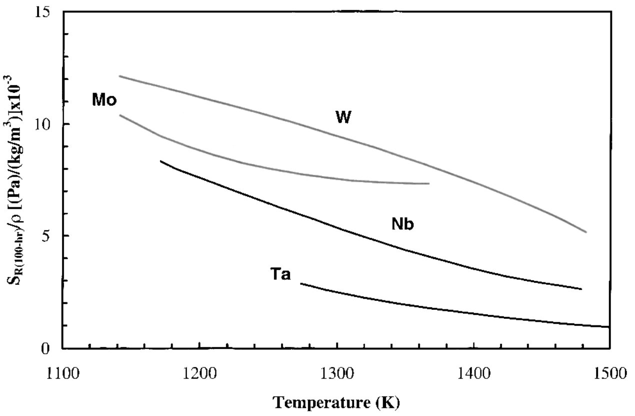

Fig. 11 Rupture strength to density ratio of the refractory metals.

been done, however, to produce ductile molybdenum alloys (such as Mo–TZM and Mo–Re) (Ref. 35). It has also been shown that highly pure, multiply refined molybdenum is much more ductile than commercial grade molybdenum[36]; however, it is not clear that the required purity levels can be obtained economically on a commercial scale. In addition to being very heavy, tungsten is brittle at AMTEC condenser temperatures (550–650 K). Based on their ductility, vanadium, niobium, and tantalum are good candidates; molybdenum may be acceptable; and tungsten should be avoided.

Yield strength is a good measure to rank the refractory metals in terms of their strength at the operating temperatures of interest in vapor-anode AMTEC cells. Based on the data presented in Fig. 8, molybdenum is the strongest refractory metal at AMTEC hot-end temperatures (1150–1200 K), followed by tantalum and tungsten.[29] Niobium and vanadium both exhibit comparably lower strengths. A large fraction of the induced structural stresses in the cell during launch will result from the weight of the structural material. Also, AMTEC cells designed for space applications need to be of low mass. Therefore, a comparison of the strength-to-density ratios of the refractory metals is also an important differentiating criterion (Fig. 9). Based on such a comparison, molybdenum is clearly superior at the cell's hot-end temperature, and vanadium emerges as a strong contender at the cell's cold-end temperature. For strength, molybdenum appears to be the best candidate, and tantalum and vanadium also warrant consideration (Figs. 8 and 9). Niobium is the poorest candidate at lower temperatures (< 1000 K), but is comparable to vanadium at the AMTEC cell's hot-side temperature ($\sim$1150–1200 K).

At a temperature that is more than one-third of a material's melting point, creep strength, or resistance to long-term deformation under load, becomes important.[37] Many parameters have been used to quantify creep resistance; however, comparable data are difficult to come by. The 100-h rupture strength has been chosen as a representative criterion. Based on the data presented in Fig. 10, tungsten is clearly the standout material.[29] Molybdenum also demonstrates greater creep resistance than either niobium or tantalum. Although comparable data for vanadium were not found, it is expected to be the least creep resistant of the five candidate refractory metals because of its lower melting point. When the rupture strength-to-density ratio is considered (Fig. 11), tungsten still comes out as the strongest refractory metal, but the attractiveness of molybdenum and niobium has increased noticeably.

Three main mechanisms govern corrosion of refractory metals in alkali metal systems: dissolution, mass transfer, and reactions with impurities such as dissolved oxygen.[26] Mass transfer of volatile metals within PX-series AMTEC cells is one of the main reasons why refractory metals and alloys are being considered as alternatives to the stainless steels and superalloys used in or proposed for the early PX-type cells.

