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DEVELOPMENT OF INSULATING COATINGS FOR LIQUID METAL BLANKETS*

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Abstract

It is shown that self-cooled liquid metal blankets are feasible only with electrically insulating coatings at the duct walls. The requirements on the insulation properties are estimated by simple analytical models. Candidate insulator materials are selected based on insulating properties and thermo-dynamic consideration. Different fabrication technologies for insulating coatings are described. The status of the knowledge on the most crucial feasibility issue, the degradation of the resistivity under irradiation, is reviewed.

1. Introduction

Lithium containing liquid metals i.e. pure lithium or the eutectic lead lithium alloy Pb-17 at % Li (Pb-17Li) are attractive breeder materials in fusion reactor blankets. They have a number of inherent advantages compared to solid breeder materials; such as high tritium breeding ratio without the need for an additional neutron multiplier (beryllium), high thermal conductivity, immunity to

irradiation damage, and the possibility to bring the breeder material outside the blanket for tritium extraction. The most simple blanket design is achieved if the liquid metal serves not only as a breeder but at the same time as a coolant, transporting the heat from the blanket to the external heat exchangers. Such self-cooled blankets are characterized by a simple geometry with a small number of large ducts. The main challenge in designing self-cooled blankets is to deal with the strong influence of the magnetic field on the liquid metal flow. If the flow direction is perpendicular to the field, a potential difference across the duct is induced in the liquid metal. This can cause a large electrical current flow if the potential difference is short-circuited by the duct walls. An electrical current flowing perpendicular to a magnetic field results in a mechanical force which is the reason for the magnetohydro-dynamic (MHD) pressure drop. This is a feasibility issue for self-cooled blankets because it could cause high internal pressure and consequently large mechanical stresses in the duct walls if there is no electrical insulation between the load carrying walls and the flowing liquid metal. The most effective way to minimize the current flow and the pressure drop is to apply an electrically insulating coating on the inner surface of the ducts, avoiding leaking of current from the liquid metal into the duct walls. This, however, requires an insulator material with a high electrical resistance, compatible with the liquid metal, and withstanding the high irradiation flux in a fusion blanket. The requirements on such coatings, candidate materials, fabrication technology and the necessary test program are the subjects of this paper.

2. MHD issues in self-cooled blankets

There are three groups of MHD-issues involved in the design of self-cooled blankets:

- a) pressure drop
- b) flow partitioning between parallel channels
- c) velocity distribution in channels

2.1 MHD pressure drop

The total pressure drop in a blanket determines the maximum internal pressure and consequently the mechanical stresses in the duct walls. A simple example shows, that in tokamaks with a high magnetic field such as ITER even a thin conducting liner in the cooling ducts would result in an intolerably high pressure drop. This shall be explained for a typical poloidal cooling duct and the boundary conditions of ITER as shown in fig. 1.

Fig. 1 Approximated geometry of a poloidal duct

A conductive steel-liner at the duct walls in contact with the liquid metal would lead to a MHD pressure drop of roughly 8 MPa for a liner thickness of 0.1 mm which is a minimum from corrosion and fabrication stand point. This intolerable high pressure drop shows clearly, that insulating coatings in contact with the flowing liquid metal are required.

A lower limit for the pressure drop would be achieved with perfectly insulated walls. In this case the pressure drop can be described [1] with the equation

$$\Delta p = v \cdot B \cdot L \cdot \sqrt{\sigma \cdot \eta} / a \quad (1)$$

where σ = electrical conductivity of the liquid metal

η = viscosity of the liquid metal

(v, B, L, a see fig. 1)

Substituting the dimensionless Hartmann number

$$M = B \cdot a \cdot \sqrt{\sigma / \eta} \quad (2)$$

leads to

$$\Delta p = \frac{\sigma \cdot v \cdot B^2 \cdot L}{M} \quad (3)$$

and the dimensionless pressure gradient

$$\frac{\delta p}{\delta x} = - \frac{\Delta p / L}{\sigma \cdot v \cdot B^2} = - \frac{1}{M} \quad (4)$$

The dimensions and boundary conditions shown in fig. 1 lead with $\sigma = 3.3 \cdot 10^6$ S/m and $\eta = 4.5 \cdot 10^{-4}$ Pa · s (lithium, 400 °C) to

$$M = 10^5$$

and

$$\Delta p = 0.22 \text{ MPa.}$$

Perfectly insulating coatings would result in such a low pressure drop. The question is which deviations from this pressure drop can be allowed in technical systems, taking into account the finite resistance of real insulators and their degradation by cracks, flaws and the irradiation. From a mechanical stand point, a pressure drop of the order of 2 MPa seems acceptable. The influence of the finite

resistance of insulating coatings on a perfectly conducting wall has been investigated by Bühler and Molokov [2]. Their result is shown in fig. 2 where the nondimensional pressure drop $K = -\delta p / \delta x$ is plotted versus the nondimensional coating resistance κ for three different Hartmann numbers. κ is defined by the equation

$$\kappa = \rho_i \cdot \delta_i \cdot \frac{\sigma}{a} \quad (5)$$

with ρ_i = resistivity of the coating

δ_i = thickness of the coating

Fig. 2 MHD Pressure drop as a function of the coating resistance

The figure shows that for large values of κ (that means for high coating resistance) the pressure drop is independent of the resistance and can be correlated to the Hartmann number M by eq. (4)

$$K = -\frac{\delta p}{\delta x} = \frac{1}{M}$$

which is valid for perfectly insulated ducts. For smaller values of the coating resistance the pressure drop depends on the actual resistance. If the allowable pressure drop is specified in this case, the required coating resistance could be determined with the help of fig. 2. Unfortunately, a blanket operation in this region would imply that changes in the resistance (for example caused by irradiation) cause changes in the pressure drop. This, however, would influence

the flow partitioning between parallel channels as explained in the following section.

