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TOKAMAK First-Wall Coating Program Development

Mark J. Davis, Robert A. Langley, Thomas S. Prevender

Prepared by Sandia Laboratories, Albuquerque, New Mexico 87115
and Livermore, California 94550 for the United States Energy Research
and Development Administration under Contract AT(29-1)-789

Printed August 1977

Sandia Laboratories

SF 2900 Q(7-73)

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Printed in the United States of America and
Available from:

National Technical Information Service
U. S. Department of Commerce
5285 Port Royal Road
Springfield, VA 22161

Price: Printed copy \$4.50; Microfiche \$ 3.00

Issued by Sandia Laboratories, operated for the United States
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TOKAMAK FIRST-WALL COATING PROGRAM DEVELOPMENT

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This work supported by U. S. Energy Research & Development Administration,
Division of Magnetic Fusion Energy.

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SUMMARY AND CONCLUSIONS

SCOPE: The Materials and Surface Science Branch of the Division of Magnetic Fusion Energy commissioned this study to develop a program using coatings to control impurities originating from the first wall of an EPR type of Tokamak reactor. Chemical conversion of a surface is considered a coating in this study.

METHOD: This study was conducted by reviewing EPR design documents and consulting personally with the designers concerning their needs. Surface scientists and materials engineers were also consulted to determine the potential of materials and processes to meet the designers' needs. The authors would like to acknowledge the openness and candor of the individuals who were contacted during this study. It is hoped that the synthesis of their needs and suggestions presented in this development program will, when executed, help provide a solution to the Tokamak plasma impurity problem.

RESULTS

The program was developed by addressing the following questions. Answers are summarized after each question. The selection of tests was based on a current assessment of the plasma impurity problem. Additional experiments in the future may suggest that other tests be added to this program.

What characteristics should the coating have?

- I. Minimal effect on plasma (if coating material is injected into plasma)
- II. Compatibility with substrate
- III. Rework capability
- IV. Minimum retention of tritium (T)
- V. Acceptable life time

What properties are associated with these characteristics?

- I. Minimal effect on plasma
 - A. Coating material should have a low atomic mass (low Z)
 - B. Low sputtering yield under plasma conditions (low S)
 - C. Low chemical erosion rates in a hydrogenic plasma
 - D. Low electrical arc erosion rates

- II. Compatibility with substrate
 - 1. Low interdiffusion
 - 2. Matched coefficient of expansion
 - 3. Lack of brittle intermetallics
- III. Rework capability
 - 1. Recoatable surface after plasma exposure
 - 2. Ability to repair coating in Tokamak.
- IV. Minimum retention of tritium
 - 1. Low solubility for T and no stable hydrides
 - 2. High tritium permeability
 - 3. Minimum T traps
- V. Acceptable life time
 - 1. Low erosion rate (sputtering, chemical erosion, mechanical)
 - 2. Low transmutation rate

What materials are attractive?

Metals with low SZ^η

Refractory high-low Z compounds (e.g., VDe_{12} , TiB_2)

Ceramics and glasses

Oxides

Carbon and Carbides

Borides, Nitrides, Silicides, Beryllides

How could they be produced on the surface?

- 1. Physical deposition - clad, plasma, vacuum
- 2. Chemical deposition - CVD, electroplate
- 3. Cementation - enamel, pack cementation, slurry
- 4. Conversion reaction - oxidize, carburize, nitride

What structural and morphological control is possible?

- 1. Grain size, shape, and orientation
- 2. Texture (crystallographic)
- 3. Surface topography
- 4. Density
- 5. Porosity

How should candidate materials be screened?

1. Low sputtering rate when subjected to 300 eV and 2 keV deuterium (D)
2. Minimum material loss in 1.3×10^{-2} Pa D plasma at 800 K (1800 K)*
3. Adhesion under EPR conditions

How should candidate coating systems be screened?

1. Maximum adhesion under thermal fatigue loading at 1.3×10^{-2} Pa D plasma at 800 K (1800 K)
2. Maximum processing flexibility
3. Reliable rework capability

* Numbers in parenthesis refer to a radiating liner.

PROPOSED DEVELOPMENT PROGRAM

The coating development program recommended has four major parts, an initial screening phase, (0), followed by three development phases, each associated with one of four milestones.

These phases are:

- Phase 0 - Screen material classes, i.e., metals, metal oxides, carbides, etc., and morphologies, plus select dominant evaluating parameters in physical sputtering and chemical erosion.
- Phase 1 - Evaluate representatives of the most promising classes of materials. Develop processes for and evaluate the most promising coatings deposited on coupons of 316SS or liner substrates.
- Phase 2 - Perform process development of the most promising coatings to accommodate and define design details and rework procedures. Evaluation will be carried out in a Tokamak test reactor or appropriate material test facility. Initial transfer of technology to industrial coating suppliers will start in this phase.
- Phase 3 - Conduct industrial coating of full-sized elements of a Tokamak. Evaluation will be in a Tokamak reactor. Completion of final material and process specification will complete the program.

CONCLUSIONS

1. Coatings should be developed for first walls operating up to 800 K and liners operating up to 1800 K in a 1.3×10^{-2} Pa hydrogenic plasma.
2. Insufficient data exists at the present time to select a coating material which will have a minimum effect on plasma operation.
3. A phased development program must be established which will screen potential coating materials using sputtering and chemical erosion tests, develop the most promising systems, test in a Tokamak environment and scale-up to full scale Tokamak components.
4. The materials selection and design of the coatings must be reviewed by reactor designers, surface scientists, and materials and process engineers at the completion of each development phase to determine if progress justifies further development.

I. INTRODUCTION

This study considers the following:

1. The need for a first wall coating in an Experimental Power Reactor (EPR) application.
2. Those factors appropriate to the selection of a coating.
3. Specification of a program to obtain the desired coating(s).

The primary sources of information used in this study were the EPR design reports prepared by ANL,¹ GA,² and ORNL,³ the UWMak I,⁴ II,⁵ and III⁶ reports, and also a series of EPRI reports.^{7,8} Table 1 provides a set of representative EPR design parameters based on the above reports. In addition, discussions were held with scientists and engineers from ANL, GA, ORNL, PPPL, and the University of Wisconsin who participated in writing the above reports.

A common concern of the EPR design community is the conclusion that some means must be employed to protect the inner wall of the stainless steel vacuum vessel from the effects of the plasma and to minimize contamination of the plasma by impurities which come from the wall. Alternative methods of achieving this protection may be divided into mechanical and nonmechanical approaches. The non-mechanical methods consist of the magnetic divertor techniques⁹ and the flowing gas blanket approach.² Mechanical methods employ the interposition of a protective surface between the plasma and the vacuum vessel wall. This study is concerned exclusively with the latter case of an EPR that uses only a mechanical means of protecting the vacuum vessel wall. In addition, we will consider only the case of a thin coating on that surface which is adjacent to the plasma, although it is clearly realized that more than one method might be required to limit the impurity levels in the plasma. This report will not address the problems associated with limiters and beam dumps, although some of the information obtained during the development phases of a coating program will have application to these areas.

There are several advantages to the concept of a coating. Perhaps the primary one is that the coating can be chosen to perform only one function---to minimize plasma contamination. It does not have to be compromised by the need to be a structural material with the attendant criteria of fabricability in thick sections, weldability, high strength, etc. In addition, because of the relatively small amount of material involved in a coating, possibilities are opened up for

Table 1. EXPERIMENTAL POWER REACTOR DESIGN PARAMETERS

	<u>Argonne N. L.</u>	<u>General Atomic</u>	<u>Oak Ridge N. L.</u>
Engineering Parameters			
Major Radius (m)	6.25	4.5	6.75
Aspect Ratio [*]	2.98	4.02	3.0
Magnetic Field on Axis (T)	4.5	3.9	4.8
Poloidal β ^{**}	1.7	2.0	2.1
Plasma Parameters			
Current (MA)	7.6	13.7	7.2
Density ($10^{14}/\text{cm}^3$)	0.7	1.4	0.7
Shape	Circular	Doublet	Circular
Ion Temp (keV)	9.6	8	9
Z_{eff}	1.3	2.0	1.34
Energy Output Electrical (MW)	30.0	14.0	-
T Breeder	Yes	No	Yes
Neutral Beam Injectors	16	16	6

*Aspect Ratio = Major radius/plasma radius

** β = Plasma pressure/magnetic pressure

the use of exotic materials that would otherwise be considered too expensive or scarce. Finally, since only thicknesses of the order of 200 μm are needed, a variety of application techniques may be possible. Some of these are physical or chemical vapor deposition, sputtering, and plasma spraying.