Figure 12 compares the sublimation vapor pressures of the refractory metals and the alloying elements of interest with the major constituents of the stainless steels and superalloys (see Ref. 38). All of the refractory metals under consideration have vapor pressures below 10^{-9} torr (133 nPa) at all AMTEC operating temperatures (< 1200 K). A sublimation vapor pressure of 10^{-9} torr has been recommended as a rule of thumb by JPL to minimize mass transfer over a 7–15 year lifespan.[8] Reliable quantitative data on the corrosion of the refractory metals by liquid and vapor alkali metals are scarce; however, several sources have summarized the results of the numerous capsule and single- and two-phase loops tests conducted during the SNAP and SP-100 programs.[26,39] In general, these tests concluded that the compatibility of the refractory metals with sodium is very good, providing that the oxygen contents of the sodium and in the refractory metal are controlled, keeping it relatively low (< 10 ppm). Niobium and tantalum are particularly susceptible to oxygen-driven attack.[26]

Table 1 Ranking of refractory metals based on thermophysical properties

Criteria	V	Nb	Ta	Mo	W
Density	Excellent	Acceptable	Poor	Acceptable	Poor
Thermal conductivity	Acceptable	Acceptable	Acceptable	Poor	Poor
Emissivity	Acceptable	Acceptable	Acceptable	Acceptable	Acceptable
Linear expansion	Acceptable	Acceptable	Acceptable	Acceptable	Acceptable
Rank	1	2	3	3	4

Table 2 Ranking of refractory metals based on structural properties

Criteria	V	Nb	Ta	Mo	W
Ductility	Acceptable	Acceptable	Acceptable	Acceptable	Poor
Yield strength	Acceptable	Acceptable	Acceptable	Acceptable	Acceptable
Yield strength/density ratio	Excellent	Acceptable	Acceptable	Excellent	Acceptable
Rupture strength	Likely acceptable	Acceptable	Acceptable	Acceptable	Excellent
Rupture strength/density ratio	Likely acceptable	Acceptable	Acceptable	Acceptable	Acceptable
Rank	2	3	3	1	4

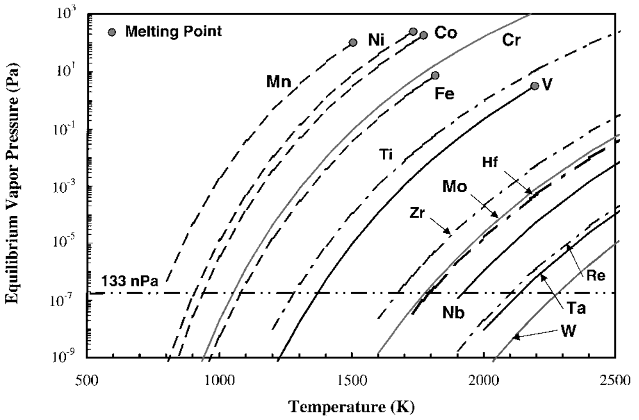

Fig. 12 Equilibrium vapor pressure of the refractory metals and alloying additions (data from Nesmeyanov[38]).

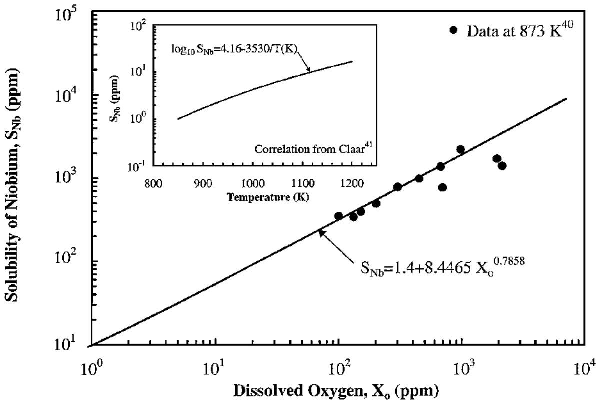

Fig. 13 Effect of dissolved oxygen and temperature on the solubility of niobium in liquid sodium.

As shown in Fig. 13, the solubility of niobium in liquid sodium at 873 K is strongly affected by the oxygen content of the liquid metal,[40] which is increasing proportionally to the oxygen content raised to the power of 0.7858. The insert in Fig. 13 shows the solubility of niobium in sodium in the absence of oxygen, which is about 1.4 ppm at 873 K (Ref. 41). In the absence of oxygen, the solubility of niobium in sodium at 873 K agrees with the correlation in the insert at the same temperature. Note that there is a large scatter in the niobium solubility data at oxygen contents >1000 ppm, which suggests that other impurities present in the experiment may have affected the results.[40] The correlation[41] used to produce the curve in the insert of Fig. 13 indicates that in the absence of oxygen the solubility of niobium in liquid sodium follows the Arrhenius relationship with temperature (see Fig. 13).