2.2 Flow partitioning between parallel channels

In general there are a number of parallel channels arranged between inlet and outlet manifold of a blanket segment. This implies that the pressure drop is identical for all these channels and the flow rate in each channel depends on the individual flow resistance. If the flow resistance depends on the conductivity of the insulating coating, this resistance could not be predicted well enough and may vary with time. Therefore, the flow partitioning could change, leading to local overheating in the channels with reduced flow rate. These unpredictable changes have to be avoided which requires that the pressure drop be independent of the actual electrical resistance of the coating. The only way to achieve this is to specify a coating resistance high enough to obtain a pressure drop very close to the one in a perfectly insulated duct. This means that the required resistance of the insulating coating is determined by the requirement of predictable flow partitioning between parallel channels rather than by an upper limit of tolerable pressure drop.

2.3 Velocity distribution in channels

The knowledge of velocity distribution in cooling channels is important for the determination of the heat transport properties. This distribution is determined by the magnetic field and the conductivity of the duct walls. Conducting walls would lead to completely different velocity fields than perfectly insulated duct walls. In a large transition zone between the two extremes the velocity distribution depends

on the actual resistance of the walls [2]. It has been already explained that insulating coatings have to provide a high electrical resistance between liquid metal and wall making leakage currents negligibly small. In this case the influence of the coating resistance on the velocity field can be neglected and the velocity distribution can be determined for the boundary condition of "perfect insulation".

3. Requirements on the electrical resistance of coatings

It has been shown in the previous section that insulating coatings have to lead to conditions very close to the ones in a perfectly insulated duct. The current flow in such a duct is shown in fig. 3 . This flow pattern shall be compared to a case, where the finite resistance of the insulating coating allows for a certain leakage current from the liquid metal region into the duct walls (see fig. 4). The requirement is, that this leakage current I_{leak} assumes a small fraction only of the current I in a perfectly insulated duct.

Fig. 3 Current flow in a perfectly insulated duct

Fig. 4 Leakage current flow

The necessary insulation quality shall be determined for the example shown in fig. 1, assuming fully developed flow in constant magnetic field. For this conditions the flow velocity v is uniform over the entire cross section with the exception of thin boundary layers ("slug flow"). The potential difference E induced by the magnetic field B is then

$$E = B \cdot v \cdot 2b \quad (6)$$

This potential difference leads to an electrical current I which results in a force F acting against the liquid metal flow

$$F = I \cdot B \cdot 2b \quad (7)$$

The force F causes the MHD pressure drop

$$\Delta p = \frac{F}{A} = \frac{F}{2a \cdot 2b} = \frac{I \cdot B}{2a} \quad (8)$$

Combining eq. (8) with eq. (1), describing the pressure drop in a perfectly insulated duct, results in

$$I = 2 \cdot v \cdot L \cdot \sqrt{\sigma \cdot \eta} \quad (9)$$

The quotient between the induced voltage E and the flowing current I describes the internal resistance in the liquid metal

$$R = \frac{E}{I} = \frac{B \cdot b}{L \cdot \sqrt{\sigma \cdot \eta}} = \frac{b \cdot M}{a \cdot L \cdot \sigma} \quad (10)$$

This internal resistance shall be used to quantify the required resistance of insulation coating. An imperfect coating opens an additional current path, leading to an increased pressure drop.

$$I_{total} = I + I_{leak} \quad (11)$$

$$\Delta p_{total} = \Delta p + \Delta p_{leak} \quad (11a)$$

$$\frac{\Delta p_{leak}}{\Delta p} = \frac{I_{leak}}{I} = \frac{R}{R_{leak}} = a_{leak} \quad (11b)$$

For a given tolerable increase in pressure drop (for example $a_{leak} = 0.1$) the minimum required resistance R_{leak} is

$$R_{leak} = \frac{R}{a_{leak}} = \frac{1}{a_{leak}} \cdot \frac{b}{a} \cdot \frac{M}{L \cdot \sigma} \quad (12)$$

R_{leak} is the total resistance in a leakage current loop.

This resistance shall be quantified for a few typical examples of imperfectly insulating coatings.

3.1 All walls are coated with a layer characterized by the product resistivity ρ_i x layer thickness t_i ;

In a first approximation the leakage resistance is given by the resistance of the coatings on the two side walls [3]

$$R_{leak} = 2 \frac{\rho_i \cdot t_i}{2 \cdot a \cdot L} \quad (13)$$

Combining Eq. (12) and (13) leads to

$$\rho_i \cdot t_i = \frac{1}{a_{leak}} \cdot b \cdot \frac{M}{\sigma} \quad (14)$$

Equation (14) shall be applied to the example shown in fig. 1

If the pressure drop is allowed to become 10% higher than the one in a perfectly insulated duct, the coating has to provide a resistance of at least

$$\rho_i \cdot t_i = \frac{1}{0.1} \cdot 0.05 \text{ m} \cdot \frac{10^5}{3.3 \cdot 10^6 \text{ S/m}} = 0.015 \Omega \cdot \text{m}^2$$

3.2 Leakage current flowing through cracks in the side wall coatings

The most severe case would be if the liquid metal in the cracks provides for a direct current path between the liquid metal and the conducting wall. The assumption that in this case the only resistance in the leakage current loop is provided by the liquid metal in the cracks results in

$$R_{leak} = 2 \cdot \frac{t_{LM}}{\sigma \cdot A_{crack}} \quad (15)$$

with t_{LM} = depth of the liquid metal filled crack

A_{crack} = cross section of the liquid metal filled cracks

Combining Eq. (12) and (15) leads with

$$f = \frac{A_{crack}}{A_{sidewall}} = \frac{A_{crack}}{2a \cdot L} \quad (16)$$

to

$$f = \alpha_{leak} \cdot \frac{t_{LM}}{b} \cdot \frac{1}{M} \quad (17)$$

If as an example the values

$$\alpha_{leak} = 0.1$$

$$t_{LM} = 10 \cdot 10^{-6} \text{ m}$$

are used for the case shown in fig. 1, the allowable ratio between crack surface to total side wall surface would be

$$f = 2 \cdot 10^{-10}$$

It is obviously not reasonable to assume such a small ratio. The situation changes completely if there is an additional resistance in the current loop, provided by one of the following mechanism:

- Cracks are not completely filled with liquid metal
- Not perfect wetting in the cracks, resulting in contact resistance
- Resistance of the conducting wall in the vicinity of the crack
- Contact between liquid metal and surface of the conducting wall leads to the growth of a new insulating layer.