There are two distinct applications for a coating. The first is a coating on the vacuum vessel itself and the second is a coating on a liner within the vacuum vessel. A liner may be needed to protect the vacuum vessel from perforation in the event of an accident such as the loss of control of the plasma. Because of the tritium inventory in the Tokamak and the inaccessibility of the vacuum vessel, a perforation could have very serious health and monetary consequences. In either application, the purpose of the coating would be to minimize plasma contamination by reduction of impurities from the liner/vacuum wall. Several times in this report two temperatures will be given to specify a test temperature where the second temperature of the pair will be in parenthesis. In this notation the second temperature refers to the case in which a liner is employed.

The necessity of reducing the plasma contamination that would be produced by structural materials such as stainless steel if used adjacent to the plasma can be convincingly demonstrated. The introduction of high atomic weight (Z) wall materials into the DT plasma results in radiative losses that at best decrease the efficiency of the plasma "burn" and at worst prevent ignition completely. Radiative losses due to bremsstrahlung and recombination are proportional, respectively, to Z^2 and Z^4 . Hopkins⁷ has calculated the maximum impurity concentration as a function of impurity atomic number to achieve ignition. Fig. 1a shows his results for the case of the General Atomic EPR design. Jensen, et al.,¹⁰ have recently investigated theoretically impurity radiation for typical Tokamak parameters using an "average ion model." Steady-state emission rates were used to investigate the effects of various concentrations of impurities on the net requirements for breakeven, ignition and beam-driven reactor experiments. The results for impurity fraction for ignition at two ion temperatures, 10 and 20 keV, are presented in Fig. 1b. The combination of erosion rates and atomic numbers of structural materials are such that the high Z contamination that they would cause would probably preclude ignition in the Tokamak. Detailed analysis of the magnitude of contamination produced as a function of atomic number and erosion rate indicates that probably only low Z coatings will be effective in

this role.

The following sections of this report consider the expected environment of a coating, candidate materials, and application techniques, a proposed coating development program, and finally, the requirements and implications of the proposed program.

Fig. 1a. Maximum plasma concentration of impurity for which ignition can be achieved.

Fig. 1b. Ignition Criteria. Plot of $n_e \tau$ vs. impurity fraction for various impurities at plasma ion energies of 10 and 20 keV

II. FIRST WALL ENVIRONMENT AND EFFECTS

A. Introduction

The first wall of an experimental power reactor is defined in this report as the first material barrier interfacing the plasma. All emissions from the plasma may interact with this wall: these include D & T fuel atoms, electrons, particles sputtered from the interior surfaces of the Tokamak, primary and scattered neutrons, x-rays, cyclotron radiation, and gamma radiation from the blanket region. These emissions can lead to plasma impurity buildup and deterioration of the first wall through the processes of sputtering, blistering, and chemical erosion. The first wall will also be exposed to a 1.3×10^{-2} Pa hydrogenic plasma.* Chemical reactions of this plasma with the hot first wall can also cause severe plasma impurity problems and first wall deterioration. Material vaporization and flaking of coatings can produce further sources of plasma impurities. These phenomena will be discussed in some detail in the following sections. A major portion of the information has been gleaned from EPR reports and review papers on the various phenomena.^{1,2,3,11,12,13}

First wall parameters taken from the various EPR studies are presented in Table 2. Energy distributions of the neutral flux (50% D and 50% T) have been calculated for a two-component Tokamak and a power reactor.¹⁴ The results are shown in Fig. 2. The energy distribution of the neutrals for an EPR probably lies between the two curves, and depending upon its size and containment time, could be as low as the power reactor curve. The conclusion to be drawn here is that the analysis requires plasma physics information in regimes where experimental data have not yet been obtained, and thus the calculated results are clearly tentative in nature. For example, sharp density gradients typical of unload divertor operation can lead to high plasma edge temperatures, whereas a flatter density profile generally leads to edge temperatures less than a few hundred eV because of neutral gas recycle.¹⁴

It is realized that in present-day machines desorption of oxygen represents a major source of plasma impurities, but it is anticipated that in the next few years this problem will be eliminated and that sputtering, surface chemical

* Hydrogenic plasma is used as a generic term to apply to hydrogen, deuterium, and tritium in both atomic and molecular forms containing excited, unexcited, and ionized species.

Table 2. EXPERIMENTAL POWER REACTOR FIRST WALL PARAMETERS

	Argonne Nat'l Labs	General Atomic Co.	Oak Ridge Nat'l Labs
Material	316 SS	316 SS	316 SS
Area (m ²)	592	-	600
Maximum Temperature (K)	800	900	300
Δ Temperature (K)	100	60	30
Burn Time (Sec)	35	105	100
Cycle Time (Sec)	60	125	115
Life Time (yrs)	10	5	--
Coating	None	None	C
Liner	Yes	Yes	
Material	316 SS	SiC	
Coating	Be	None	
Maximum Temperature (C)	407	1400	
Δ Temperature (C)	50	--	
Life Time (yrs)	4	2	
<u>Neutrons</u>	B = 8 B = 10		
Energy Flux (MW/m ²)	0.35 0.86	0.85	1
Fast n Flux (10 ¹⁷ /m ² -s)	1.7 4.2		
Total n Flux (10 ¹⁷ /m ² -s)	7.0 17	40	
Charge Exchange Neutral (50%D 50%T) Flux (10 ¹⁹ /m ² -s)	2.8 7.0	3	
Spectrum	$\bar{T}$ = 200 eV, Maxwellian		
<u>Alphas</u>			
Fast (10 ¹⁵ /m ² -s)	3.7 5.9	30	
Spectrum	E = 3.5 MeV	E = 3.5 MeV	
Thermalized (10 ¹⁷ /m ² -s)	1.7 4.2	3	
Spectrum	$\bar{T}$ = 200 eV Maxwellian	Maxwellian	
<u>Radiation</u>			
Bremsstrahlung (10 ⁻³ MW/m ²)	7.8 19	--	
Spectrum	$\bar{E}$ = 10 keV, Maxwellian	--	
Synchrotron (10 ⁻³ MW/m ²)	2.2 4.5	--	
Spectrum	Flat (W _c -20 W _c)	--

Fig. 2. Neutral D-T flux to the liner

reactions, bulk radiation damage, and thermomechanical effects will represent the major impurity sources. The remainder of this section will deal with these four aspects.

B. Sputtering (Review of Data)

One of the most important surface data requirements is the knowledge of sputtering yields for incident hydrogen isotopes and helium in the 50 eV - 15 keV range on various samples of pure materials, alloys, and compounds which are contemplated for first walls and first wall coatings. If neutral beam injectors are used, then the energy range of interest should be extended to 200 keV. Most sputtering yields to date have been measured under conditions which are not applicable to a fusion reactor first wall. To be applicable, the measurements must be done on the highly damaged, steady state surfaces in a 1.3×10^{-2} Pa hydrogenic plasma. The sputtering yields need to be known for various temperatures ranging from room temperatures to approximately 1900 K (600 K to 900 K for a first vacuum vessel wall coating and 1300 K to 1900 K for a liner coating).

The field of sputtering has been reviewed up to 1967 in an excellent publication by Carter and Colligon in a book entitled Ion Bombardment of Solids.¹⁵ Subsequent to this publication, many papers have been published on the sputtering yields of heavy ions, but little in the area of light atoms such as hydrogen and helium isotopes. Behrisch¹¹ and Rossing, et al,¹³ have reviewed sputtering and wall erosion mechanisms of first walls of fusion reactors.

