HIGH TEMPERATURE COMPATIBILITYISSUES FOR FUSION REACTOR STRUCTURAL MATERIALS*

B.A. Pint, J. R. DiStefano and P. F. Tortorelli
Metals and Ceramics Division, Oak Ridge National Laboratory,
P. O. Box 2008, Oak Ridge, TN, 37831-6156 865-576-2897 pintba@ornl.gov

* dedicated to the memory of E. E. Hoffman

The primary functions of the blanket in a deuterium/tritium-fueled fusion reactor are to convert neutron kinetic energy into heat, breed tritium for the fuel cycle and accommodate a heat transfer system. Various blanket concepts have been considered including those incorporating liquid metals, molten salts, water and He. The objective of this review is to outline some of the critical compatibility issues associated with structural materials being considered for designs operating at >500°C. Examples are given for vanadium alloys, magnetohydrodynamic coatings, oxide dispersion strengthened ferritic alloys and silicon carbide composites.

I. INTRODUCTION

Various blanket concepts are being considered for deuterium/tritium-fueled fusion reactors. The blanket has two primary functions: to convert the neutron kinetic energy of the reaction to heat and to breed tritium for the fuel cycle. The blanket also must accommodate the transfer of heat to the power conversion system. Several different concepts are being considered including those incorporating liquid metals, molten salts, solid breeders, water and He. Each concept involves specific materials compatibility issues that need to be addressed to assure feasibility and, in the long term, adequate lifetime of the reactor.

Increasing the operating temperature of the blanket is attractive from an economic perspective as higher temperatures are anticipated to increase the efficiency of the reactor. The objective of this review is to outline some of the critical compatibility issues associated with the structural materials being considered for designs operating above 500°C. Thus, issues for near-term designs of demonstrations reactors involve water cooling and ferritic steels will not be considered.

There has been a long-running dialogue between fusion reactor designers and materials scientists about the compatibility of structural materials proposed for potential blanket and heat transfer concepts.[1-3] A focus of the current review is on critical compatibility issues associated with structural materials of current interest[4] to the fusion energy community, such as vanadium alloys,[5] magnetohydrodynamic coatings,[6-7] oxide dispersion strengthened alloys[8-9] and silicon carbide composites.[10]

II. VANADIUM ALLOYS

Vanadium alloys are being considered as a first wall structural material because of low activation, radiation resistance and compatibility with Li. A large amount of work has focused on V-4Cr-4Ti. However, vanadium has very poor oxidation resistance in air above 400°C.[11] In air at 500°C, V-4Cr-4Ti forms a multi-layer oxide of VO_x.[12] Because V_2O_5 melts at 690°C, vanadium alloys undergo catastrophic oxidation in air at higher temperatures. Because of its high oxygen solubility, the oxidation behavior of vanadium and its alloys is significantly different than conventional Fe- and Ni-base alloys which have relatively low oxygen solubilities.[13] For conventional alloys, high temperature oxidation resistance is improved by alloying with Cr, Al and/or Si which results in the formation of a surface oxide enriched in the less noble alloying elements. If dense and adherent, this external oxide or scale inhibits further oxidation because the reaction is limited by solid state diffusion through this layer. However, improvements in oxidation resistance by alloying additions to vanadium have been largely unsuccessful.[14,15] Alloying additions of 30% were unable to prevent the formation of vanadium oxides.

Oxidation resistance is also a concern with vanadium alloys in a He cooled design. In this case, where the oxygen partial pressure (due to impurities in the He) would be much lower, vanadium alloys are susceptible to embrittlement by O and H. In any blanket design, hydrogen would be present from plasma implantation fluxes and transmutations.