Especially the fourth mechanism which is called self-healing (see section 6.2) is important in the assessment of the feasibility of coatings.

If the cracked area assumes the ratio f of the total sidewall surface and is covered with a new layer characterized by the product $\rho_c \cdot t_c$, the required insulation resistance is

$$\rho_c \cdot t_c = \frac{1}{a_{leak}} \cdot f \cdot b \cdot \frac{M}{\sigma} \quad (18)$$

A fraction $f = 0.01$ for example would obviously require a resistance at the cracked surface a factor 100 smaller than the one determined for the entire coated surface (compare Eq. (14) and (18)).

3.3 Perfect coatings at most of the surface area, but a few spots at the side walls with reduced resistance

The resistance at these spots is characterized by $\rho_s \cdot t_s/A_s$. It is important to mention that such spots would not influence the pressure drop if restricted to one of the two side walls. Even a completely uninsulated side wall would not increase the pressure drop if it is perfectly aligned with the magnetic field. A leakage current is possible only if there is a path at the second side wall too. This is especially important for the first wall cooling channels because imperfections are more likely to occur at the highly stressed first wall than at the second wall. In the following it is assumed that there is one spot at each of the two side walls with a resistance $\rho_s \cdot t_s/A_s$.

This leads to the leakage resistance

$$R_{leak} = 2 \cdot \frac{\rho_s \cdot t_s}{A_s} \quad (19)$$

combining Eq. (12) and (19) results in

$$\frac{\rho_s \cdot t_s}{A_s} = \frac{1}{a_{leak}} \cdot \frac{b}{2a \cdot L} \cdot \frac{M}{\sigma} \quad (20)$$

If a pressure drop increase of 10% is allowed for the case shown in fig. 1 the minimum resistance at the two spots would be

$$\frac{\rho_s \cdot t_s}{A_s} = \frac{1}{0.1} \cdot \frac{0.05 \text{ m}}{2 \cdot 0.1 \text{ m} \cdot 12 \text{ m}} \cdot \frac{10^5}{3.3 \cdot 10^6 \text{ S/m}} = 0.0063 \Omega$$

If one of the two side walls has a much smaller resistance and the total resistance is concentrated in one spot, the required resistance of this spot would be twice as high, i.e., $\rho_s \cdot t_s / A_s = 0.0126 \Omega$.

Such a resistance can be provided, for example, by the combinations of A_s , ρ_s , and t_s as shown in tab. 1.

The required resistivity values as shown in tab. 1 are really low when compared, for example, with the resistivity of at least $10^9 \Omega \cdot \text{m}$ for pure unirradiated alumina at 400°C . This high value leaves a large margin for degradation of the resistivity by impurities and by irradiation.

Table 1. Required insulation to provide the resistance $\rho_s \cdot t_s / A_s = 0.0126 \Omega$

Spot Size $A_s [\text{m}^2]$	Insulating Properties $\rho_s \cdot t_s [\text{ohm} \cdot \text{m}^2]$	Resistivity for $t_s = 0.1 \mu\text{m}$ $\rho_s [\text{ohm} \cdot \text{m}]$
$10^{-6} (1 \text{ mm}^2)$	$1.26 \cdot 10^{-8}$	0.126
$10^{-4} (1 \text{ cm}^2)$	$1.26 \cdot 10^{-6}$	12.6
$10^{-2} (1 \text{ dm}^2)$	$1.26 \cdot 10^{-4}$	$1.26 \cdot 10^3$
$1 (1 \text{ m}^2)$	$1.26 \cdot 10^{-2}$	$1.26 \cdot 10^5$

3.4 Localized imperfections in the coating leading to a larger leakage current

The previous analysis is valid only for imperfections resulting in a leakage current small compared to the total current in the liquid metal. In this case the main current pattern shown in fig. 3 remains basically unchanged. If there are well conducting spots at the side walls, the whole structure may change. This case has been analysed by Bühler [4, 5], using a 3D-MHD code based on the core flow approach [6]. In this calculation the resistance of two spots at opposite locations on the sidewalls has been varied. The results of the code calculations agree well with the analytical solution (Eq. 20) as long as the resistance is large enough to limit the increase in pressure drop to about 20%. For smaller resistances the analytical solution overestimates the pressure drop. In the extreme case, when there are two spots with zero resistance, the code predicts an increase in pressure drop by a factor of 2.5 for the example shown in fig. 1. In the analytical model a zero resistance would lead to an infinitely high pressure drop.

3.5 Equilibrium between generation and healing of imperfections

It became obvious in the previous section that self-healing of coatings is mandatory if the generation of cracks or flaking of the insulating layer cannot be excluded. Such damages are likely because the ceramic layer has an expansion coefficient different from the substrate, low tensile strength and ductility and has to survive many thermal cycles. Fortunately, the insulation requirements for damaged parts of the coating are not high if the damages are restricted to small fractions of the duct surfaces.

Very thin new layers on damaged parts of the surface can provide sufficient insulation. This points to the potential of self-healing. Self-healing requires a metallic substrate below the insulating ceramic which can react with a gas

dissolved in the liquid metal. If the ceramic layer develops cracks, the substrate will be exposed to the liquid metal. The reacting gas can diffuse into the substrate, generating a new ceramic layer. Diffusion controlled formation of layers can be described by the parabolic growth rate

$$\left(\frac{t_A}{t_B} \right)^2 = \frac{\tau_A}{\tau_B} \quad (21)$$

with τ_A = time to generate a layer of thickness t_A

τ_B = time to generate a layer of thickness t_B

If for example a layer with a thickness t_A is sufficient to insulate the entire walls and can be generated in 10^6 seconds (12 days), the time required to grow a layer with

$t_B = 0.01 t_A$ (sufficient for $A_{\text{crack}}/A_{\text{side wall}} = 0.01$) is

$$\tau_B = \left(\frac{t_B}{t_A} \right)^2 \cdot \tau_A = 100 \text{ seconds}$$

Considering this fast healing process it is very unlikely that there are major imperfections simultaneously at both side walls. The question is, if any equilibrium will be reached where damages are healed as fast as new damages are generated.