At the present time only estimates can be made of the sputtering yield for low energy charge exchange neutrals which impinge on the wall. Measurements by Gronlund and Moore¹⁶ have been made for H^+ , D^+ , H_2^+ , D_2^+ , H_3^+ , D_3^+ , He^+ , N^+ , O^+ , and Ne^+ on silver from 2 to 12 keV. They found that the angular distribution of the sputtered silver followed a cosine dependence about the normal-to-target surface. Their results showed sputtering yield to decrease monotonically with increasing energy from 2 - 12 keV and increase almost linearly with the mass of the projectile ion for the same atomic number, but to have a Z dependence which was somewhat nonlinear. Finfgeld measured the sputtering coefficient of H and D (0.5-8 keV) on Co, Mo, Ta, W, and Au.¹⁷ These measurements were made at room temperature and on undamaged surfaces. Earlier data by Rosenberg and Wehner¹⁸ contained low energy sputtering yields of helium on many elements over the energy range 100 - 600 eV.

This earlier data was taken using a glow discharge as a source of ions and is suspect from two points:

1. Uncertainties resulting from the undetermined angle of incidence and possibility of impurity ions.
2. Return of sputtered material to the surface caused by scattering from the gas near the surface.¹⁹

Theoretical studies of sputtering yields have been made by Weissman and Sigmund²⁰ and others. Hydrogen isotope sputtering of stainless steel has been studied by Farebrother, et al,²¹ Kenknight and Wehner,²² Borovik, et al,²³ Guseva,²⁴ and Behrisch, et al.²⁵ Oechsner²⁶ measured the sputtering yield for 300 - 1000 keV He on Ni. None of these experiments were done on highly-damaged surfaces, with the preimplantation of gas particles, or as a function of angle of incidence. These factors will probably affect the sputtering coefficient substantially.^{27,28}

Colligon, et al,²⁹ have observed a marked dependence of sputtering yield on the bombarding ion dose. Sputtering yield was found to change with ion dose, as well as with the bombarding ion energy. Experimental evidence suggested that this change could be associated with the formation of dislocation loops. These results would tend to indicate that previous experimental work might not be applicable to sputtering yields in a fusion reactor where heavily-damaged surfaces will be the rule rather than the exception. Sputtering yield is also a function of the target surface morphology.³⁰

C. Surface Chemical Effects

There are a variety of surface chemical effects that can influence the performance of a first wall coating. Hydrogen isotopes implanted in the surface region of the coating and interaction with the hydrogenic gas could lead to chemical erosion through processes such as lowering of the coating vapor pressure and surface binding energy due to surface compound formation. Chemical sputtering has been studied experimentally for only a few materials under very limited conditions.³¹ Probably the most important materials with regard to surface chemical effects are the carbides and graphite.³² Serious problems involving the formation of hydrocarbons have been reported in certain temperature ranges upon reaction with hydrogen.³³ Since carbon and carbides have many attractive features, their chemical reactivity in a Tokamak environment must be determined if they are to

remain viable candidates. Even very stable oxides have a negative free energy for reduction by atomic hydrogen and need to be evaluated. Experiments on chemical reactivity need to include surface structural changes influenced by radiation-induced damage.

D. Bulk Radiation Damage

Many processes can lead to degradation of a coating other than physical sputtering and chemical interactions. Some of these are blistering, swelling, embrittlement, transmutation, and gas diffusion. Only recently has an understanding of the processes begun to develop. Candidate metal coatings may be divided into two groups with regard to their interaction with hydrogen. The first group has a very high solubility for hydrogen, while the second has extremely low solubility. For the first case, a solute gas of single atoms in the host lattice is expected to form and extensive blistering by hydrogen would not be expected to occur; while for the second case, blistering would be expected unless improved morphologies are developed.

In contrast, helium is not soluble in metals to any significant extent and is expected to diffuse rapidly in a metal lattice as long as it is not trapped at a defect. Unfortunately, many defects are expected in the first wall due to the radiations imposed on it, so significant trapping of helium is expected. The amount trapped is expected to depend on the types and amount of traps created and their mobility. Helium and hydrogen buildup, especially in the interface region between a coating and its substrate, could provide a mechanism for failure of the coating through debonding and/or flaking.

Blistering has been reviewed recently in the literature.³⁴ It has been suggested that in some materials an equilibrium surface structure is reached after a fairly large implant fluence and that little or no blistering occurs after this equilibrium is attained, although other studies have shown that this is not universally true. Much work remains to be done before blistering is fully understood.

Swelling is expected due to the creation of voids and the collection of gases in the coating. This effect combined with blistering could provide an interconnected pathway from deep within the coating for release of the trapped gases, in other words, a very porous coating. It is thought that the more porous a coating, the less the problems due to blistering, swelling, and possibly thermal expansion mismatching.

Transmutation rates have been calculated for many candidate materials. The main problem appears to be the creation of He in the lattice and its subsequent buildup as discussed earlier.

Embrittlement could present a problem and must be considered as a failure mechanism for the coating as well as thermal stress cracking.

E. Temperature Effects

The thermal cycling in an EPR has been studied by Argonne National Laboratory.¹ They found significant stresses are induced during the thermal cycling of the structural components. Thermal expansion matching of the coating and its substrate must be considered in any selection process. The final selection must provide a coating which will withstand $\sim 10^6$ cycles without falking or debonding and subsequent vaporization. Debonding could be very detrimental in a fusion reactor because without good thermal conduction between the substrate and coating, the coating could reach vaporization temperatures.

Although adhesion is very important, it is very difficult to obtain a quantitative measure of this parameter, especially when adhesion is good. Excellent discussions of the stress and adhesion of films to substrates have been made by Mattox³⁵ and Westwood.³⁶ If the stress in the coating exceeds the adhesion strength, the coating will part from the substrate. A review of coating adhesion measurements has recently been done by Dini and Johnson.^{37,38}

The mechanical loading of a first wall is complex because of the time variable heat flux. The varying stress produced when the metal expands and contracts produces damage in the material which can accumulate and induce fracture. During periods at elevated temperature, partial recovery can occur, along with creep. The interaction of creep and fatigue is currently a subject of intense research. When the effects of radiation and surface damage are added, the behavior can only be determined empirically and that with difficulty.

III. CANDIDATE MATERIALS

At this time it is not possible to be very specific in presenting a list of candidate coating materials because there are large gaps in the data needed to reliably predict the response of coatings in an EPR environment. Of course, the main criteria by which a coating is judged is its ability to minimize contamination of the plasma; however, just what combination of properties will achieve this objective is not obvious. Clearly, one very important parameter is atomic number. Hopkins⁷ has examined the relationship between Z and concentration to achieve ignition for the particular case of the General Atomic EPR design. His results, shown in Fig. 1a, while calculated for a particular case, are expected to have general validity in the sense that the allowable concentration of an impurity is a rapidly decreasing function of increasing Z of the approximate form shown in Fig. 1a. Calculations such as these present a strong argument for using as low a Z coating as possible; therefore, low Z materials such as Be, B, C, BeO, Be₂C, B₄C, BN, SiC, and Al₂O₃ must be considered as coating candidates.

Given that low Z is important, the problem is to determine other criteria pertinent to the performance of a coating. One could select a material to interface with the plasma on the basis of information such as that contained in Fig. 1a, coupled with a knowledge of the mechanisms whereby the material enters the plasma. For example, if sputtering were the most important contamination process compared to other possibilities such as vaporization, chemical reactivity, desorption, or blistering, then with the aid of Fig. 1a, it would be possible to define a maximum allowable sputtering rate as a function of Z. Then, if sputtering rates for all materials of interest were known, a selection of the "best" material subject to the sputtering rate criteria could be made.

Unfortunately, the most important plasma contamination mechanism for one material may not be the same as for another. In addition, the details of how an impurity atom behaves once in the plasma; that is, its residence time, probability of leaving the plasma, etc., are not well known. The point to be made is that the selection of the best coating material is not simple and cannot be made by the materials engineer without experimental research and interaction with both the plasma and surface physics community, the specifics of which follow in this report.

Independent of what material or materials seem to be attractive for a coating, they must have the potential for being applicable to large areas at costs that are consistent with the expected benefit of the coating. Because of the unique

requirements of the first wall environment, it may be necessary to not only perform scale-up development of coating methods, but incorporate design features in the Tokamak that facilitate in situ coating repair or rework. The remainder of this section will deal with what appears to be the major candidate application techniques. One criteria that will influence the selection of an application process is the capability to remotely recoat or repair a coating in situ. This will be important since the radiation environment inside a Tokamak once it has become operational will probably prohibit personnel from entering the torus.