In order to study their high temperature behavior, vanadium alloys have been tested in vacuums containing low levels of O and H additions. At low oxygen partial pressures, O adsorbed onto the alloy surface diffuses into the alloy rather than form a surface layer because of its

high O solubility. The uptake of oxygen in these low oxygen partial pressure environments causes a loss in room temperature and high temperature ductility for V-4Cr-4Ti, Figure 1.[16] (To date, most mechanical property information has been based on tensile specimens. Fracture specimens would provide useful information on the environmental effect on the ductile to brittle transformation temperature.) Application of standard oxidation principles to this environmental degradation would suggest that formation of a surface oxide should help prevent this type of embrittlement by inhibiting the diffusion of oxygen into the substrate. In fact, when high levels of O are observed, a surface oxide does form and the measured ductility was not as low as anticipated, Figure 1. However, for alloys with high O solubility, the surface oxides is not in thermodynamic equilibrium with the underlying metal. Thus, when specimens of V-4Cr-4Ti with a surface oxide were subsequently annealed for 2000h at 700°C,[16] the ductility dropped to zero, Figure 2. Other specimens with little or no added O showed only a minor drop in ductility after the anneal. Rather than acting as a barrier for oxygen uptake, the surface oxide was a source of oxygen for further embrittlement of the substrate.

There has been some debate as to the reaction kinetics for vanadium alloys exposed to low levels of oxygen in vacuum with some studies suggesting that parabolic kinetics were observed.[17,18] Parabolic kinetics are typical for high temperature oxidation of conventional alloys[13] but for initial oxygen uptake in a refractory metal with significant oxidation solubility, linear kinetics are

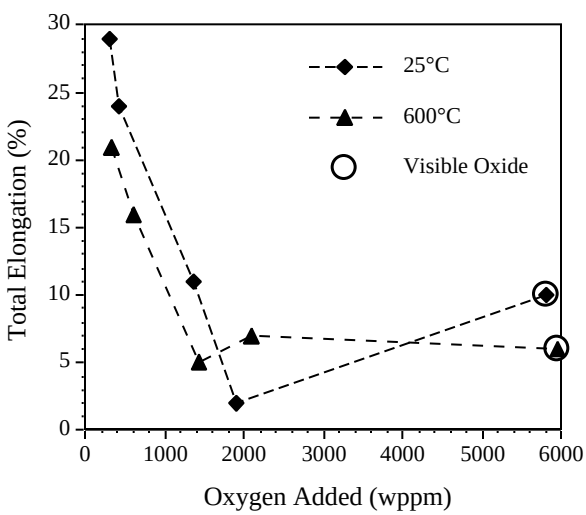

Figure 1. Total elongation at 25° and 600°C for V-4Cr-4Ti as a function of oxygen uptake during exposures to low oxygen partial pressures at 600° and 700°C. At very high oxygen uptakes, a surface oxide was visible.

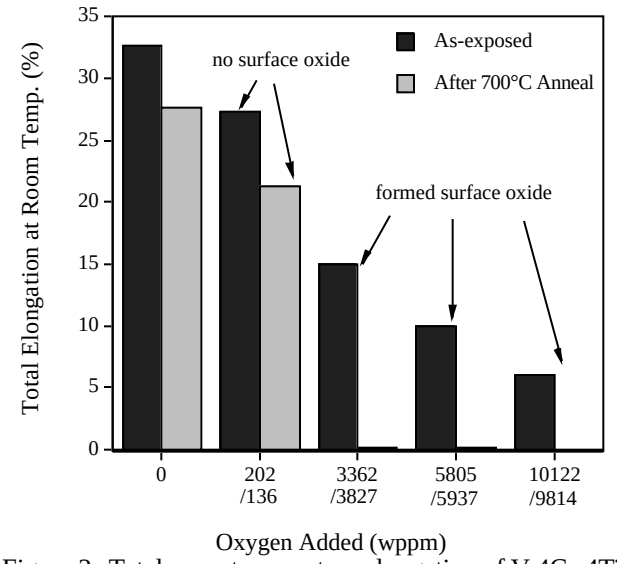

Figure 2. Total room temperature elongation of V-4Cr-4Ti specimens with and without a 2000h, 700°C anneal. Specimens with a surface oxide showed a loss of all ductility after the anneal.

expected. Figure 3 shows a range of mass gain data for oxygen partial pressures of 10^{-3}-10^{-6}Pa at 600° and 700°C. At the lower pressures, the reaction kinetics are linear (n=1). However, at higher pressures where most of the early work was performed, the reactions do not follow a linear relationship and values of n range up to 3 for exposure in 10^5Pa(1 atm) of He. Thus, parabolic and