Any interaction between a leakage current and the growth of a new insulating layer is neglected here. In a real case, however, the growth rate of a new layer at the location of an imperfection may be influenced by the high electric field at the

tip of a crack as well as by increased temperatures caused by locally generated ohmic heat [5].

For cyclic operation it can be assumed that cracks or other imperfections are generated at a constant rate

$$c = \frac{da}{dt} \quad (22)$$

with a = surface area of a metallic sublayer exposed to the liquid metal by cracking or flaking.

τ = operating time

For a parabolic growth rate the thickness of the new layer can be described by the equation

$$t(\tau) = K \cdot \sqrt{\tau} \quad (23)$$

K is a constant to be determined by experiments. Its temperature dependence can be described by the equation

$$K = A \cdot \exp(-B/T) \quad (24)$$

with T = operating temperature

A, B = constants determined from experiments

Each crack opens a path for a leakage current from the liquid metal into the conducting wall. It is assumed that a crack does not contribute to the leakage current any more if it is covered by a new layer with thickness t_H grown in the

time τ_H . With this assumption the equilibrium resistance of all unhealed cracks can be determined by an integration over all cracks and is described by the equation

$$R_{leak} = \frac{\rho \cdot K}{2 \cdot c \sqrt{\tau_H}} \quad (25)$$

The equivalent thickness of all unhealed layers is

$$\bar{t} = \frac{t_H}{2} \quad (26)$$

A combination of eq. (20), (23) and (25) leads to the tolerable crack generation rate

$$c \leq \frac{\rho}{4} \cdot \frac{t_H}{\tau_H} \cdot a_{leak} \cdot \frac{2aL}{b} \cdot \frac{\sigma}{M} \quad (27)$$

or, if normalized by the surface area of the side walls, to

$$c^* \leq \frac{\rho}{4} \cdot \frac{t_H}{\tau_H} \cdot \frac{a_{leak}}{b} \cdot \frac{\sigma}{M} \quad (28)$$

Equation (28) shall be applied to the example shown in fig. 1 with the assumption

$$\rho = 10^4 \Omega m$$

$$t_H = 10^{-6} m$$

$$\tau_H = 10^6 s (12 \text{ day})$$

$$\sigma = 3.3 \cdot 10^6 S/m$$

$$a_{leak} = 0.1$$

In this case the tolerable crack rate, leading to 10 % increase in pressure drop, is

$$c^* = 0.014 \frac{1}{d} \approx 1 \% / day$$

4. Candidate Coating Materials

Extensive thermodynamic calculations have been made to evaluate potential candidates that are (a) chemically compatible in liquid lithium environment and (b) possess adequate insulating characteristics for use as a coating on the first wall and blanket structural material. A review of available information on the electrical resistivity values for several oxides, nitrides, and oxynitrides shows that a number of oxides (e.g., CaO, MgO, SiO₂, Al₂O₃, MgAl₂O₄) and nitrides (e.g., AlN, Si₃N₄) have resistivities $> 10^5$ Ohm-m at temperatures below 600 °C. It has been shown in section 3.1 that the product of insulator coating electrical resistivity and thickness should exceed a nominal value of 0.01 Ohm m² under operating conditions. This translates to a minimum resistivity value of 10^4 Ohm-m for a coating thickness of 1 μm or a resistivity of 10^3 Ohm-m for a coating thickness of 10 μm. Based on the resistivity values of materials mentioned above, a coating layer below 1 μm in thickness of any of these materials would be adequate from the insulating standpoint, provided the resistivity is not reduced during operation, i.e., by irradiation.

Based on chemical compatibility in liquid sodium-potassium (NaK) alloy, materials such as Al₂O₃, CaO, MgO, and Y₂O₃, and nitrides such as AlN and Si₃N₄ will be acceptable. Main candidate for use in the lead-lithium alloy Pb-17Li is Al₂O₃. On the other hand, Al₂O₃ is not stable in liquid lithium environment and other candidate oxides and nitrides need to be applied. While the bulk oxides mentioned

above are potential candidates, it is difficult to apply them with sufficient bonding onto the first wall structural material such as Type 316 stainless steel, martensitic steel such as MANET, and V-5wt.% Cr-5wt.% Ti alloy so that the coating will be intact for long times under thermal cycling conditions.

The primary candidate for application in Pb-17Li and NaK environment is Al_2O_3 and the prime candidate for use in lithium environment is AlN . The thermodynamic stability of several of the nitrides as a function of temperature is shown in fig. 5. Superimposed in this diagram are the lines corresponding to different concentrations of nitrogen in lithium.

Fig. 5 Thermodynamic stability several nitrides compared with different levels of nitrogen concentration in lithium

It is evident that AlN will be stable in lithium with a wide range of nitrogen concentrations while Si_3N_4 will be stable up to about 400 °C in lithium containing >300 ppm nitrogen. The calculations show that Ti in the V-alloy can also form nitride in the same lithium environment but the TiN is not viable as an electrical insulator. As a result, it is desirable to aluminize the surface regions of the channel-wall and nitride the surface aluminum to form the insulating layer. Further, Al is favored because a reservoir of Al can be built into the alloy surface by different techniques and if the coating layer cracks/spalls, the aluminum enriched surface could be renitrided by nitrogen in lithium and reoxidized by oxygen in Pb-0.7 wt.%Li or NaK.