A point to keep in mind when considering application techniques is that there is a good possibility that a coating will be made up of several layers. The outer layer will be chosen to minimize plasma contamination and underlying layers will act to maximize adhesion and minimize thermal stress and fatigue damage.

Application Techniques - There are a number of coating techniques that could possibly be used to deposit a coating on a first wall. A list of these is as follows:

1. Plasma spray
2. Chemical vapor deposition (CVD)
3. Physical vapor deposition (PVD)
4. Electrodeposition
5. Thermal-Mechanical bonding, e.g., diffusion bonding, brazing
6. Alloy surface treatment, e.g., oxidizing
7. Cementation

Rather than describe these processes in detail, only those characteristics pertinent to the EPR application will be discussed.

1. Plasma Spray - Plasma spraying appears to be one of the most promising application techniques due to the variety of sprayable materials possible, the rapid deposition rates attainable, and the possibility of in-situ coating. In particular, Be, BeO, beryllides, and BN have been plasma-sprayed. Coatings typically possess 5 - 15% porosity and are made up of mechanically-adhered lamellar particles. Such a microstructure has the disadvantage of relatively low mechanical strength, but the attributes of a tailorable porosity and particle size. Since the resistance of a material to blistering is a function

of the ability of He to diffuse out, it should be possible to vary particle size and porosity to minimize blistering.

2. CVD - Like plasma spraying, CVD appears to be a very promising, high deposition rate, low Z coating process. It also has great versatility having been used to apply Be, BeO, BN, B, C, and a host of other oxides and carbides. Perhaps the most significant features of CVD for a Tokamak application are the ability to produce a preferentially-oriented crystal structure and the possibility of in-situ application. A possible disadvantage of CVD is the use of chemically-reactive gas atmospheres that could pose corrosion or compatibility problems for in-situ application and a generally high deposition temperature.

3. PVD - The coating techniques of ion plating, sputter deposition, and evaporation are grouped together because they all require similar deposition conditions. Although there are significant differences between these processes, one or more of them can be used to coat almost any material of interest. High-rate sputter deposition is one of the most versatile application techniques considered. The many variables available to the sputter deposition process make it possible to produce coatings or series of coatings (individual or graded composition layers) with wide ranges of structural, chemical, and physical properties from practically any desired material. Inherent to the sputtering process is the ability to bombard the surface to be coated with ions, thereby providing a clean predictable interface for good bonding. The sputter deposition process for critical materials requires a clean vacuum environment similar to the EPR chamber itself and with proper design, sputter deposits could be applied in situ with remote controls.

4. Electrodeposition - Electrodeposition is taken to include electroplating from both aqueous solutions and fused salt baths and also electrophoresis. This probably is not a very attractive process for a first wall coating. The range of candidate low Z materials that could be applied with this technique is

small and the requirement of having to immerse large panels in an appropriate electrolyte is restrictive. In addition, there appears to be no realistic way of making electrodeposition an in-situ process.

5. Thermal-Mechanical Bonding - The term thermal-mechanical bonding is meant to include processes such as roll bonding or brazing. Such processes could be used to fabricate large Tokamak parts; for example, by bonding thin sheets of a low Z material like Be to a stainless steel substrate. A disadvantage of these techniques is the inability to repair a damaged area once it has operated and become radioactive. Perhaps a combination of processes such as brazing a low Z coating in place initially, followed by plasma spraying to "patch up" defects, could be made to work.

6. Alloy Surface Treatment - Alloy surface treatment is meant to describe those processes that chemically condition an alloy to produce a low Z coating on its surface. For example, it has been found that a Be-Cu alloy, when exposed to steam, forms a BeO surface layer.^{39,40} It may be possible to devise a structural material such as a Be, containing stainless steel that could be treated to form a low Z coating on its surface. In principle, such a coating may be renewable by periodic conditioning treatments. Although such a process is only in the conceptual stage, it does merit consideration.

7. Cementation - This is a generic term which applies to a variety of processes. Pack cementation, for example, involves immersing a part in a bed of solid coating particles. Hot dipping, as the name implies, consists essentially of dipping the part in a bath of the coating material and then firing. Either of these might be used to put an initial coating on a substrate. Another form of cementation, painting or glazing, seems to have some real possibilities for first wall coatings. The coating could probably be applied either directly by plasma spraying or, if mixed with an organic, it could simply be sprayed with a paint gun. In either case, heat would be needed to fuse and anneal the coating.

IV. PHILOSOPHY OF COATING DEVELOPMENT PLAN

The previous sections have reviewed the reasons for a first wall coating, the environment of the first wall, the requirements on a first wall surface, and have discussed the factors involved in material and process selection. We will now describe the specific approaches one can take in coating development.

The reader must keep the following points in mind as he proceeds:

1. It is not certain that a satisfactory first wall coating exists.
2. Even if a perfect first wall coating were available, it could not be recognized since no valid testing environment exists.
3. However, we may never know if the Tokamak approach is feasible unless the best available first wall material is provided for the designer to interface with the plasma.

Three first wall coating development methods will be discussed briefly, and the approach recommended by the authors described in detail.

1. Quick and Dirty Method - The development would proceed by screening a large number of candidate first wall coatings by:
 - a. Measuring their chemical interaction with a hydrogenic plasma,
 - b. Comparing the relative sputtering yield of the candidates in a Tokamak simulator; namely, using a large low energy ion source in a qualitative test at one temperature.
 - c. Developing a coating process for the best two or three candidates determined in these tests, and finally,
 - d. Selecting the most adherent, fatigue-resistant coating for full scale development.

This approach is fast with minimum cost, but may overlook the best first wall coating system, and provides little quantitative data to influence research programs. In essence, it is a go no-go gamble.

2. The Iterative Method - This development program would proceed by first determining the material and morphology most suited to give the desired low sputtering yields by testing a limited number of material classes. The chemical interaction of single representatives of material classes would also be measured. These results would be assumed general and a limited number (~ 12) of materials from the best classes would be prepared as coatings. These coatings would be ranked in terms of their

performance in abbreviated sputtering tests and simulated chemical interaction tests. The best candidates (~ 6) would have coating processes developed that give good adhesion in short time elevated temperature tests. The best of these coatings (3 or 4) would be evaluated in more complete sputtering tests, and the best pair would have their coating process scaled up, evaluated after irradiation, and tested for design flexibility and repairability.

The Iterative Method technique takes longer than the Quick and Dirty Method, costs more, but has less chance of overlooking a good material, and provides output to guide supporting research

3. The Brute Force Method - This method proceeds by exhaustively examining each parameter and interaction to assure that the optimum test and material is developed. The advantages are obvious, as are the drawbacks. The approach would, however, at great expense, produce the "very best" coating, though probably too late to influence EPR.

The approach taken in this study is the Iterative Method. It has inherent flexibility and a good interaction potential with supporting research. The guiding principle is "learn no more than is necessary to justify the next decision." Strong coupling with the confinement and research programs is essential if the development is to have impact.

This program was developed to produce a material and process specification with appropriate design standards acceptable to industry. The program was formulated by noting what information was required for specifications and standards and working backwards. This technique produces little extraneous information, since it emphasizes what information is required rather than what information is not known.

The elements of a coating system process specification are as follows:

- Specification of input material
- Description of acceptable classes of substrates
- Specification of acceptable substrate surface conditions, for example, finish, cleaning, maximum time and storage condition between cleaning and coating
- Specification of acceptable surface condition and any additional surface preparation for recoating or repair
- Process Description
 - Composition of materials consumed in processing
 - Substrate temperature limits
 - Limits on rate of deposition
 - Acceptable variation in process variables
 - Frequency, number, and validation of in-process controls
 - Required documentation
 - Rework
- Product acceptance test and standards
- Subsequent treatments (if any) to remove damage. For example, periodic anneals in special atmospheres could remove damage before blistering or cracking occurs.

V. RECOMMENDED PLAN

This section is intended to describe the proposed coatings development and evaluation plan. In general, the procedure adopted was to understand and stimulate the anticipated environment of a first wall coating and select those factors of the environment that seem to have the greatest effect on the performance of the coating. These factors were then used to design tests or qualification criteria that provide a basis for initially ranking materials. The function of a coating in this context is to minimize contamination of the plasma. While there might be unanimous agreement that this is a worthwhile objective, there would be disagreement as to specifically what contamination level is acceptable. The problem is quite complex being a function of the machine parameters that determine the plasma dynamics in a Tokamak, in addition to a host of material properties. This program does not attempt to specify what the properties, such as the sputtering coefficient of a coating material or lifetime of a sputtered atom in the plasma, must be. This is an area that is better left to the Tokamak designers who have developed computer programs to predict the plasma performance as functions of time, material properties, and machine parameters. Rather, the objective of this program is to evaluate coatings against a small set of parameters thought at this time to be the most important and to interact frequently with Tokamak designers to produce the best practical coating possible.