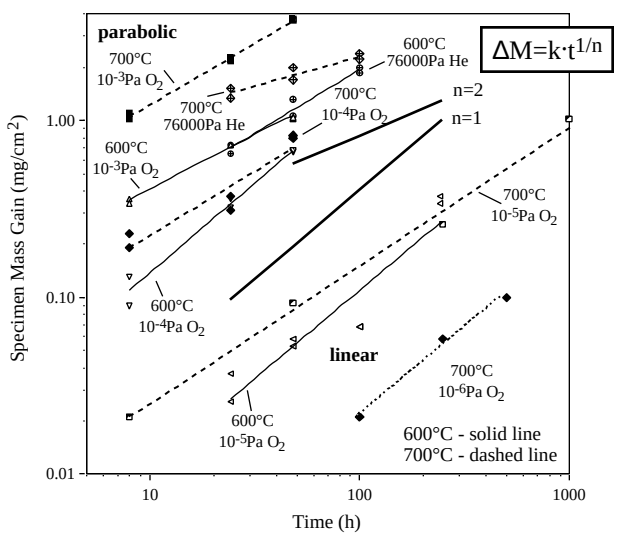

Figure 3. Power law fit of V-4Cr-4Ti mass gain (O uptake) data at 600° and 700°C in 1 atm He and 10^{-3}-10^{-6}Pa of O. Linear kinetics (n=1) were measured at low pressures while higher values of n were measured at higher pressures. However, at higher pressures the alloy was quickly embrittled due to the high mass gain.

linear kinetics both may be observed depending on the oxygen pressure. However, the higher pressures of oxygen at these temperatures quickly result in high oxygen uptakes and embrittlement. Therefore, for the purposes of modeling long-term performance in low oxygen pressures or He, the linear kinetic relationship is of more technological interest.

A model for the oxygen uptake in low oxygen pressures has been developed based on the available data.[16] The model suggests that the He would need to be purified to very low levels of O (< 1vppb) in order for V alloys to operate for extended periods at 700°C without embrittlement. However, current approaches rely on reaction kinetic data taken in vacuum with low oxygen pressures rather than high pressure He with low impurities. Also, the time to embrittlement is dependent on the surface area to volume (SA/V) ratio of the components. The model makes an assumption about the effect of this ratio on the time to embrittlement that only have been experimentally confirmed for other Nb- and Ta-base refractory alloys.[19,20] Assuming embrittlement occurs with an O uptake of 2000ppmw O, Figure 4 shows the anticipated effect on the time to embrittlement for test specimens (high ratio) to structural components with a significantly lower ratio. The effect of the SA/V ratio needs to be studied experimentally in order to verify the current model predictions with respect to long-term life predictions.

The effect of H uptake on the mechanical properties of V-4Cr-4Ti with and without O uptake has been considered in two studies.[21-23] The earlier work showed that a severe loss in room temperature ductility occurred with a H uptake of more than 500ppmw, Figure 5. It also found that there was a synergistic effect of O and H. Tensile specimens that were initially exposed to a low pressure O-containing environment where they picked up ≈850ppmw O showed a large decrease in ductility after a subsequent treatment where they only picked up 100-200ppmw H, Figure 5. Similar drops in ductility were observed in the subsequent study.[23]

In general because of the potential embrittlement problems, it appears unlikely that vanadium alloys could be used with a He-cooled blanket design. In other power generation technology concepts, refractory alloys like vanadium are typically operated in ultra high vacuum, rather than He, to avoid such high temperature embrittlement. A more attractive concept for vanadium structural alloys is a liquid lithium blanket concept because vanadium and its alloys generally have good compatibility with lithium. However, that concept must address the need for coatings, as discussed in the next section.