Niobium and tantalum require alloying with an oxygen getter such as zirconium or hafnium (to stabilize dissolved oxygen) to withstand long-term alkali-metal exposure. Vanadium has demonstrated behavior similar to those of niobium and tantalum in compatibility tests.[42] Molybdenum and tungsten do not appear to be as susceptible to oxygen-assisted alkali-metal attack, and nongettered alloys have demonstrated acceptable performance in capsule tests.[22] Given that the group 6 elements (molybdenum and tungsten) have low vapor pressures and are better able to withstand sodium attack without alloying additives, they are preferred over the group 5 metals (vanadium, niobium, and tantalum). The latter have similarly low vapor pressures (with the exception of vanadium, which is still in the acceptable range), but will require further investigation to identify acceptable alloys.

A rating of the candidate refractory metals based on the selection criteria detailed earlier is presented in Tables 1 and 2. A metal rated poor for a given property either fails to meet an accepted criterion or is significantly worse than other metals considered. An acceptable metal appropriately meets a criterion and is not significantly better or worse than other metals. A metal that is significantly better than others considered is rated excellent. The ratings for each category were used to determine a relative ranking for each of the metals in terms of acceptable thermophysical and structural properties.

Based on the present evaluation and the relative ranking of the thermophysical properties of the refractory metals, vanadium is the first choice as an AMTEC structural material followed by niobium. Based on the structural properties, molybdenum is ranked first, followed by vanadium. Because of limited data available for vanadium, particularly concerning long-term creep-rupture performance, and because of the very limited experience with the use of vanadium as a space structural material, niobium and molybdenum appear to be the best candidates. Unfortunately, both niobium and molybdenum possess certain weaknesses. Niobium is susceptible to oxygen-driven alkali-metal corrosion, and molybdenum is brittle at low temperatures. Alloys of these two refractory metals, which are reviewed next, have addressed both of these problems.

Refractory Alloys

During the SNAP program in the 1960s, a number of companies were involved in the development of many different niobium alloys.[23] Unfortunately, only a few of those alloys remain commercially available.[13] Nb–1Zr was an early development and remained in use in the nuclear industry and as a general purpose high-temperature alloy. PWC-11 was also developed in the 1960s as a higher strength alternative to Nb–1Zr (Ref. 25) but never entered commercial production; nonetheless, a large amount of research and development was performed on PWC-11 during the SP-100 program.[23] Because the fabricability and the cold-work characteristics of PWC-11 are very similar to those of the commonly available Nb–1Zr, it is a possible candidate for use as an AMTEC cell structural material. The niobium alloy C-103 (niobium–10% hafnium–1% titanium–0.5% ziconium) is a commercially available aerospace alloy that was developed during the Apollo program and has seen significant use in military aircraft. All three niobium alloys contain either zirconium and/or hafnium, which have been demonstrated to improve dramatically the alloys' resistance to oxygen-driven alkali metal corrosion.[26] The density of these niobium alloys is very similar (Nb–1Zr, 8.58 g/cm^3; PWC-11, 8.6 g/cm^3; and C-103, 8.86 g/cm^3).

During the same time frame, tantalum alloys received significant attention, and several alloys were developed, including T-111 (tantalum–8% tungsten–2% hafnium) and ASTAR-811C (tantalum–8% tungsten–1% rhenium–0.7% hafnium–0.025% carbon) (Ref. 21). These alloys had high yield and creep strengths[21] and demonstrated good compatibility with liquid alkali metals.[26] However, because of the high density of tantulum, tungsten, hafnium, and rhenium (Fig. 3a), these alloys are all very heavy and will not be considered further.