It is recognized that there are a large number of material properties and radiation-related phenomena that will affect the performance of a coating; time, money, and manpower constraints prohibit the complete investigation of all variables and suggest concentrating on only the more important. We have made a selection of what we consider to be the most important performance criteria of a coating. These criteria are sputtering behavior, reactivity with deuterium, and adhesion of coatings to the substrate.

In general, the format of the development plan is to select materials that have both acceptably low reactivity with deuterium and have low sputter yields. These materials will then be used to make coatings with the required adhesion characteristics. The development program illustrated in Figs. 3 and 4 is divided into four main areas: fabrication, sputtering tests, deuterium reactivity tests, and adhesion tests. A detailed explanation of these activities will be provided in the later report; therefore, just an overview to explain the interactions between the main activities will be given here.

The program begins with Phase 0 simultaneously in three areas: sputtering, deuterium reactivity, and adhesion testing. In the sputtering program, the first activity is an investigation of the effect of three variables on sputter yields: surface morphology, angular dependence of the incident particles, and chemical state of the material being sputtered, all considered to be potentially important. Concurrently, the initial experiments on bulk materials are begun to examine the effect of chemical state on deuterium reactivity. The initial adhesion activity involves developing the apparatus and the computational tools necessary to test and understand the behavior of coating adhesion under service conditions.

Results of the preliminary sputtering and chemical reactivity tests are analyzed and the surviving material types are used in Phase 1. The first series of coated samples (Lot 1-1) are produced on compatible substrates. The samples are approximately 20 x 50 mm². These specimens are tested for reactivity with deuterium and then their sputter yields are determined. Preliminary adhesion tests are also begun with these parts. The results of testing the Lot 1-1 samples are analyzed, some materials are discarded, and the information is used to make a more selective set of samples, Lot 1-2.

Lot 1-2 is intended to provide detailed sputtering and deuterium plasma reactivity behavior on a small set of samples. In addition, the fabrication of these samples will be the first attempt to make coatings designed to adhere to a substrate in a simulated Tokamak environment; that is, the samples will consist of a 316SS substrate or candidate liner substrate, an outer material layer chosen from the survivors of Lot 1-1 sputtering and chemical tests, and several anticipated intermediate layers. The purpose of the intermediate layers will be to maximize adhesion by attempting to mitigate the effects of thermal expansion differences between the substrate and outer layer.

The results of testing the Lot 1-2 samples should provide detailed performance information on the sputtering and deuterium reactivity of the candidate materials in addition to valuable information on adhesion behavior. At this state of testing, it should be possible to select one or perhaps two materials that will minimize plasma contamination if their adhesion is satisfactory. The remainder of the development program is essentially devoted to insuring that the adhesion behavior is optimized. To this end, an extensive series of adhesion tests on Lot 1-3 employing high temperature cycling are utilized. A number of iterations using the chosen substrate and outer layer, but varying the intermediate layers

are envisioned.

Once a satisfactory coating system is designed, which includes the outer layer and all necessary intermediate layers, a series of larger ($\approx 100\text{mm} \times 300\text{mm}$) samples (Lot 2-1) employing assembly features such as welds and boltheads, will be made. This is Phase 2 of the development plan. These samples will be used to investigate the effects of actual production features on the adhesion properties of the coating. If possible, the effects of radiation provided by a source such as ion implantation or in-pile exposure, will be combined with adhesion to explore synergistic effects. In addition, these samples will be tested, if possible, in a simulated Tokamak environment such as that described in Ref. 41 or in a Tokamak test.

The final phase ($\phi 3$) of the coating development program will involve coating parts of a size representative of actual EPR parts. There are two main reasons for this effort. It will provide a test of the ability to scale up the coating process to the size ultimately required and also provide parts to be inserted in an existing Tokamak for complete testing. Coating technology will be transferred to commercial sources and final design and process standards developed in this phase.

Fig. 3 Process Flow Diagram

- indicates flow of samples

- indicates results obtained on those samples

The program progresses in time from top to bottom.

Each of the fabrication loops might need to be cycled more than once in order to accomplish the objective of a particular task. The process is set up as a set of tasks and are named accordingly.

The nomenclature is:

S = sputtering tests

C = chemical interaction tests

F = fabrication

A = adhesion tests

O = initial samples

1 = 20 x 50 mm samples

2 = 100 x 300 mm samples

3 = full size Tokamak samples

a,b,c,d = subdivisions (identified in Figs. 3 and 4)

For example, Sla = sputtering of samples 20 x 50 mm

Fig. 4 Time and Milestone Chart

Time in months is given at the top of the chart.

Milestones are denoted by . They represent points where a decision to continue onto the next phase, repeat a phase, or discontinue the development must be made.

Nomenclature is the same as in Fig. 3

Fig. 4. Time and Milestone Chart

FABRICATION

The fabrication task has two elements: (1) process development and (2) fabrication of coating sets* for evaluation in other tasks. Prior to actual sample coating, a period of process selection and development, material selection, and coating system design will be completed. Each new generation or lot of material coating sets will be designed to overcome any weakness discovered in the preceding tasks. Many specimens may be required during the process development phase, but they will be evaluated only mechanically and will not be documented as a part of a set.

The specimen fabrication lots required are as follows:

Lot FOa: These specimens will be prepared from either pure bulk materials or coatings and consist of the materials Be, BeO, Be₂C, V Be₁₂. They will be disc-shaped and at least 10. mm in diameter. The surfaces will be smooth and damage free. These will be tested in Task SOa.

Lot FOb: These specimens will be prepared from deposits having the required morphology and will be at least 10. mm in diameter. They will be tested in Task SOb.

Lot FOc: These specimens will be prepared from the same pure bulk materials as FOa, plus CVD pyrolytic graphite and SiC. Surfaces will be mechanically polished. They will be used in Tasks COa,b,c. The samples prepared for COa will be at least 10. mm in diameter, while those prepared for COb,c will have at least a 1000 mm² area.

Lot F1-1: These specimens will contain the first coatings. Substrates will be selected from 316SS, Ni, or Mo to minimize adhesion problems, since the emphasis is still sputtering now in Task Sla and chemical interaction Tasks Cla,b,c.

Lot F1-2: These specimens will be deposited on actual EPR wall or liner substrates. The materials will be selected from the best performers in Tasks Sla and Cla,b,c. Intermediate

* A coating set contains nine identical coated specimens and one uncoated control.

layers will be used to improve adhesion and short time mechanical testing Task Alb used to evaluate the systems. Chemical interaction will be evaluated in Cld, and sputtering performance will be evaluated in Slb.

Lot Fl-3: These coatings will be designed and fabricated to optimize mechanical performance. No additional sputtering or chemical test will be run unless significant changes are made. Testing will be conducted in Alc where simulation of the Tokamak thermal-mechanical and chemical environments will exist. These tests will be repeated on pre-irradiated samples to size the effect of bulk radiation damage.

Interspersed with the sample preparation phases above will be phases of process development. During these phases, process variability and the interaction of process variables on mechanical performance will be studied. The emphasis will be to acquire a data base so that preliminary process specification can be prepared for Phase 2 in which industrial participation will commence.

Phase 2 of this program addresses problems of scale-up, accommodation of engineering details, and coating repair. The specimens in Phase 2 are larger, i.e., 100 x 300 mm, and include welds, fasteners, various radii, corners, and different surfaces including recoating overdamaged coatings. A proposed sample shape is shown in Fig. 5.

A set of Phase 2 specimens will include two coated and one uncoated large plates, plus six Phase 1 size process control specimens. Coatings produced in ERDA laboratories and industrial sources will be tested in a simulated Tokamak material test facility. Final design standards and process specification will be developed jointly by ERDA coating developers - DMFE Tokamak designers and industrial coating suppliers.

