DOE/OR/22242--T2

ADVANCED THERMAL BARRIER COATING SYSTEM DEVELOPMENT

CONTRACT # DE-AC05-95OR22242

TECHNICAL PROGRESS REPORT

to the

U.S. DEPARTMENT OF ENERGY

Oak Ridge Operations Office

Oak Ridge, Tennessee

August 7, 1996

Submitted By

WESTINGHOUSE ELECTRIC CORPORATION

Power Generation Business Unit

4400 Alafaya Trail

Orlando, Florida 32826-2399

Advanced Thermal Barrier Coating System Development

Program Objectives

The objectives of the program are to provide an improved TBC system with increased temperature capability and improved reliability relative to current state of the art TBC systems. The development of such a coating system is essential to the ATS engine meeting its objectives.

The base program consists of three phases:
- Phase I: Program Planning - Complete
- Phase II: Development
- Phase III: Selected Specimen - Bench Test

Work is currently being performed in Phase II of the program. In phase II, process improvements will be married with new bond coat and ceramic materials systems to provide improvements over currently available TBC systems. Coating reliability will be further improved with the development of an improved lifing model and NDE techniques. This will be accomplished by conducting the following program tasks:

- II.1 Process Modeling
- II.2 Bond Coat Development
- II.3 Analytical Lifing Model
- II.4 Process Development
- II.5 NDE, Maintenance and Repair
- II.6 New TBC Concepts

Phase III of the program will proof test the best of the newly developed TBC systems on airfoil sections in a combustor test passage at the Westinghouse Science and Technology Center.

Table 1: HVOF Study 1 Coating Trends

Desired Response	H2 Flow	Ratio O_2/H_2	Orifice	Feed Rate	Powder	Carrier Flow
low porosity	High*	High	Large	Low	1	Low
low oxide	Low	Low	Large	High	2	High
rough surface	Low	Low	Small	High	2*	High
high efficiency	Low	Low	Large*	Low	2	Low
HVOF Study 2	**X**	**X**	**Large**	**High**	**2**	**High**

An * indicates the variable with the greatest effect for a given response and underlined values show only a minor effect.

Levels are given to minimize porosity and oxides and maximize surface roughness and deposition efficiency.

As incicated in the table 1, there is no clear cut set of variables that will optimize all of the response variables. Some of the effects of process variables on the response variables are, however, neglegible. As a result, the orifice size, feed rate and powder can be eliminated from further investigation as shown above. In addition, carrier gas flow is eliminated as a variable as its response is secondary compared to fuel flow and oxygen to fuel ratio.

Given the results of the initial optimization study, HVOF optimization study 2 was conducted to examine the effects of fuel flow and oxygen to fuel ratio. Coatings were deposited utilizing three levels of these two variables. The metallurgical evaluation is underway. The optimal microstructure resulting from this study will be used for furnace evaluation.

In addition to the above optimization, a reproducibility study is being conducted. This simple study compares coatings from six different HVOF runs to evaluate reproducibility of the coating process, a vital requirement for coating reliability. The baseline coating parameters will be used for this study. Evaluation of the coating will consist of metallography only. Substrates have been shipped to the coater for this study.

SPS Optimization - Previously, bond coats have been deposited by SPS using either the Miller shroud or the Drexel shroud. In the current reporting period, metallography has been completed on shrouded plasma spray, optimization study 1 bond coats. Coatings deposited using the Miller shroud resulted in extremely poor coating quality. Most notable were the deposition efficiencies which were approximately 40% less using the Miller shroud as compared to coatings applied using the Drexel shroud (figure 1). The Miller shroud was therefore abandoned for further evaluation. Evaluation of the bond coats was conducted in a manner similar to the HVOF process. The process and response variables are shown in Table 2.