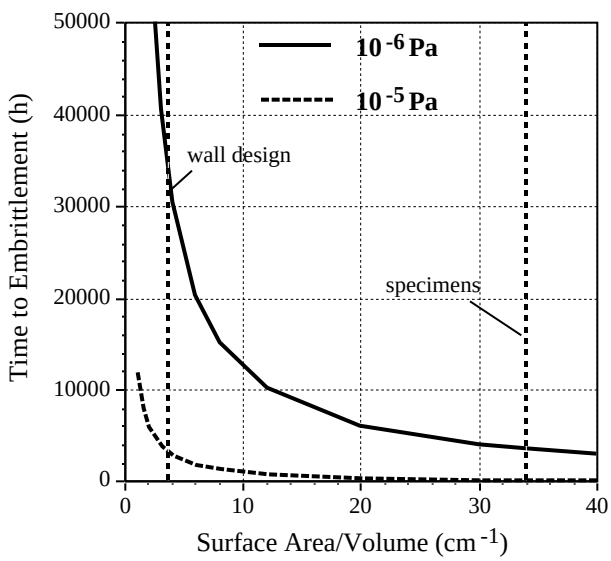

Figure 4. Time to embrittlement (defined as uptake of 2000ppmw O) as a function of the surface area to volume ratio of V-4Cr-4Ti. The ratio for the test specimens is much higher than that for a structural component.

III. MAGNETOHYDRODYNAMIC COATINGS

In all alloy - lithium blanket concepts where a strong magnetic field is used to contain the fusion plasma, a magnetohydrodynamic (MHD) pressure drop is developed when the electrically conductive lithium flows across the magnetic field lines. To minimize the MHD resistance to flow, it is necessary to have an insulating barrier to decouple the lithium and the alloy structure. The coatings

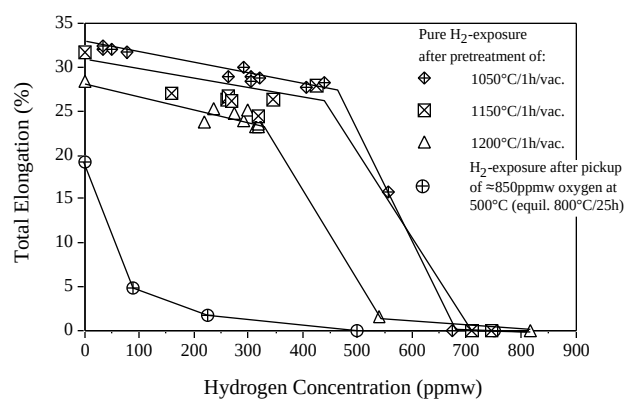

Figure 5. Total room temperature elongation of V-4Cr-4Ti specimens with various levels of H uptake. Specimens with both O and H uptake showed embrittlement with much lower H levels.

must be thin, durable, electrically resistive and compatible with Li at temperatures up to ≈700°C.[6,7]

It has been recognized since 1960's research on space power reactor concepts that there are few, if any, ceramics that are compatible with flowing Li for extended periods at high temperature (≈1100°C).[24,25] The limitations of vanadium alloys make the requirement only ≈700°C for fusion reactors. However, the fundamental issue remains that there are few materials that are compatible with Li. Carbides and nitrides generally have better compatibility than oxides because of the low free energy of Li_2O relative to its carbide or nitride. However, most metal carbides do not have the needed resistivity for this application. Among the nitrides, AlN is a candidate material[26,27] but there is some question about a potential reaction between Ti in the V-4Cr-4Ti alloy with the nitride coating. For oxides, a first screening looks for oxides with a lower free energy of formation than Li_2O, which leaves only a handful of candidates. As an example, Al_2O_3, which would be an excellent candidate because of its high resistivity, does not have sufficient stability. Alumina specimens have been observed to completely dissolve in static tests at 375°-450°C.[28-30]

For the past decade, CaO has been investigated as a candidate coating material.[7,31,32] One of its attractive features is its high solubility in Li which suggested that it might be possible to have a self-healing coating. However, more recent experimental work[27,33] on bulk CaO specimens and thermodynamic calculations have

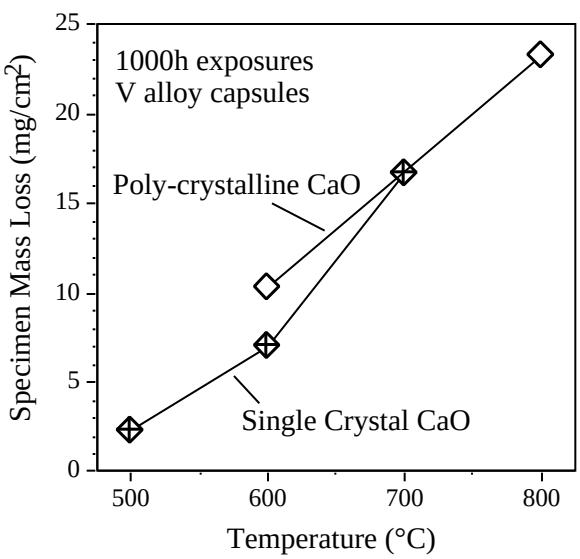

Figure 6. Mass loss of CaO specimens in Li as a function of temperature after a 1000h exposure in a V alloy capsule. Both polycrystalline and single-crystal specimens showed high dissolution rates.