Phase 3 will involve producing full-sized elements for use in reactors and in final tuning of the coating materials and process specifications. The number of samples needed for sputtering and chemical interaction tests is given in Table 3.

Coating Sample Sets

Phase 1

The output of a coating process run will be documented as a unique set if it is to be characterized further in this program. A minimum set size is necessary to assure adequate analysis and to provide archive samples for more detailed analysis, if needed.

The minimum set size for preliminary or Phase 1 coupons is ten coupons, each about 20 x 50 mm. Nine will be coated and one left uncoated as a control.

Each set should be produced under similar conditions at the same time using the same substrates (i.e., from the same sheet). Each specimen will be serial-numbered and process records kept. Miscellaneous samples coated for process control or development should not pollute the program.

Samples from each set will be distributed to various laboratories for performance testing, so careful documentation of each set is necessary.

Phase 2

A coating set in Phase 2 will consist of three large plates, i.e., 100 x 300 mm. Two will be coated and one uncoated as a control. Along with each large plate, six Phase 1 size coupons will be coated for characterization and lot acceptance. Each plate will have the following features as deemed appropriate by Tokamak designers:

1. Three surfaces
 - (a) as received
 - (b) as welded
 - (c) welded and machined
2. Edges with different radii, i.e., .1, 1, 3, 10 mm
3. Step with angles of 90°, 60°, 45°, and 30°.
4. Fasteners, i.e., boltheads or rivets.

Specimen Distribution Plan for a Phase I Set

NOMENCLATURE: Laboratory Process Materials Date Run Sample

Example: SIA CVD TiB₂-316 1/1/78 2 B

SERIAL NO.: Six digits stamped on coupons

Each of the ten specimens will be assigned a letter and used in the following ways: (Extra specimens will continue using letters.)

- A Set acceptance - destructive test (pieces to archive)
- B Uncoated control - archive
- C Unassigned spare - archive
- D Sputtering
- E,F Chemical interaction
- G,H,I,J Mechanics Test (broken pieces can be used for analysis or to supplement the D (sputtering or E (chemical interaction) samples, or any other evaluation. They will be kept with archive samples.)

INSPECTION: All samples will be weighed before and after coating.
Thickness and surface roughness will be measured.
Physical dimensions before and after coating will be recorded.

SET ACCEPTANCE TEST: Coating bulk chemical analysis by emission spectroscopy, metallography, hardness, coating bend strength, and acoustic emission rate will be recorded for each set.

Fig. 5. TYPICAL PHASE 2 SPECIMEN

SECTION DETAILS APPROX. FULL SIZE

TABLE 3. NUMBER OF SAMPLES NEEDED FOR SPUTTERING AND CHEMICAL TESTS ⁺

Task	Experimental Conditions					Total Runs	Total Samples
	Material	Energy	Angle	Temperature	Morphology		
COa	6	2	1	2	1	2	48
b	3	1	1	2	1	12	12
c	3	1	1	2	1	12	12
SOa	4	2	1	1	1	16	16
b	2	2	3	1	≥ 4	96	96
Cl a	12	1	1	2	1	2	48
b *	6	1	1	2	1	24	24
c *	6	1	1	2	1	24	24
Sl a	≤ 6	1	1	1	1	12	12
Cl d *	~ 4	1	1	4	1	32	32
Sl b	~ 2	2	3	2	1	48	48
SCA2a	2	Simulated Tokamak Test			2	8	8
SCA2b	2	Simulated Tokamak, including repair			2	8	8

⁺ This chart was constructed for a first wall coating. For a liner coating the number of materials would be somewhat smaller, but the number of temperatures at which tests are run could be larger

* Larger samples, approximately 1000 mm²

PHYSICAL SPUTTERING

An adequate data base for sputter yields as a function of various parameters does not exist at the present time. It is suggested that experimental and theoretical programs, independent of the coating project, be pursued so that a thorough understanding of physical sputtering may be developed. The specifics of this research area are being addressed by the Subtask Group on Physical Sputtering, Reflection and Neutron Effects. To support this coating program, the basic DMFE sputtering program should address sputter yield measurements for the following conditions:

- (1) incident species Z: H, D, T, and He
- (2) effect of $Z > 2$ (impurities, wall and limiter materials)
- (3) incident particle energies (E); 50 eV-15 keV and at selected energies typical of neutral beam injectors.
- (4) effect of angular dependence of incident ions
- (5) effect of ion implanted species
- (6) temperature 300 K - 900 K and 1600 K - 2000 K
- (7) inclusion of synergistic effects
- (8) well-characterized clean surfaces and ultrahigh vacuum.

Measurements using T should be minimized because the use of T beams presents many problems for the experimenter. Some limited data should be taken, though, to establish a scaling for T based on the results obtained for D. Hopefully, in the near future, theoreticians can accommodate the lower energies and light ions into a theory which will give meaningful results. The use of ion beams instead of atomic beams should make no difference in the sputter yield because at the energies expected on the first wall of a Tokamak, the atom or ion velocity is low compared with the electron orbital velocities and as the atom or ion approaches the surface, charge equilibrium is established between the atom and the surface; as a matter of fact, an electron may be exchanged many times during the entrance into the surface.

Sputtering problems to be addressed in the initial phase of the coating project will be limited, as much as possible, to measurements which will qualitatively rank various parameters with regard to their effect on sputtering yields for a limited set of materials. The later phases will address an expanded set of materials, synergistic effects, and other dependencies.

Initial screening for sputtering will be accomplished using D at 300 eV and 2 keV. If a continuous energy source, which approximates the energy spectrum of neutrals hitting the first wall, can be developed before the initial phase, then it should be used instead of monoenergetic beams. Data will be taken at one temperature, 800 K, for a SS first wall (1900 K for a liner coating) with a D₂ pressure of 10⁻⁴ Torr at the sample surface. An appropriate magnetic field could be used if it is strongly felt that it is needed for simulation.

Task SOa

The first parameter to be studied is the effect of the chemical state of the material, Task SOa. To obtain an initial evaluation of this factor, the sputtering yield will be measured for Be, BeO, Be₂C, and V Be₁₂. In an attempt to eliminate morphological effects, the samples will be bulk materials prepared so that the surface is flat, reasonably strain free, and reproducible. One method of accomplishing this is to heat the sample near its melting point and then melt the surface. These samples will be provided by Task FOa. The material should be characterized, at a minimum, with respect to texture, surface roughness, composition, and grain size, both before and after sputtering until equilibrium surfaces are attained. The operating parameters are given in Table 4. An angle of incidence of 50° was chosen because preliminary theoretical data has shown that a peak in the charge exchange flux to the wall exists near 50°. (See Fig. 6)⁴² In addition, the sputter yield curve for relatively high mass particles has a maximum somewhat larger than 50°. ^{43,44,45} Of the various methods of measuring sputtering yields shown in Fig. 7, Methods 1, 2, 3, and 5 are applicable; 2, 3, and 5 are preferred and may be used simultaneously.

Task SOb

The second parameter to be varied is morphology and texture, Task SOb. Two materials, Be and Mo, were chosen for this portion of the study because they lend themselves to easy manipulation of the morphology and texture. Beryllium was chosen as a representative low Z material and molybdenum as an intermediate Z material. Comparisons are to be made with a set of samples of a particular metal for various morphologies and textures. Initial sputtering yields should be measured for films with uniform thickness, near maximum density, and a smooth surface as a control so that a baseline can be established. The operating procedures are given in Table 4. The angle of incidence (measured from the surface normal) will be varied to study the effects of topology (roughness) and texture. Measurements

Fig. 6

Three-dimensional calculation of the angular distribution of charge-exchange particles that escape from the ST Tokamak and strike the vacuum vessel at the mid-plane. Theta is the angle of the charge-exchange particles with respect to the vacuum vessel normal. The various curves are for charge-exchange particles of different energies ($E = 20, 50, 100, 200, 500$, and 1000 eV). The angular distributions for larger devices, including reactors, will be similar. MCF simulation on experiments should allow for azimuthally symmetric target irradiation at 500.35

Fig. 6. Three-dimensional calculation of the angular distribution of charge-exchange particles that escape from the ST Tokamak and strike the vacuum vessel at the mid-plane.