The development of the molybdenum alloys has been historically less energetic than the development of the niobium alloys, but several promising candidates exist. TZM (molybdenum–0.5% titanium–0.1% zirconium) is readily available and offers improved strength and ductility over the base molybdenum metal.[22] Recent work on the ductilization of molybdenum by the addition of rhenium has resulted in the availability of a range of molybdenum–rhenium binary alloys. These alloys, with 14–44.5% rhenium, offer significantly improved ductility; however, above 44.5% rhenium, an undesirable sigma phase is formed (see Fig. 14).[43,44] Mo–14Re (molybdenum–14% rhenium) and Mo–44.5Re (molybdenum–44.5% rhenium) are worthy of further consideration as AMTEC structural materials. Mo–44.5Re is commercially available and is currently being considered for use in AMTEC cells.[44] Mo–14Re is only available as a special order, but offers the best ductility improvement from a lower rhenium addition and at a lower density. The density of the molybdenum alloys spans a greater range than the niobium alloys (TZM, 10.16 g/cm^3; Mo–14Re, 11.89 g/cm^3; and Mo–44.5Re, 13.50 g/cm^3).

In ranking the refractory metal alloys, only the properties that have led to significant discrimination between the refractory metals (thermal conductivity, surface emissivity, yield strength, and yield strength to density ratio) were reconsidered. The thermal conductivity of the candidate refractory alloys is presented in Fig. 15 (see Refs. 8, 32–34, 43, 45, and 46). In each case, the alloys have lower thermal conductivities than the parent metals. This is particularly significant for C-103, which has a thermal conductivity that is close to that of stainless steel and Haynes-25, which result in significantly lower parasitic heat conduction losses in the AMTEC cell. The thermal conductivity of TZM is very high. The thermal conductivity of the Mo–Re alloys has been noticeably reduced, and the conductivity of Mo–44.5Re is comparable to that of Nb–1Zr and PWC-11.

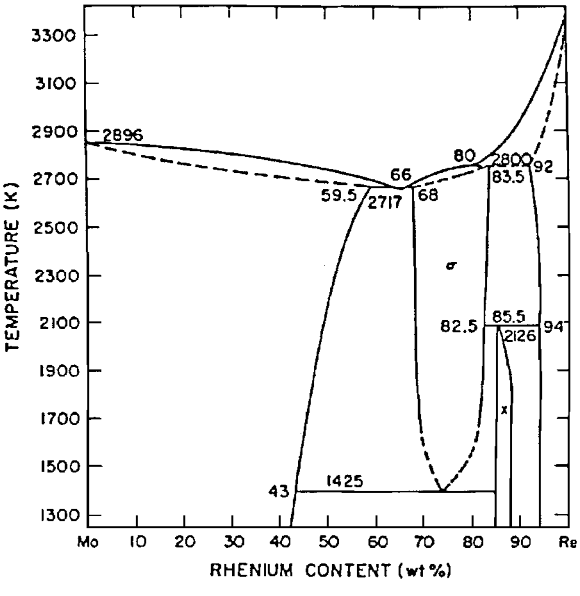

Fig. 14 Molybdenum–rhenium alloy phase diagram (Lundberg[43]).

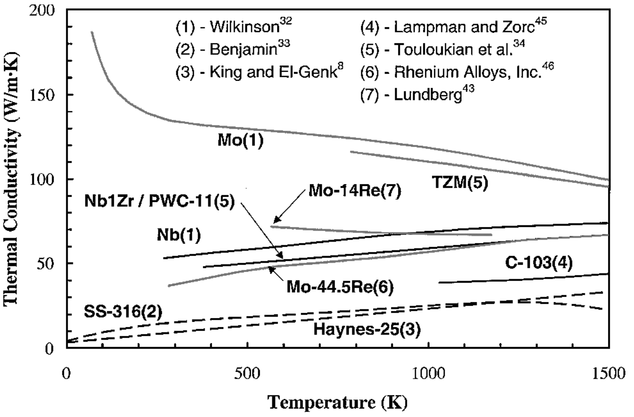

Fig. 15 Thermal conductivity of selected refractory alloys.