5. Fabrication Technology

Several possible approaches are being examined to develop Al_2O_3 coating (for Pb-17Li and NaK application) and AlN coating (for Li application) on the structural material. Some of these are: (a) pre-aluminize the surface of the material by a (pack) diffusion process and subsequently convert it to oxide or nitride in an external gas atmosphere; (b) a physical vapor deposition process with and without bond coats; (c) chemical vapor deposition process at temperatures between 600 and 900 °C; (d) a low temperature method involving sequential reactions; (e) pre-aluminize the surface of the alloy and convert it to nitride in high-nitrogen lithium environment; (f) pre-expose the material to liquid aluminum and convert it to nitride in lithium; (g) in-situ formation of coating in lithium with high thermodynamic activities for aluminum and nitrogen; and (h) pre-aluminize specimens of structural material and nitride it using nitrogen cover gas during lithium exposure.

The pack process is a well established approach to cover stainless steels and nickel base alloys with layers of an intermetallic phase [7]. The substrate materials are contacted and heated for 4-12 hours at temperatures around 900 °C with a pack of powders. The composition of such powders (e.g., 65 wt.% Al_2O_3 , 33 wt.% Al, 2 wt.% NH_4Cl) provides the packing with metallic aluminum, alumina as filler material, and NH_4Cl as activator. The amount of aluminum can be reduced by partly replacing with nickel.

The aluminum deposited on the substrate surface diffuses into the subsurface regions of the material, where it forms intermetallic phases as aluminides of iron or nickel. Since the substrate materials are heated to temperatures close to the annealing range for times sufficient to cause solution processes in the matrix, the materials need a final treatment in order to get the optimized structure. The aluminide layers reach thicknesses of 0.025 to 0.20 mm, depending on the composi-

tion of the substrate materials. Figure 6 shows the elemental concentration profiles for Al, V, Cr, and Ti in a V-5Cr-5Ti alloy sample after aluminizing treatment [8].

Fig. 6 Depth profiles for Al, V, Cr, and Ti for an aluminized specimen of V-5Cr-5Ti alloy

Similar information has been developed for aluminizing treatment on stainless and martensitic steels. The diffusion processes lead to layers of very good adhesion to the substrate. The high temperature of the formation process creates layers which develop compressive stresses at lower temperatures, thus the layers do not contain cracks after the completion of the preparation. Results on the effects of Al_2O_3 coating (developed via pack process and subsequently oxidized at elevated temperature) on pressure drop in NaK environment is presented in a companion paper at this conference [9].

The aluminizing of stainless and martensitic steels and also vanadium and its alloys by means of the "HOT-DIP" process covers the substrate materials at about 800 °C in 1-2 minutes with a layer of solid solutions of the substrate metals in Al or of aluminides of the alloying elements [10]. The process requires clean surfaces of the structural materials in order to get a good wetting and well adherent layers. The adherence can be improved by a thermal treatment of the couples at 500 to 600 °C. The thermal treatment may result in pores or cavities in the outer part of the aluminide layer. The aluminide layer consists of two parts, the inner one being in close contact with the substrate by means of diffusion bonding. The outer one contains pores and cavities due to diffusion effects similar to the Kirkendall effect. The complete layers have a thickness of about 0.2 mm, the inner ones about 0.12 mm. The aluminide layers have to be oxidized in order to get an insulating surface layer. Figure 7 shows the oxidized aluminide layer on a specimen of MA-

NET steel. The aluminizing of vanadium and the alloy V-1Si-3Ti required a temperature of 810 °C and a dipping time of up to 20 minutes [11].

Fig. 7 Metallographic cross section of the oxidized aluminide layer
(air, 30 h at 950 °C)

The aluminide layers had a larger thickness on the Ti bearing alloy, the layer contained about 25 at%V and 0.25 at%Ti. The aluminide layers were well adherent to the substrate due to the interdiffusion of components as evidenced by metallographic examinations (see fig. 8).

Fig. 8 Metallographic cross section of the aluminide coating
on the alloy V-1Si-3Ti

The "HOT-DIPPING" process might be applied for covering the complete blanket structure in a way in which liquid aluminum may be pumped through the heated blanket segment at about 800 °C to have a resident time of about 2 minutes. Welded structures have to be descaled before using the covering procedure. Inspection methods convenient for the use in such complicated structures have to be developed. The powder pack process may cause some difficulties, if it should be applied for the covering of complete blanket structures with aluminide layers. A close contact between the substrate material and the powder pack is necessary for the formation of the layers. The filling of the blanket structure and the densification of the powder pack need further development of the technology for this particular application.

6. Testing of Coatings in Liquid Metals

6.1 Kinetics of coating development

6.1.1. Formation of alumina scales

Since the aluminides could not be oxidized by electrochemical procedures, the high temperature oxidation seems to be the only way to form the insulating Al_2O_3 scales [7]. The oxidation process requires a temperature of at least 900 °C, at which the martensitic structure of MANET steel remains stable. Kinetic studies were performed in air and $\text{H}_2/\text{H}_2\text{O}$ gas mixtures at this temperature. These studies showed that layers of 0.005 to 0.01 mm thickness may be obtained in such atmospheres after oxidation time of 10-30 hours. The insulating character of the alumina layer depends on the completeness of the covering of the metallic phase. Such completeness may be reached, if the surface of the aluminide layers would be of sufficient quality. The alumina layers on aluminide/MANET steel substrate were stable against corrosion in flowing Pb-17Li at 450 °C for 10000 hours [12]. Oxidation of the aluminide cover on the inner side of the blanket structure might be performed in situ after producing a reliable aluminide layer. The oxidation process needs, however, optimization in order to obtain alumina layers of the required insulating properties. An in-situ examination method for quality control of the insulating layer is yet to be developed.

6.1.2. Formation of AlN layers

AlN can be produced by nitridation of aluminized layers on the structural material either externally in gas phase or in-situ in lithium. Based on the stability of the AlN, a nitrogen concentration of >100 ppm in lithium will be sufficient to form AlN layer. Based on available data on nitridation of Al containing alloys which form AlN layers it is estimated that AlN growth rates at 500 and 400 °C will be 0.07 and 0.035 $\mu\text{m/h}$. At present, no kinetic data is available for the growth of AlN layers in liquid metal environment.