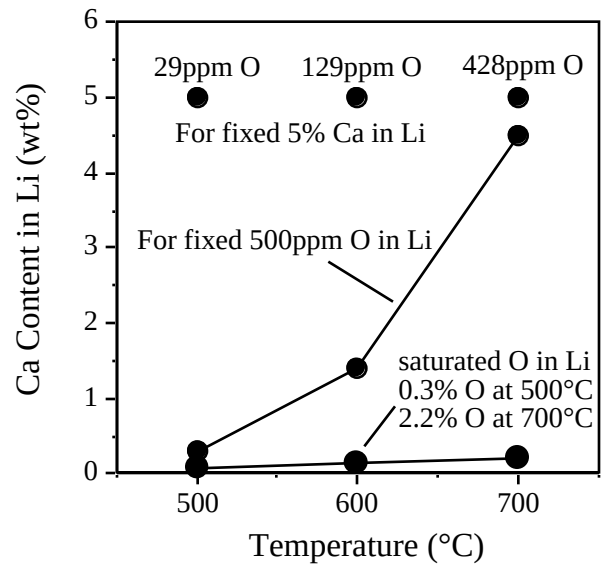

Figure 7. Calculated equilibrium solubilities of Ca and O in Li using three assumptions (i) a fixed 5% Ca content, (ii) a fixed 500ppmw O and (iii) Li saturated with O.

proven that CaO cannot perform adequately at 600°-800°C in static Li tests, Figure 6. A mass loss of ≈3mg/cm^2 is equivalent to a 10µm loss of material. Therefore, the mass losses at these temperatures are unacceptably high for a thin coating. The experimental results were consistent with thermodynamic calculations which showed that the equilibrium solubility of Ca in Li increased dramatically with temperature, consistent with the mass losses, Figure 7. These results suggest that oxides with cations with a low solubility in Li will likely have better compatibility. Because of its poor high temperature compatibility, the CaO coating development program in the U.S. has recently been concluded.

Currently, there are a few candidates (e.g. Y_2O_3, AlN and Er_2O_3) that have shown promise in static Li tests at 800°C, Figure 8. Testing has begun on Y_2O_3 coatings made by physical vapor deposition.[34] For coating evaluations, the figure of merit is the change in coating resistivity at 700°C after exposure to Li. The coating must maintain adequate resistivity in order to warrant further testing. Thus, development of MHD coatings is progressing, but as yet no coatings have been tested in a flowing Li environment with a temperature gradient. Prior experience with Y_2O_3 showed only limited dissolution in static Li testing at 1100°C but complete dissolution in flowing Li in 109h at 1143°C due to mass transfer.[24,25] There is a critical need for testing of ceramic candidates in flowing Li at ≈700°C in order to determine if any of the current candidates has sufficient Li compatibility to be used as a MHD coating.

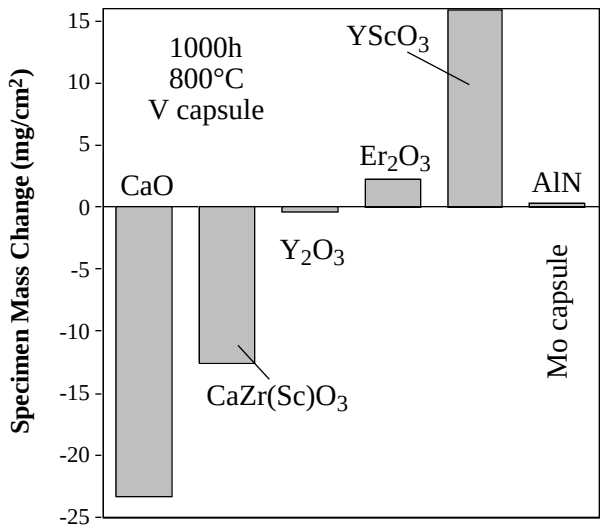

Figure 8. Mass change of bulk ceramic specimens in Li after 1000h at 800°C in a V alloy or Mo capsule (for AlN). Mass gains were attributed to Li infiltrating preexisting porosity in the ceramic specimen.