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SPUTTER YIELD MEASUREMENT TECHNIQUES (Fig. 7)

1. WEIGHT LOSS (GAIN)

2. PROFILOMETER

3. CATCHER PLATE (RUTHERFORD BACKSCATTERING, N ACTIVATION)

4. THIN FILM (RUTHERFORD BACKSCATTERING, AUGER, PROFILOMETER, ETC)

5. IMPLANTATION MARKER (RUTHERFORD BACKSCATTERING, GAS RELEASE)

will be performed at 20°, 50°, and 70°. In addition to the control samples, the following types of films should be studied at a minimum: (1) highly textured columnar structure, (2) uniform thickness but with a high degree of porosity, and (3) near maximum density but with a rough surface. Methods 1, 3, and 4 could be used to measure the sputter yields, but Methods 3 and 4 are preferred and could be used simultaneously.

The material should be characterized at a minimum with respect to texture, surface roughness, composition, and grain size, both before and after sputtering, until equilibrium surfaces are attained.

From the results of Task SOa and Task SOb, it is hoped that the effects of chemical state, morphology, and texture can be ranked as to their importance in modifying sputtering yields. The information gleaned from the first iteration of this task could be used to provide a second set of samples from FOa and FOb. The number of loops made depends upon the results obtained. The results obtained on specific coatings and materials will be passed to Task F1 for fabrication of coated samples. In addition, the results obtained on the importance of the various parameters will be passed to S1 for a refinement on the testing procedures.

A major requirement for this task is the development of an experimental apparatus which will provide an intense beam of deuterons, 1-10 mA/cm², (10-100 μA/mm²) and very low impurity (e.g., C and O) impingement rate on the target. It is felt that an intense beam is required since sputter yields are expected to be low (10⁻³ - 10⁻⁴), many samples must be investigated, and yield measurements for one sample should be completed in a day or less.

Task Sla

Task Sla will use the results obtained in Task SO to evaluate an expanded set of materials so that candidate materials and coating procedures can be quantitatively ranked with regard to their physical sputtering characteristics.

The expanded set of materials and coating techniques will be based on information gathered in Tasks SO and CO and incorporated into the coating fabrication Task F1. It is hoped that a selection of the general types of materials, i.e., metals, oxides, carbides, etc., can be made as an output of SO and CO and those general types with the more promising characteristics can be expanded to include a larger set than was included for SO and CO. The samples used for this Task will be tested in C1 and only the more promising candidates tested. In addition,

the important sputtering parameters as determined in S0 will be emphasized in this Task.

The results will be outputted to F1 so that a further refinement in the fabrication of coated samples is effected.

Task S1b

In Task S1b testing will be performed with an extended set of parameters on only the better candidates from C1d.

The sample set will be obtained from F1 after the inputs of S1a and C1 are integrated into F1. The objective is to rank order the candidates with regard to their sputtering yields.

Table 4. OUTLINE OF SPUTTERING TASK SO

Objective: Determine the effectiveness of coating parameters on changing sputtering yields (i.e., chemical state, morphology, and texture).

(a) Chemical State

E: 300 eV, 2 keV

T: 800 K

P_{H_2} : 10^{-4} Torr

Angle: 50° (0° is beam $\perp$ surface)

Material: Be/BeO/Be₂C/VBe₁₂

Surface treatment - Samples heated to near melting point or hot isostatically pressed, surface melted to produce flat, reasonably strain-free and reproducible surfaces.

Techniques: 1, 2, 3, and 5*

(b) Morphology Texture

E: 300 eV, 2 keV

T: 800 K

P_{H_2} : 10^{-4} Torr

Angle: 20° , 50° , and 70°

Material: Be, Mo

Techniques: 1, 3, and 4*

* Note: Key on Fig. 7.

SURFACE CHEMICAL REACTIONS

The interaction of surface impurities with hydrogen* may in fact represent a greater contribution to the plasma impurity problem than does interaction with the underlying coating. The active forms of hydrogen (atomic, ionic, and metastable) emitted by the plasma are the most important reactants.

COa

Since such a poor data base exists, the major objective of COa will be to establish the dominant parameters involved and the magnitude of the effects. The most direct way the plasma can be affected is by gaseous contaminants produced by chemical reactions. The determination of the potential magnitude of gaseous contaminant production is relatively straightforward. Thermal hydrogen atoms can be produced at the surface of a hot tungsten filament, or high frequency discharges can be used to produce a less well-defined reactive hydrogen environment. Two of the critical parameters which should be studied are the temperature of the substrate and the energy of the incident particle. The temperatures to be investigated will be 600 K and 800 K (1300 and 1800 K for coatings on liners) and the energy range 1 - 50 eV. For this task, studies to identify those types of materials for which potential problems exist will be surveyed by studying samples obtained from Task FOb. The materials to be studied will be Be, BeO, Be₂C, VBe_{1.2}, Mo, and CVD pyrolytic graphite with mechanically-polished surfaces. The extent of impurity reactions can be followed by weight loss measurements. This, however, yields no information on the nature of the reaction products or the mechanism of the reaction. Morphology change and D retention will be investigated after each exposure using SEM and RBS or thermal desorption, respectively. The samples will be obtained from Task FOb and the experimental apparatus should have the capability of comparing several samples simultaneously. The samples will be of bulk materials and mechanically polished.

COb

As a first step in understanding the chemistry involved, the products must either be determined in situ, e.g., mass spectrometry, or collected for subsequent identification provided the reaction rate is large enough. The more promising materials, as determined in COa, will be investigated singly

*The word hydrogen encompasses the three isotopes, H, D, and T.

in the same apparatus to identify and quantify the major gaseous contaminants. The temperatures investigated will be 600 and 800 K (1300 and 1800 K) and energy range 1 - 50 eV. The results will be outputted to COc.

COc

Surface chemical processes could be strongly dependent on synergistic effects. Surface damage can strongly affect the reactivity. Enhancement of reactivity as large as 10^7 has been observed on damaged surfaces so that materials inert in terms of the COa and COb studies, may prove to be unacceptable under damaged conditions. The purpose of this task will be to repeat the experiments done in COa and COb on damaged surfaces or in a simulated Tokamak test where the damage can be created at the same time as the surface reactivity is monitored.

Possible Experimental Techniques for COa,b,c

It is suggested that an RF-sputtering system with hydrogen gas could be used. The gas pressure and flow rate could be held constant and the sheath potential between the sample and discharge varied, thus varying the energy of the particles hitting the substrates. The energy range is 1 - 3000 eV and the pressure range 10^{-4} to 10^{-1} Torr. The low energy range, < 50 eV, can be obtained in a triode system. The temperature of the samples can easily be varied by varying the cathode plate temperature to which the samples are attached. Various analytical techniques will be used to study the (1) emitted products, (2) retained hydrogen, (3) surface topography change, and (4) weight change.

Listed below are some of those techniques:

- (1) Mass spectrometry and laser resonance fluorescence
- (2) Nuclear reaction microanalysis and outgassing
- (3) Optical micrography and SEM
- (4) Microbalance weighing

It may be possible to simulate surface damage by doping the D_2 in the RF triode sputter system with Ar. The results will be outputted to Fl.

Cl a,b,c

These tasks are repeats of Coa,b,c using the coated samples of Lot 1 produced in Fl. The results are outputted to Fl for further refinement of the next set of sample fabrication. The objective is to try to understand what the dominant effects are, and rank the materials as to their resistance to chemical reactivity with hydrogen.

Cld

This task will be performed only if chemical effects appear to be large and strongly temperature-dependent. This study would be performed on Lot 2 samples with a thorough study of the temperature dependency of the chemical interactions. The techniques used in COa,b,c would be refined for use in this task.

ADHESION

There are three goals of an adhesion program established to provide a usable coating for an EPR first wall:

1. To study the bonding mechanisms and the debonding characteristics of the candidate coatings so that the coating processes and materials can be optimized prior to fabrication of full-sized reactor components.
2. To aid in the design of reactor parts by observing the effects of substrate geometry on the ability of each candidate coating to adhere in simulated reactor environments.
3. To provide information necessary for vendor qualification for the supply of reactor components and also to provide the techniques to evaluate these coatings and their reactor performance.