6.2 Chemical compatibility and self healing

Aluminized layers have been found to be stable in lithium environment at a temperature of 400 °C in a recirculating lithium loop [13]. However, the kinetics of layer growth and chemical/mechanical integrity of the layer needs to be determined, especially at temperatures in the range 250 - 400 °C of interest in ITER application. Bulk samples of AlN have been exposed to liquid lithium environment at 400 °C and were found to be intact after 100 h exposure [14]. Further, Al transport/deposition to a specimen surface was achieved by additions of 2-5 wt.%Al to liquid lithium [15]. This indicates that if the externally preformed AlN layer cracks or spalls in flowing lithium environment, in-situ repair or self healing of the layer is possible by control of the activities of aluminum and nitrogen in lithium environment. The ongoing experiments in liquid metals such as NaK, lithium, and Pb-Li will determine the growth kinetics and chemical composition for these layers.

Further, experiments are underway to determine the repair procedures for reformation of layers in-situ via control of thermodynamic activities of nitrogen and aluminum in lithium, nitrogen in cover gas, and exposure temperature.

7. Electrical conductivity during irradiation in candidate insulating layers

The purpose of the barrier layer is to provide electrical insulation between the moving liquid metal coolant and the structural material. Aluminum oxide (Al_2O_3) and aluminum nitride (AlN) are primary candidates for this layer. The MHD forces impeding the flow of the liquid metal are considerably reduced with even modest values of electrical resistance in the layer, so the electrical demands on the insulating layer would not seem to be severe. However, many uncertainties regarding the layer thickness and integrity, corrosion, abilities to maintain stoichiometry during use, and radiation effects are causes for concern. The state of knowledge about these issues is addressed below.

7.1 Summary of Radiation Effects

The environment in the fusion blanket consists of both neutron and gamma radiation. The neutron energy spectrum consists of a fission-like spectrum centered near a few MeV but with a large component at 14 MeV. These neutrons cause large, multi-atom displacement cascades in the crystalline lattice and also generate transmutation elements, particularly hydrogen and helium. The gamma radiation causes primarily ionization (electron-hole pairs). Accumulation of both types of radiation leads to changes in the mechanical, physical and electrical properties of the material. The strength of alumina and aluminum nitride are degraded at fluences above a few dpa (displacements per atom, one dpa is approximately 10^{25} n/m²). The thermal conductivity is typically reduced to about 1/3 of its initial value by low fluences, typically 10^{-3} dpa and the electrical conductivity follows a more complicated behavior. The unknown changes in the electrical conductivity are a

primary cause for concern in the candidate insulators. These issues have been discussed in several review papers on the subject [16, 17].

7.2 Mechanical stability issues

The reduction in strength following irradiation in ceramics and other brittle materials is generally recognized to be caused by swelling. Swelling is a result of formation of voids and dislocation structures and is highly dependent on material type, irradiation temperature and type of irradiating particle. The swelling can lead to catastrophic loss of strength at relatively low fluences, such as in BeO and AlN, or in nearly no change at all even at high fluence, such as in magnesium-aluminum spinel (at least in fission reactor irradiation). In brittle materials of anisotropic crystal structure, each grain swells anisotropically and causes microcracking at the grain boundaries. This microcracking is the major cause of strength degradation in fission reactor irradiation [17, 18, 19]. The fusion neutron spectrum will generate hydrogen and helium from transmutation reactions. These gases will undoubtedly cause additional swelling and strength loss, but no data is available to support any predictions of this effect. Clinard, Zinkle and Farnum are currently irradiating oxygen-17-doped alumina and spinel in the HFIR reactor and intend to measure post-irradiation strength. Figure 9 shows the strength of AlN ceramic following reactor irradiation [20] and fig. 10 shows the effect of temperature on swelling in alumina [17]. This is an example of the importance of the material state on irradiation response, because the sudden drop in mechanical strength of commercial AlN reported by Iseki et. al. in fig. 9 was not observed by Dienst in his study of hot pressed AlN [21].

Fig. 9 Bending strength of AlN following fission neutron irradiation
(from [20]).

Fig. 10 Temperature-depending of swelling for five ceramics,
(from [25], fig. 10).

There is relatively little data on the strength of ceramic films following irradiation to fluences that would degrade the strength of bulk materials. Ranken [22] and Ivanov [23] have irradiated plasma sprayed alumina coatings to about 3×10^{24} n/m² without mechanical failure, but bulk alumina retains strength to more than 20 times this fluence. It is likely that fabrication method and coating morphology will play a crucial role in the behavior of barrier coatings for fusion blankets.

7.3 Electrical Conductivity

Electrical conductivity in ceramics is caused by the motion of electrons in the conduction band (electronic conduction) and/or by the migration of ions (ionic conduction). Electronic conduction depends on the equilibrium number of electrons in the conduction band, which in turn depends on the generation rate of free electrons (electron-hole pairs) and the electron-hole lifetime that is dominated by the crystal defect structure (trapping centers). Electron-hole pairs are made by both thermal and ionization processes, so the electronic conductivity depends on the amount of ionizing radiation, the temperature and the concentration of trapping centers for the electrons. The behavior of electronic conductivity to radiation has been well documented [24, 25, 26]. For a fixed concentration of trapping centers the electronic conductivity (σ) may be expressed as:

$$\sigma = \sigma_0 \exp - (E_a/kT) + K_1 \phi^\delta$$

Where E_a is the thermal activation energy, k is Boltzman's constant, T is the temperature, ϕ is the ionizing flux and δ , σ_0 and K_1 are material-dependent parameters. The first term is the intrinsic conductivity of the material and the second term is called radiation-induced conductivity (RIC). The parameter δ is generally between 0.5 and 1.5, most typically, 1.0, but it depends on temperature and the materials prior state [25]. Hodgson has also shown that at least for MgO, δ itself is a function of flux over an extended temperature range [27]. A summary of RIC data for a variety of insulators has been compiled by S. Zinkle of ORNL and is shown in fig. 11.

Fig. 11 Radiation-Induced Conductivity (RIC) in Oxide Ceramics.