IV. ODS FERRITIC ALLOYS

Oxide-dispersion strengthened (ODS) ferritic alloys (e.g. Y_2O_3-TiO_2 dispersed Fe-13wt.%Cr-3W) have received recent interest because of the possibility of increasing the maximum creep-limited operating temperature of this class of alloys by 150°C or more.[4,8,9] However, in order for ODS alloys to impact the overall system, they must be compatible with a blanket and heat transfer concept. To date very little work has been published about the compatibility of this class of alloys except for long-term oxidation in dry and humid air.[35,36] Based on the performance of wrought ferritic alloys, potential limitations can be anticipated for various systems:

i. Liquid Metals

Iron-base alloys have a reputation for poor compatibility with some liquid metals. For Pb-Li, both Fe and Cr have a high solubility and relatively high dissolution rates even at 500°C.[37] A similar high rate of attack is anticipated in Sn-Li. However, it is possible that either metallic or ceramic coatings could reduce the rate of attack in such a system. Such strategies should be explored.

For liquid Li, Ni and Mn are the most susceptible elements to dissolution. Thus, higher rates of attack have been measured for austenitic alloys than ferritic alloys.[2] Loop testing of Fe-12Cr showed a relatively low dissolution rate at 600°C (3 mg/m^{2}h) after 7000h.[38] There may be some slight preferential attack of Cr but Fe and Cr have similar rates of dissolution in Li. Thus, ODS ferritic alloys may be able to operate at 600°C. Even with optimistic assessments, the maximum temperatures with liquid metals are significantly lower than the creep limited temperature. But these materials should be evaluated in loop testing at higher temperatures to confirm these assessments and investigate strategies for reducing the dissolution rate.

ii. Molten Salts

Of the various Li-containing molten salts considered for fusion applications, only the flourides (Li_2BeF_4, i.e. FLiBe) have sufficient bonding energies needed for radiation stability. Loop testing of ferritic alloys has shown that Cr is selectively attacked due to the formation of CrF_2 which deposits in the cold regions. Low Cr alloys like Hastelloy N (7%Cr) were developed for use in FLiBe.[39] Because of the relatively low Cr contents in ODS ferritics, 700°C would be a conservative limit of the maximum use temperature. Experimental work should be done to further explore the limit.

iii. Helium

Perhaps the most likely concept for ODS ferritics would be a solid breeder coupled with He cooling. This would allow higher operating temperatures consistent with the creep properties. Impurities in the He could result in oxidation to form a Cr-rich surface oxide. This has not been investigated yet, but long term (≤10,000h) air oxidation studies suggest that the oxidation resistance of ODS Fe-Cr-W alloys is very good at 700°-800°C. Accelerated attack, likely due to the relatively low Cr contents, was observed at 1000°C. A much higher parabolic rate constant was measured for ODS Fe-13Cr-3W at 1000°C compared to commercial ODS Ni-20Cr, Figure 9. Even at 900°C, a Fe-12Cr-3W ODS alloy showed breakaway attack (i.e. rapid mass gain due to iron oxide formation) after only 7,500h,. Figure 10. The onset of breakaway attack suggests that the alloy was sufficiently depleted in Cr that in could no longer form a protective Cr-rich scale. For the current class of alloys, the maximum operating temperature would likely be ≈850°C. However, slightly increasing the Cr content could increase that number.

iv. Beryllium

If Be components are needed in the blanket, there is a possible solid state reaction that could occur between Fe and Be and a low temperature eutectic (1165°C) on the Fe-