There are a number of coating processes and materials which have been developed and which may be applicable to this program. The primary difficulty is in evaluating how a candidate coating will perform in an EPR environment. The coating and substrate will be exposed to both thermal and mechanical cycling, reactive hydrogen, and will accumulate bulk radiation damage. Adherence and cracking resistance will depend upon complex interactions of physical and mechanical properties, operating temperatures, cycling rates, and radiation damage. Traditional test techniques to evaluate coating adhesion at room temperature probably will not be indicative of the ability of a coating to withstand the first wall EPR environment. Some complex and expensive tests which more nearly duplicate the EPR conditions will be necessary to evaluate promising candidate coatings. It is hoped, however, that fundamental understanding of the behavior of coatings might be used to qualify simpler and cheaper room temperature test techniques which could be used by a commercial vendor for quality control.

The focus of any proposed R&D program must be to supply EPR designers with a usable coating process and material which can be provided by currently-existing vendors. Recoating and repair capability is also a major performance criterion for any coating material and process development. There must be a balance, therefore, in the proposed work between gathering the necessary engineering data for candidate coatings and conducting sufficient fundamental

experiments to insure that all of the coating process and material variables have been optimized to produce the most effective coating. Detailed specifications for coating processes and quality assurance techniques developed in the program will be presented in suitable form to EPR designers and commercial vendors.

Description of a Balanced Coating Mechanical Performance Development Program

The development of a first wall coating involves several phases of experimentation in which each phase represents sequentially-increased sophistication and cost. As discussed earlier, the most important qualities of a coating are:

1. Its ability to maintain mechanical integrity and not debond from the substrate and flake off into the plasma.
2. A low plasma contamination rate.

The following discussion describes a program to develop coatings with the necessary mechanical integrity. Such a program would contain the following tasks

- AO - The objective of this task is to develop the testing apparatus that will be used in adhesion testing and also the analysis and computational techniques that will be used to evaluate coatings.

Analytical stress codes will be developed as an element of all adhesion tasks to allow generalization of data. It will also be necessary to develop and qualify NDT techniques both for coating acceptance and for periodic inspection. In addition to standard destructive techniques, new methods particularly suited to nondestructive testing coatings should be evaluated such as:

- (a) Correlation of adhesion to acoustic resonance of the coating as measured by photo-acoustic spectroscopy.
- (b) Correlation of adhesion to heat transfer through coating as measured by IR photometry.
- (c) Correlation of adhesion to surface deflection induced by local heating as measured by laser holography.

- Ala - In this task the first adhesion testing of coatings made in this program will be performed. The experiments will be done at room temperature and will include four-point bend tests both in tension and compression, and also high fatigue rate tests. More detailed tests are not necessary at this point since the best material from a sputtering and D₂ reactivity viewpoint will not yet have been selected.
- Alb - By the time this task is begun, a selection of one or two materials to serve as the outer layer of a coating will have been made. This task is designed to begin the elevated temperature testing that will be required to qualify the layers needed between the substrate and outer layer. Elevated temperature tests will include four-point bending and high strain rate fatigue. Several iterations of coatings may be needed before this task is completed.
- Alc - This task is intended to provide a series of more stringent tests of the coating and to attempt to simulate the background gas and temperature vs. time environment of a Tokamak. This will be accomplished by placing the specimens in a chamber maintained at an elevated temperature and fill with D₂ at 10⁻⁴ Torr. High intensity lamps will be used to vary the specimen temperature in an attempt to simulate the time-temperature relationship expected in a Tokamak.

MECHANICAL PROPERTY EFFECTS OF RADIATION DAMAGE

Significant radiation damage is expected to be experienced by the first wall which will affect the mechanical properties of both the coating and the substrate materials. The integration of radiation damage effects into the Tasks AO-Alc has not been discussed since there is presently no test facility available which adequately duplicates the EPR environment. It is expected that the effects of temperature, stress, and radiation damage will be highly synergistic. Accordingly, radiation damage of some form should be included as an experimental variable as early as possible. Exposures to the most damaging species (ions, neutrons, etc.) for specific coatings could be provided by different facilities. This would necessitate continually evaluating alternate radiation testing facilities during the course of the coating development program.

The initial test development work would allow design of the most suitable test specimens to undergo irradiation and subsequent mechanical testing. From

radiation damage studies, such quantities would be determined as:

1. The most important radiation damage processes for specific coatings as a function of species, temperature, fluence, and dose rate.
2. A knowledge of degradation rates and mechanisms to allow decisions on the feasibility of various recoating processes.

The incorporation of radiation in adhesion testing is addressed by Tasks Ald, A2, and A3.

- Ald - The function of this task is to investigate the effects of radiation. The specifics of how best to accomplish this will depend on the availability of radiation sources in the future. An ideal simulation source would permit the irradiating of specimens during the low cycle fatigue tests discussed in Alc.
- A2 - This task will involve the adhesion testing of large ($\approx 100 \times 300 \text{ mm}^2$) specimens that incorporate the features discussed in Task F2, i.e., welds, steps, etc. The adhesion test will be a high temperature, low cycle fatigue in a D_2 atmosphere. Radiation will be used directly or simulated, depending upon the availability of large area irradiation sources.
- A3 - The third phase of testing EPR first wall coating materials concerns the testing of full scale reactor components in the reactor environment. A large scale test facility will be necessary to evaluate the coating processes to be used for full-sized components and to study the thermal fatigue behavior of large structures. Remote application and touch-up techniques will be developed that are suitable for full-sized components. Another important part of Phase 3 study will be the correlation of the simple measuring techniques developed during Phase 1 with the sophisticated techniques developed in Phase 2 to establish relatively simple quality control procedures to be used by the coating vendors. The exact nature of the work done in Phase 3 will be strongly dependent on the nature of the specific coatings chosen for use in the EPR.

Conclusion

The result of the coating development program should be the selection of an adequate coating material and recoating process which can be manufactured by currently-available vendors. At the completion of the developmental program, the coating process and the quality assurance techniques will be turned over in usable form to the commercial vendors.

ANALYSIS

The philosophy is to analyze samples only minimally, but to retain archive samples so that further analysis may be accomplished, as required. For each set of coated samples, one will be used for metallographic analysis and for examination of its purity by emission spectroscopy. The remaining samples will be analyzed for

1. Dimensional change during coating
2. Weight change during coating
3. Surface morphology

In all cases the option is reserved to do extensive analysis on all samples and substrates at any stage of their processing and testing, if it is dictated by the material performance.

Listed below are the analysis areas of interest and some of the techniques which may be used:

1. Surface composition
 - (a) Auger
 - (b) SIMS (Secondary Ion Mass Spectrometry)
2. Near-surface composition and gradients
 - (a) Nuclear microanalysis
 - (1) Rutherford backscattering
 - (2) Nuclear reactions
 - (b) Sputter Auger
 - (c) SIMS
 - (d) Ion beam microprobe
3. Bulk composition
 - (a) Emission spectroscopy
 - (b) Chemical analysis
4. Surface topography
 - (a) Optical microscopy
 - (b) SEM
 - (c) Surface profilometer

5. Phases
 - (a) X-ray diffraction
 - (b) Electron diffraction
 - (1) STEM
 - (2) ESCA
6. Inclusions
 - (a) e-beam probe
 - (b) ion beam microprobe
 - (c) metallography, Quantimet +
7. Grain size and shape
 - (a) metallography
8. Texture
 - (a) x-ray pole figure
9. Defects
 - (a) TEM
 - (b) positron annihilation
 - (c) resistivity
10. Interstitial distribution
 - (a) x-ray diffraction
 - (b) nuclear microanalysis
11. Physical state
 - (a) density
 - (b) porosity (open/closed)
 - (c) stress state

+ registered trademark

VI. IMPACT

The coating development program will impact other aspects of the DMFE program in the following areas:

1. Rank the processes responsible for material loss and impurity buildup, in order that other research can be focused on the dominant processes.
2. Screen materials to select most promising. This information will help focus supporting research.
3. Develop standards for surface cleaning or passivation
4. Develop valid simple, accelerated screening tests for material intended for use in Confinement experiments, thus reducing the risk of poor material performance clouding results in physics experiments.
5. Determination of equilibrium surface morphology.
6. Serve as a source of materials and coatings that have been well characterized and are metallurgically similar for study and evaluation in DMFE research program.
7. Provide interim coatings for Confinement experiment components to minimize plasma contamination.
8. Provide a framework for failure analysis for Confinement Projects.

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