Generally, RIC in a high-quality alumina ceramic, for fluxes near the ITER first wall, will be on the order of 1×10^{-6} S/m. At the ITER wall temperature of less than 500 °C, the intrinsic conductivity will be in the order of 10^{-12} S/m [28]. Therefore, we currently believe that RIC itself will not be a serious limitation for blanket applications.

However, recently a type of electrical degradation, called radiation-induced electrical degradation (RIED) has been observed during experiments in which a continuous electric field was applied during the irradiation [29,30]. This effect, which in some cases has been observed to lead to electrical conductivity as high as 10^{-2} S/m [31], depends strongly on temperature, irradiation parameters, electric field and material state. The causes for this effect have not yet been confirmed, but are be-

lieved to be due to enhanced oxygen ion vacancy diffusion and aggregation. A summary of the recent development and current work in the area follows.

7.4 The RIED effect

This effect was first reported in 1989 by Hodgson [29]. Hodgson used electrons of 1.8 MeV energy to irradiate alumina and simultaneously applied a DC electric field of 200 kV/m. At temperatures between about 200 and 600 °C a dramatic increase in electrical conductivity was observed after an incubation irradiation fluence of only 10^{-5} dpa as shown in fig. 12.

Fig. 12 The Radiation-Induced Electrical Degradation effect as first reported by Hodgson.

Subsequently, this effect was confirmed by Pells [32] using 18-MeV proton irradiation and by Shikama [33] using fission-reactor neutron irradiation. While the conductivity increase began between 10^{-4} and 10^{-5} dpa with electron irradiation, it began at about 10^{-3} dpa with proton irradiation and at more than 3×10^{-2} dpa with neutron irradiation.

Hodgson then expanded the measurements to MgO and MgAl_2O_4 spinel, to AC applied fields, using frequencies as high as 126 MHz [34] and to temperatures between about 250 and 530 °C [27]. The effect continues to be important at the AC frequencies as shown in fig. 13. The effect varies with temperature, reaching a maximum efficiency at about 450 °C as shown in fig. 14. Voltage applied during the irradiation has also been shown to be important, a lower limit to the RIED effect

measured by Hodgson to be about 50 kV/m as shown in fig. 15. Hodgson also showed that the fluence required for the onset of RIED is proportional to the square root of the flux [35].

Fig. 13 Observation of the RIED effect at high frequencies (from [27]).

Fig. 14 The effect of temperature on the onset of RIED during electron irradiation of alumina [34] (total dose required to degrade single crystal (SC) and polycrystalline (PC) alumina to 10^{-6} S/m).

Fig. 15 Observed electrical conductivity after a fixed fluence of electron irradiation vs. applied voltage at 450 °C in alumina.

However, some experiments have added confusion to the above result. Ivanov [23] and Ranken [22] irradiated sprayed alumina coatings in fission reactors without observing an RIED effect. However, Ranken's temperature of nearly 800 °C and Ivanov's electric field of 15 kV/m put both of these experiments outside the expected RIED range. Farnum [36] and Kennedy [37] irradiated alumina at 395, 615 and 667 °C with spallation neutrons and found no RIED when the samples were in argon atmosphere but did observe a RIED-like response when the samples were in an evacuated capsule. The fluence achieved in these experiments was less than the fluence required for the onset of RIED under fission-reactor neutron irradiation [32] and Farnum suggested that the effect observed in the evacuated capsules could have been caused by degradation of the capsule atmosphere [36]. Kesternich [38] reported no RIED effect during alpha particle irradiations at 500 °C and 350 kV/m field. Kesternich reported a RIED-like effect that seemed to be related to the vacuum conditions during the experiment and suggested that the RIED effect

could be caused by the growth of carbon film or other surface contaminant during the irradiation. In addition, Jung [39] reported that the absorption spectrum of the carbon film deposited on the surface during RIED experiments nearly duplicates the absorption spectrum expected in alumina for the F and F⁺ centers. Thus, these two papers suggested that a carbon film deposition could explain both the RIED and optical absorption data of Hodgson and others. Zong [40, 41] irradiating in air, observed an RIED effect that saturated at about 10⁻⁶ S/m, the first such saturation reported and well below what other experimenters have reported. Kennedy and Farnum's data in the evacuated capsules saturated too, but at levels above 10⁻⁴ S/m [37, 42].

At the International Conference on Fusion Reactor Materials (ICFRM-6) meeting (Stresa, Italy, September, 1993) the RIED results were discussed at length by all of the above investigators (except X. Zong) with no definite resolution to the apparently conflicting data. Important parameters were identified to be temperature, both ionization and displacement fluxes, applied voltage, material type and atmosphere. There was agreement that flux and atmosphere were important variables that require further exploration and it was agreed that experiments would be conducted with a standard material, temperature and applied voltage at several laboratories to try to isolate the causes for the different results. This "round-Robin" experiment is currently being conducted by Kesternich using helium ions, Hodgson using electrons and Shikama, Farnum and Zinkle using fission reactor neutrons.

With the present state of knowledge we can only state the following information. In certain ceramic materials, including Al₂O₃, MgO and MgAl₂O₄ at temperatures between 200 and 600 °C, with a continuously applied voltage greater than about 50 kV/m under irradiation that causes both ionization and atomic displacements, a dramatic increase in the electrical conductivity is observed at fluences substantially below 0.1 dpa. This effect can increase the electrical conductivity to

at least as high as 10^{-3} S/m and seems to be a function of all of the above variables, as well as the radiation flux and the local atmosphere. Whether the effect is primarily surface controlled or bulk controlled is still unproven. This effect is of great importance to blanket insulator coatings because an MHD voltage of only 1 volt across a barrier film 10 micrometers thick creates an electric field of 100 kV/m. In addition, the expected blanket operating temperature of 300 to 500 °C is well within the RIED temperature range and the film has a high surface to volume ratio making it susceptible to both surface and bulk effects. In addition, unknown environmental effects are likely to be discovered.