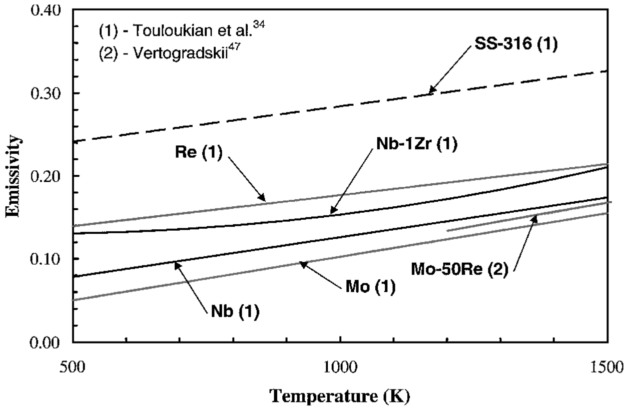

Fig. 16 Thermal radiative emissivity of selected refractory alloys (data from Touloukian et al.[34] and Vertogradskii[47]).

One important criterion for evaluating the refractory alloys is that the addition of alloying materials should not significantly increase the surface emissivity of the resulting alloy. This was a particular concern with the Mo–Re alloys because the surface emissivity of rhenium is notably higher than that of the other refractory metals under consideration.[34] Figure 16 shows the surface emissivities of Nb–1Zr and Mo–50Re (Refs. 34 and 47). Although the emissivity of both alloys is higher than that of the alloys' base metal, it is still below that of stainless steel. Data for the other alloys were not available. Mo–50Re probably bounds the molybdenum alloys, and C-103 may have an emissivity between that of Nb–1Zr and stainless steel.

Figure 17 compares the yield strengths of the candidate alloys (see Refs. 23, 29, 45 and 48). Of note are the particularly high strengths of Mo–44.5Re and TZM. Whereas the strength of the C-103 is comparable to that of the other niobium alloys at the cells' typical hot-end temperature (1150–1200 K), it is superior to the other niobium alloys near the condenser temperature (<650 K). With C-103, a thinner cell wall above the BASE tubes could be used, which would reduce heat loses by conduction to the condenser, increasing the cell efficiency. PWC-11 is stronger than Nb–1Zr, but not dramatically so, considering the readily available C-103 as an alternative. The yield strength to density ratios of the candidate alloys are presented in Fig. 18. Because the density of the niobium alloys are similar, there is little impact on their relative ranking. The molybdenum alloys,

Table 3 Ranking of refractory alloys

	Niobium alloys			Molybdenum alloys		
Criteria	Nb-1Zr	PWC-11	C-103	Mo-14Re	Mo-44.5Re	TZM
Thermal conductivity	Acceptable	Acceptable	Excellent	Likely acceptable	Acceptable	Poor
Yield strength	Acceptable	Acceptable	Excellent	Acceptable	Excellent	Excellent
Yield strength/density ratio	Acceptable	Acceptable	Excellent	Acceptable	Acceptable	Excellent
Emissivity	Acceptable	Likely acceptable	Likely acceptable	Acceptable	Acceptable	Likely acceptable
Rank	2	3	1	2	1	3

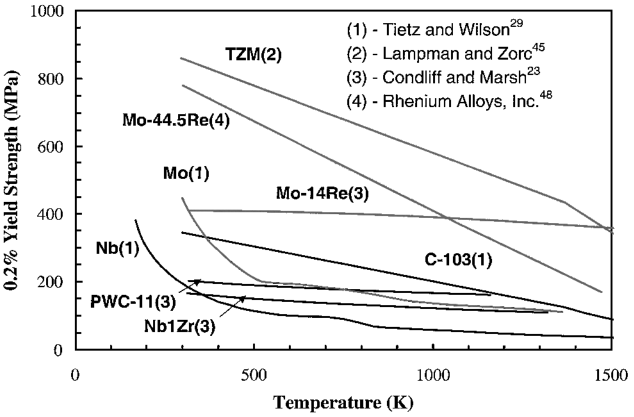

Fig. 17 Yield strength of selected refractory alloys.