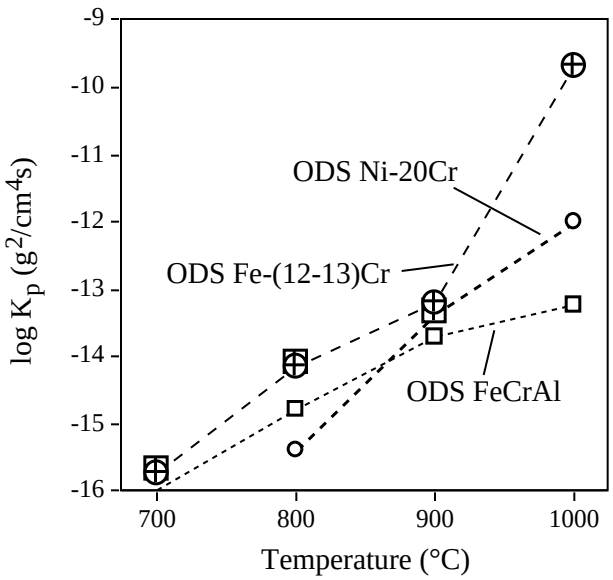

Figure 9. Parabolic rate constants based on 1,000-10,000h exposures for several ODS alloys.

rich side of the Fe-Be phase diagram.

V. SILICON CARBIDE COMPOSITES

Silicon carbide composites are being explored as a higher temperature option to most metals.[10] A variety of issues, including compatibility remain to be addressed with these materials.

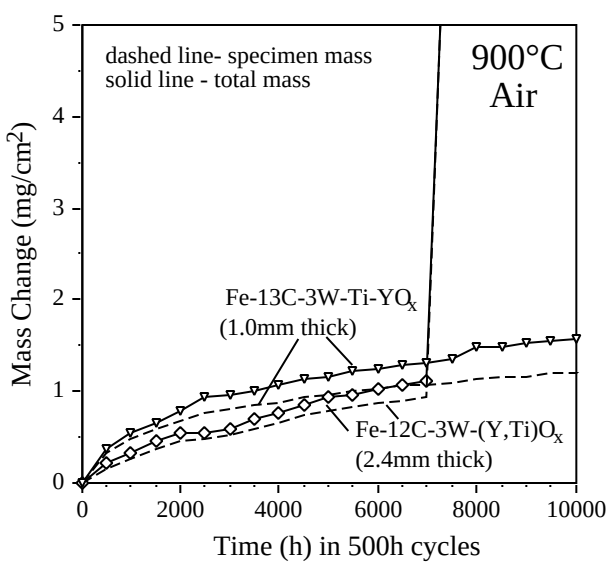

Figure 10. Mass gain during 500h cycles at 900°C in laboratory air for several ODS ferritic alloys. The high mass gain for ODS Fe-12Cr-3W after 7,000h indicates the onset of breakaway oxidation.

Reactions with solid breeding materials have been investigated at 700°-900°C with a Li_4SiO_4 coating used to reduce potential interactions. A reaction of SiC with Be to form Be_2C and Si was observed at 900°C.[39] The only liquid metal compatibility testing for SiC composites was a static test in Pb-Li at 800°C.[10] Additional compatibility tests in Pb-Li need to be completed to determine the degradation mode and an upper temperature limit for this concept.

The high temperature stability of these composites has improved in recent years, due to better processing techniques and fibers with better high temperature durability. However, the fiber-matrix interface region in particular is potentially corrosion susceptible under conditions where the remainder of the composite is stable.[40-41]

VI. CONCLUSIONS

Examples of high temperature compatibility issues of current concern have been highlighted for vanadium alloys, MHD coatings, ODS ferritic alloys and SiC composites. In most cases, the compatibility issues are being addressed in a limited fashion, with MHD coatings currently having the highest priority in the U.S. materials program. Many issues remain unknown or are speculative because compatibility testing, especially with flowing liquid metals or molten salts, has not been conducted on these newer classes of materials. Thus, the extent of any potential corrosion problems and the applicability of existing lifetime models cannot be fully assessed at this time.

There is a great need for experimental results for two reasons. First, data are required for development and verification of appropriate corrosion models and second, to focus the current program on the most viable blanket concepts - a prime example being MHD coatings. If MHD coatings cannot be developed, then continued research on vanadium alloys will have questionable value due to a lack of a viable blanket concept.

VII. ACKNOWLEDGMENTS

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