7.5 Environmental concerns

There is little that is known about the effect of environment or stoichiometry on the electrical conductivity of candidate barrier coatings under irradiation. Most ceramics show ionic conductivity at high temperature that is caused by the motion of impurities and atomic and structural defects in the lattice [43]. The motion of such defects depends strongly on their electronic charge state that can be readily modified by ionization, displacement damage, oxidation or reduction. These defects are poorly characterized and the effect of ionization state on their contribution as donors, acceptors and ion conductors is not known. Most ceramics show conductivity that leads to electrical breakdown at very high temperatures, but single-crystal alumina stands out as being particularly resistant to these effects [43]. This is one reason for alumina being chosen as a candidate fusion material. However, even in alumina, impurities and the method of preparation cause large variations in the intrinsic conductivity, varying from 10^{-13} to 10^{-6} S/m at 1000 K for virgin material [41].

The fact that impurities in alumina can be used intentionally to reduce RIC during irradiation is well known [23]. In this case impurities act as trapping centers, reducing the electron-hole lifetime. In addition, atomic defects, such as oxygen vacancies are active ionic conductors [44]. Therefore, the oxygen (or nitrogen in the case of AlN) vacancy concentration, the aluminum vacancy concentration, and the concentrations of lithium or sodium impurities that may diffuse into the material from the liquid-metal coolant and the formation of transmutation by-products, may have a strong and unpredictable effect on the onset and extent of RIED in barrier coatings. We expect, based on its high temperature resistance to dielectric breakdown that high-purity alumina will be the most resistant to such effects, possibly being improved with some type of intentional doping. We have little data to predict the performance of aluminum nitride, but its RIC performance is worse than that of alumina [45].

8. Conclusion

Self-cooled blankets in tokamaks with high magnetic fields such as ITER are feasible only with insulating coatings at the duct walls. The resistance of these coatings is determined by the requirement of a predictable flow partitioning between parallel channels. This implies that the pressure drop must not depend strongly on the actual resistivity of the coatings which may vary from channel to channel in a certain range. The only way to achieve this is to have coatings leading to a pressure drop nearly identical to the one in perfectly insulated ducts. A simple analytical model is provided determining the required coating resistance in order to limit the leakage current to a given fraction of the total current. For uniform coatings without any imperfections the required product resistivity $\times$ layer thickness has to be in the order of $10^{-2} \Omega\text{m}^2$. This value corresponds to a resistivity of $10^4 \Omega\text{m}$ for a layer with $1 \mu\text{m}$ thickness. Pure unirradiated alumina for example has a resistivi-

ty of a least $10^9 \Omega\text{m}$ at 400°C , leaving a large margin for degradation by impurities and radiation. The pressure drop caused by localized imperfections in the insulating coatings (so called "bad spots") can be estimated with the same model, provided the resistance in these spots is large enough to limit the increase in pressure drop to roughly 10 - 40%. For smaller resistances the model overpredicts the pressure drop. In this case a three-dimensional computer code can be used to calculate pressure- and velocity distribution.

It should be mentioned that "bad spots" can lead to an increased pressure drop only if occurring on both side walls (walls parallel to the magnetic field) simultaneously. Even a completely uninsulated side wall would not result in a large pressure drop if the other walls are perfectly insulated.

Completely uninsulated spots on both side walls would result in an intolerable high pressure drop. To avoid this, a mechanism is required leading to a new insulating layer at spots where a damaged coating results in a direct contact between liquid metal and conducting wall. The method envisaged for this self-healing is to employ a sublayer between the insulating coating and the duct walls which can react with a gas dissolved in the liquid metal. In this case the growth rate of the new insulating layer is diffusion controlled and can be described by a parabolic law. The equilibrium between the generation of imperfections and the healing mechanism can be estimated with a simple model. A screening process of a number of ceramic materials showed that from a chemical point of view the most promising candidates are aluminium oxide for use in lithium-lead and sodium-potassium, and aluminium nitride for use in lithium. In both systems an aluminium-rich sublayer is employed in order to obtain a potential for self-healing. Methods are under development to coat the candidate structural materials (ferritic steels, vanadium alloys) with this sublayer and to convert a part of the sublayer into alumina or aluminium-nitride. Progress has been made in the fabrication technology of suit-

able layers but the kinetics of self-healing under operating conditions is not yet proven.

The largest uncertainty in the feasibility of insulating coatings, however, is the degradation of the electrical resistance under irradiation. This effect cannot be predicted from current data. We know that the initial state of the material, including its impurity content and defect structure is important. We also know that the environmental conditions expected for the coating makes it susceptible to the RIED effect that can increase conductivity to unacceptable levels. We expect that the liquid metal and the stoichiometry of the coating, including the chemistry of self-healing, will have an important effect on the onset of RIED, but do not know what effect that will be. We expect that alumina will have the best performance of the candidate materials, but currently cannot predict what the performance of alumina, or any other ceramic, will be. We do not know whether the RIED effect saturates, or can be made to saturate, at an acceptable conductivity level. Finally, we do not know what differences will occur between fusion-simulation radiations, such as fusion reactors, electrons and protons, and the fission radiation condition.

In spite of this lack of knowledge, since it is important in the fusion program, relevant data can be acquired relatively quickly if adequate funding is provided. Because the candidate coatings are thin layers, the fusion environment can be simulated at a number of available ion-accelerator sources. Such a simulated irradiation can duplicate both the atomic displacement damage and the transmutation gas production with a combination of self-ion and transmutation-product-ion irradiation. Such screening tests have to be followed by long-term irradiations in fission reactors.

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Figure Captions

- Fig. 1 Approximated geometry of a poloidal duct
- Fig. 2 MHD pressure drop as a function of the coating resistance
- Fig. 3 Current flow in a perfectly insulated duct
- Fig. 4 Leakage current flow
- Fig. 5 Thermodynamic stability of several nitrides compared with different levels of nitrogen concentration in lithium
- Fig. 6 Depth profiles for Al, V, Cr, and Ti for an aluminized specimen of V-5Cr-5Ti alloy
- Fig. 7 Metallographic cross section of the oxidized aluminide layer (air, 30 h at 950 °C).
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