Technical Progress Report

Task II.2 Bond Coat Development

Task II.2.1 Bond Coat Deposition Process

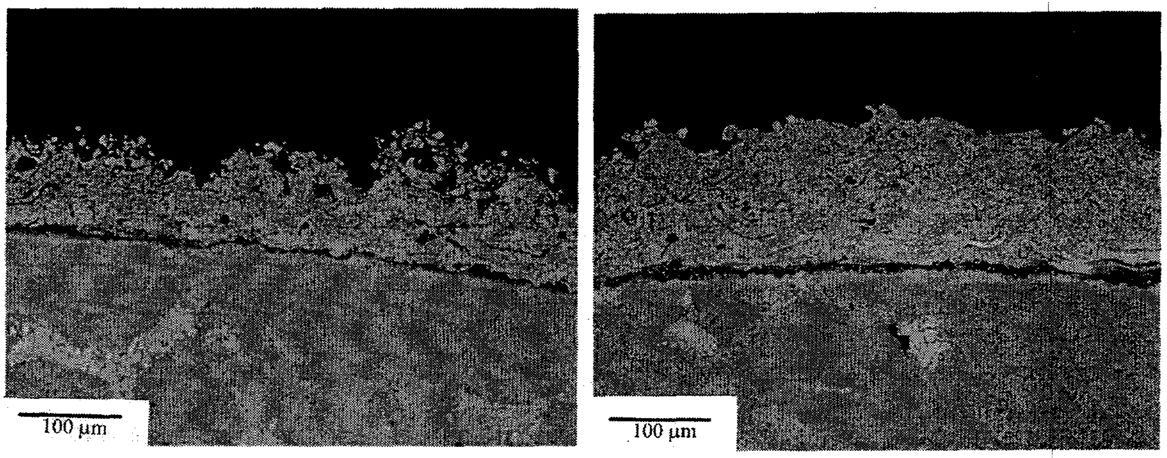
Coating process optimization continued for the deposition of bond coats using high velocity oxy-fuel (HVOF), Gator Gard, shrouded plasma spray (SPS), low pressure plasma spray (LPPS) and electron beam-physical vapor deposition (EB-PVD). In addition, discussions were initiated to examine the merits of axial powder feed to plasma spray and shrouded plasma spray.

Baseline system evaluation - All process optimization and furnace evaluation in this task is being performed using the bond coat composition Co-32Ni-21Cr-8Al-0.5Y (hereafter referred to as CO-211). Coatings to be evaluated are being applied to the component alloys CMSX-4, MarM002, and IN-939. CMSX-4 is the baseline substrate alloy for this program and LPPS is the baseline deposition process. Test pins have been coated with CO-211 using the LPPS method on all three substrates with an APS 8% YSZ top coat. Coatings were deposited using the vendors standard practice prior to any additional optimization. In addition, a baseline plasma sprayed bond coat + APS top coat set of test pins was coated.

Coatings have also been applied to test pins using LPPS bond coat + EB-PVD TBC and EB-PVD bond coat + EB-PVD top coat.

Furnace testing has been initiated on baseline and EB-PVD ceramic coated test pins. Testing is conducted at design and accelerated temperatures. Samples are removed periodically, out to times of 10,000 hours, for destructive evaluation. In addition, a number of pins are reserved in the furnace for testing to failure. The test pins are subject to thermal cycles consisting of removal from the furnace for 30 minutes daily. Coating failure is defined as 10% spallation or a crack running 50% of the length of the test pin (a 0.5" crack). Preliminary results will be presented at the program review on September 5, 1996.

HVOF Optimization - Bond coat microstructures were evaluated from optimization study 1. The response variables porosity, internal oxide content, deposition efficiency, and surface roughness were examined and the coatings ranked to identify trends between process variable and response (table 1). Within in the range of process parameters examined, the following trends were detected.



1a 1b

Figure 1: Shrouded plasma spray deposition of Co-211 bond coat using 1a) Miller shroud, 1b) Drexel shroud.

Table 2: SPS Study 1 Coating Trends, Drexel Shroud

Desired Response	Anode#	Amps	Shroud Gas Flow	Primary Gas Flow
low porosity	2	High*	Low	Low
low oxide	2	Low*	High	High
rough surface	2*	Low	High	High
high efficiency	2	Low*	High	Low
SPS Study 2	**2**	**Low**	**High**	**X**

From these observations, the common variables are Anode # 2, Low Amps, High Shroud gas flow rate. Building on these results, study 2 coatings look to further optimize the primary gas flow. In addition, spray distance has been introduced to accommodate changes in manufacturing necessary when considering engine components. Study 2 coating deposition has been initiated.