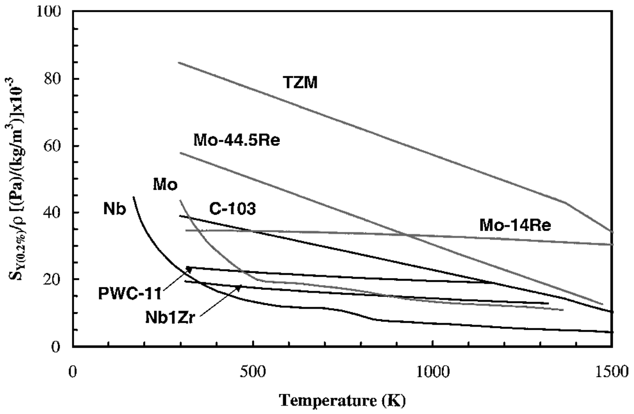

Fig. 18 Yield strength to density ratio of selected refractory alloys.

however, are more affected by their differences in density. The high strength of Mo–44.5Re, compared to pure molybdenum, is offset by the alloy's high density. TZM is still a very strong material.

As indicated earlier, the selected refractory alloy should be compatible with the AMTEC operating environment. This means that the vapor pressure of the constituent metals should be low ($< 10^{-9}$ torr or 133 nPa) and that the alloy demonstrates adequate resistance to alkali metal corrosion. The vapor pressures of the metal additions in each of the candidate alloys are shown in Fig. 9. In every case, the vapor pressure is below the 133-nPa rule of thumb, although titanium approaches this limit at the AMTEC hot-end temperature (1150–1200 K). Capsule testing of Nb–1Zr and TZM has demonstrated good resistance to attack by liquid and vapor sodium.[26] Such data are not available for PWC-11, C-103, and molybdenum–rhenium. Data for niobium alloys have shown that alloys containing zirconium or hafnium perform satisfactorily, and so PWC-11 and C-103 are judged to be acceptable. The only data available for molybdenum–rhenium alloys are for Mo–50Re in lithium and for tungsten–rhenium in several other liquid metals.[22,39] In both cases, acceptable resistance was noted, and the molybdenum–rhenium alloys are likely to perform satisfactorily in the AMTEC environment.

Table 3 ranks the candidate niobium and molybdenum alloys relative to the other alloys with the same primary metal based on the same screening criteria used for the refractory metals. Whereas all of the niobium alloys in Table 3 are acceptable, C-103 is rated excellent with regard to every criterion. From a strength standpoint, all of the molybdenum alloys are rated acceptable to excellent; unfortunately, their high thermal conductivity is problematic. Only the addition of large amounts of low-thermal-conductivity rhenium makes Mo–44.5Re acceptable, at the expense of significantly increasing the density of the resulting alloy. Whereas the preferred niobium alloy is clearly C-103, Nb–1Zr is rated acceptable in every category; significant experience with this particular alloy has been developed during the SNAP and SP-100 programs. The higher thermal conductivity of Nb–1Zr will be of benefit at the hotter end of the cell when it is matched with a lower thermal-conductivity structural material (such as C-103) in the cooler part of the cell, particularly the thin (~100-μm) cell wall above the BASE tubes.

King and El-Genk[17] have recently investigated the performance of an Nb–1Zr/C-103 vapor-anode AMTEC cell. The cell used Nb–1Zr for the hot plate, the BASE tube support plate, the evaporator standoff and evaporation wick, and the thick (~200-μm) wall facing the BASE tubes and C-103 for the thin (~100-μm) side wall above the BASE tubes, the condenser structure, the casing and the wick of the liquid sodium return artery, and the interior thermal-radiation shield. This refractory cell performed equal to or better than a nickel/Haynes-25 cell of similar design and was only slightly heavier.