Gator Gard Optimization - Past experience has indicated that the plasma spray MCrAlY particle size distribution has been a major variable in determining the quality of coatings deposited using the Gator Gard process. As such, three different MCrAlY powders were introduced into the optimization test matrix: Praxair CO-210, CO-211 and Starck 415 powders. All powders have the same nominal composition with different powder size distributions. Other process variables included the spray nozzle hardware, current (high/low) and gas flow (high/low). The coatings were deposited according to the experimental design. Upon receiving the diffusion heat treatment, a number of the bond coats developed cracks in the surface. Metallurgical examination of the coatings has been initiated to evaluate the process/response trends as well as determine the cause of the bond coat cracking. These results will feed into a second optimization iteration.

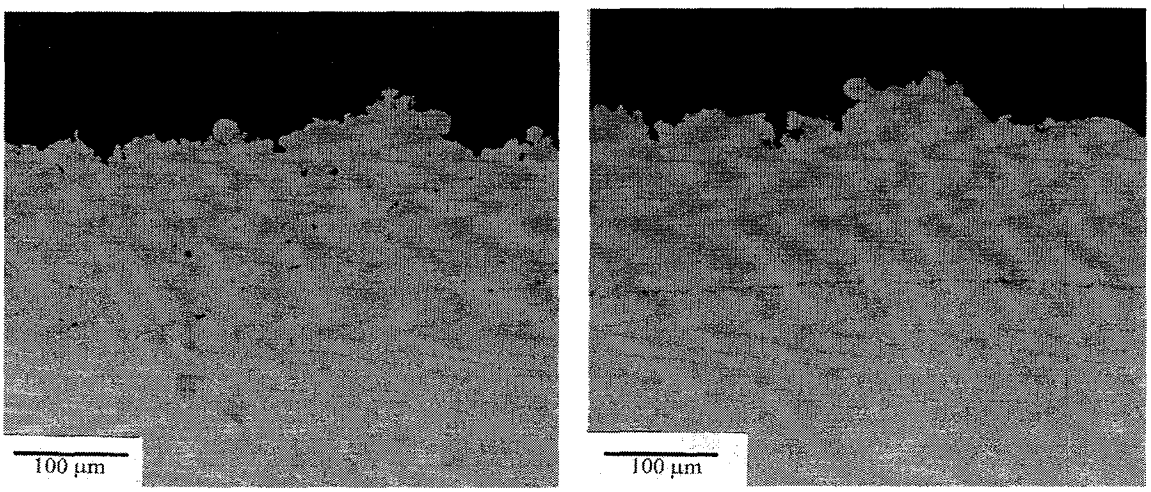
LPPS Optimization - Substrate temperature is a critical process variable in optimizing the LPPS process. To ensure that the substrate is optimized giving proper consideration to substrate temperature, the work is being delayed pendings installation of an optical pyrometer.

Axial Plasma Spray - In the past few years, new plasma spray equipment has been developed in which the bond coat powders are introduced axially along the hot plasma path. Standard equipment introduces the powder perpendicular to the plasma plasma path. Such a system promises to increase deposition efficiency, increase coating homogeneity, and decrease oxidation of the bond coat. Due to the unique features of this equipment, discussions have been initiated to introduce an evaluation of the equipment and coatings into the process optimization of the TBC development program.

Task II.2.2 Evaluate Bond Coat Chemistry

Initial coating trials have been initiated on the new bond coat chemistries. Due to equipment and resource limitations, new bond coat chemistry optimization will have to be performed serially rather than in parallel. This is not expected to cause delay.

The first of the bond coat chemistries (CO-362) has been deposited in trial runs. The resulting bond coat microstructure is shown in figure figure 2a. The microstructure of the baseline coating is shown in figure Figure 2b. Although the microstructure of the CO-362 coating is reasonable, there is still significantly more porosity than is present in the baseline coating and additional optimization is necessary. The new bond coat optimization (10 chemistries in all) will continue throughout August and into September.



2a

2b

Figure 2: LPPS deposited bond coats: 2a) CO-362 new chemistry, 2b) CO-211, baseline.