Whereas the potential for oxygen embrittlement is likely to apply to most of the niobium alloys, including C-103, the higher thermal conductivity of the molybdenum alloys will increase the parasitic conduction heat losses in the cell and is likely to reduce the cell efficiency. Also, because the Mo–44.5Re is ~50% more dense than C-103 and Nb–1Zr, it would increase the cell mass. Therefore, the niobium alloys, Nb–1Zr and C-103, are recommended for use as structural materials in vapor-anode AMTEC cells. In case oxygen embrittlement turns out to be intractable, Mo–Re alloys should be considered in conjunction with extensive cell design and stress analyses to reduce the total cell mass.

Summary and Conclusions

Vapor-anode, multitube AMTEC cell technology has been under development for more than six years. These cells are currently being considered for use in conjunction with radioisotope heat sources to provide the electric power requirements for several of NASA's future space exploration missions. Early PX-series cells had stainless-steel structures, and several have been tested in vacuum without failure. However, the test results raised concerns regarding the use of stainless steel as a structural material for vapor-anode, sodium AMTEC cells. The volatility of iron and other alloying elements such as manganese, nickel, and chromium, along with their potential reactions with the BASE and the AMTEC electrodes, were considered potential contributors to the observed degradation in cell performance in the vacuum tests.

Recently, refractory metals and alloys are being considered to replace stainless steel and superalloys as structural materials in vapor-anode AMTEC cells. Despite their strength and very low vapor pressure at typical AMTEC cell operating temperatures (1150–1200 K and 550–650 K for the cell hot plate and condenser, respectively), several of the refractory metals are incompatible with sodium at these temperatures, particularly in the presence of small amounts of oxygen (< 10 ppm). To enhance the corrosion resistance of these refractory metals, alloying elements with a high affinity for oxygen (gettering elements) are added to the primary metals to sequester the sodium soluble oxygen.

The existing database for refractory metals and alloys for their potential use as structural materials in vapor-anode AMTEC cells is reviewed. In addition to requiring that the vapor pressure of the selected material be below 10^{-9} torr (133 nPa), at a typical hot-side operational temperature of 1200 K, other screening criteria were considered, namely, 1) low–moderate thermal conductivity; 2) low density, surface emissivity, and ductile-to-brittle transition temperature; 3) high yield and rupture strengths and high strength-to-density ratios; 4) low thermal expansion; and 5) good compatibility with the AMTEC operating environment, including high corrosion resistance to sodium in both the liquid and vapor phases.

None of the refractory metals were perfect candidates, and none were ranked first by all of the screening criteria; however, niobium and molybdenum were ranked the highest. Niobium, however, is subject to corrosion by sodium even in the presence of less than 10 ppm oxygen. This problem could be alleviated by alloying niobium with gettering elements such as zirconium and hafnium, which sequester oxygen and, hence, increase the corrosion resistance of the primary refractory metal. The use of molybdenum is constrained by its low ductility, particularly at room temperature, and its high thermal conductivity. Alloying additives, particularly rhenium, can dramatically improve the ductility of the primary metal and lower the thermal conductivity of the resulting alloy, but at the expense of increasing the density of the alloy.

The alloy Nb–1Zr is recommended for the hot-cell structure. This includes the cell's hot plate, the BASE tube support plate, the evaporator standoff and evaporation wick, and the thick (~200-μm) wall facing the BASE tubes. The cell wall above the BASE tubes is almost half the thickness of the hotter part of the wall to minimize parasitic conduction heat losses to the cell condenser. For this part of the structure, a strong material with low thermal conductivity is preferred. Therefore, the niobium alloy C-103, containing the oxygen getters zirconium and hafnium along with titanium, is recommended for the cold structure of the AMTEC cell. This includes the cell wall above the BASE tubes, the condenser structure, the casing and the wick of the liquid sodium return artery, and the interior thermal radiation shield. C-103 is stronger and less thermally conductive than Nb–1Zr.

Mo–Re alloys, with 14–44.5% rhenium, may also be considered as structural materials in vapor-anode AMTEC cells. However, the cell design should be optimized for strength, performance, and lower mass. Otherwise, the high density and thermal conductivity of these alloys would result in a heavy, low conversion efficiency cell.

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