Task II.2.2.7 Diffusion Modified Bond Coats

As a proof of concept, several test pins were coated using two different slurries to modify MCrAlY bond coats for APS top coat application. Two different ceramic TBC's were applied. The coating matrix was as shown in table 3.

Table 3: Diffusion Coating Summary

Substrate/MCrAlY	Slurry Coating	Ceramic
MarM002/CO211	1501	TBC1
MarM002/CO211	1501	TBC2
MarM002/CO211	1545	TBC1
MarM002/CO211	1545	TBC2

Test pins coated with TBC1 spalled during the coating process. TBC2 coatings did adhere to the test pins. These were sectioned and placed into the cyclic furnace for evaluation. Although the coat did not perform to contemporary standards, the results were encouraging.

A significant factor in the failure of the diffusion modified coatings is the leveling effect that the slurry coating process has on the surface roughness of the MCrAlY. The degree of final surface roughness is dependent on initial surface roughness and slurry coating thickness.

To optimize the bond coat surface to receive an APS TBC, a second experiment has been devised. The test matrix consists of depositing a series of base MCrAlY coatings with various surface roughnesses. These coatings will be modified with three different levels of a slurry modification. Finally an APS top coat will be applied. The surface roughness of the of the MCrAlY will be measured before and after slurry modification and related to the top coat adhesion. This task will define the necessary surface roughness for future work.

Task II.2.2.8 Sol-Gel Bond Coats

Difficulties have been encountered in the use of sol-gel for oxygen diffusion barrier deposition due to the significant coating shrinkage associated with drying, calcining, and sintering of the coating. These issues are exacerbated when the part to be coated has a rough surface such as results from the cutting and grinding. To alleviate this issue, test pins have been polished by 1) electropolish, and 2) mechanical polish. Subsequent coating of the electropolished pin results in a uniform, crack free coating. Microscopic evaluation is being performed to determine coating thickness and the integrity of the coating. Furnace evaluation of the coating (without topcoat) is being performed in parallel to assess the coatings ability as an oxygen diffusion barrier.

Task II.3 TBC Analytical Lifing Model

Task II.3.1 Evaluate Existing Lifing Models - Complete

Task II.3.2 Develop Life prediction Model

Effort in this reporting period was spent in developing the TBC life prediction model. Programming of the life prediction into a FORTRAN code was initiated in this period. A summary of the status of the computer code development effort is presented in Figures 3 and 4. Figure 3 shows the overall structure of the TBC Life Prediction Model (TBCLPM). Subroutines that have been completed are indicated by a dark shade, while those in progress are indicated by a light shade. The unshaded boxes indicate subroutines that remain to be developed. Figure 3 shows that the main portion of the model, which serves the function of data input, applying the thermomechanical loading cycles, interfacing with the spallation model, and data output, is about 60% completed. Figure 4 shows the current status of the spallation model, which is about 30% completed. Oxidation data from several systems of interest were sent by Westinghouse to SwRI, and equations for the oxidation kinetics have been derived.

The TBCLCF failure model for APS TBC was developed based on burner rig data found in the literature. In addition, interdiffusion between the bond coat and the substrate was investigated using the finite-difference COSIM diffusion model supplied by Dr. James Nesbitt at NASA-Lewis. The original model supplied to SwRI was suitable for 1000°C only. This limitation was alleviated by modifying the coefficients in the diffusion model as explicit functions of temperature and alloy contents. The modified diffusion model is now capable of predicting interdiffusion for arbitrary temperatures. The changes in bond coat chemistry could potentially lead to failure in the TBC system. The diffusion model will be incorporated into the TBCLPM code at a later time.

Specimen fabrication of samples to derive critical mechanical properties for the model is underway. The APS TBC sintering specimens have been fabricated and are now ready for sintering. All TBC substrate materials have been machined and have been sent for coating.

Task II.4 Manufacturing Process Development

Task II.4.2 Cooling Hole Masking Technology

Application of coatings to turbine component surfaces can cause restriction of cooling holes and alter the heat management of the engine. Altered cooling air flow will lead to increased component temperatures and will shorten the life of the part. Therefore, it is critical to understand the extent of hole restriction caused by the coating process and to 1)account for the restriction in cooling hole design, 2)prevent cooling hole restriction during TBC deposition, or 3)remove the coating material from the holes after deposition.

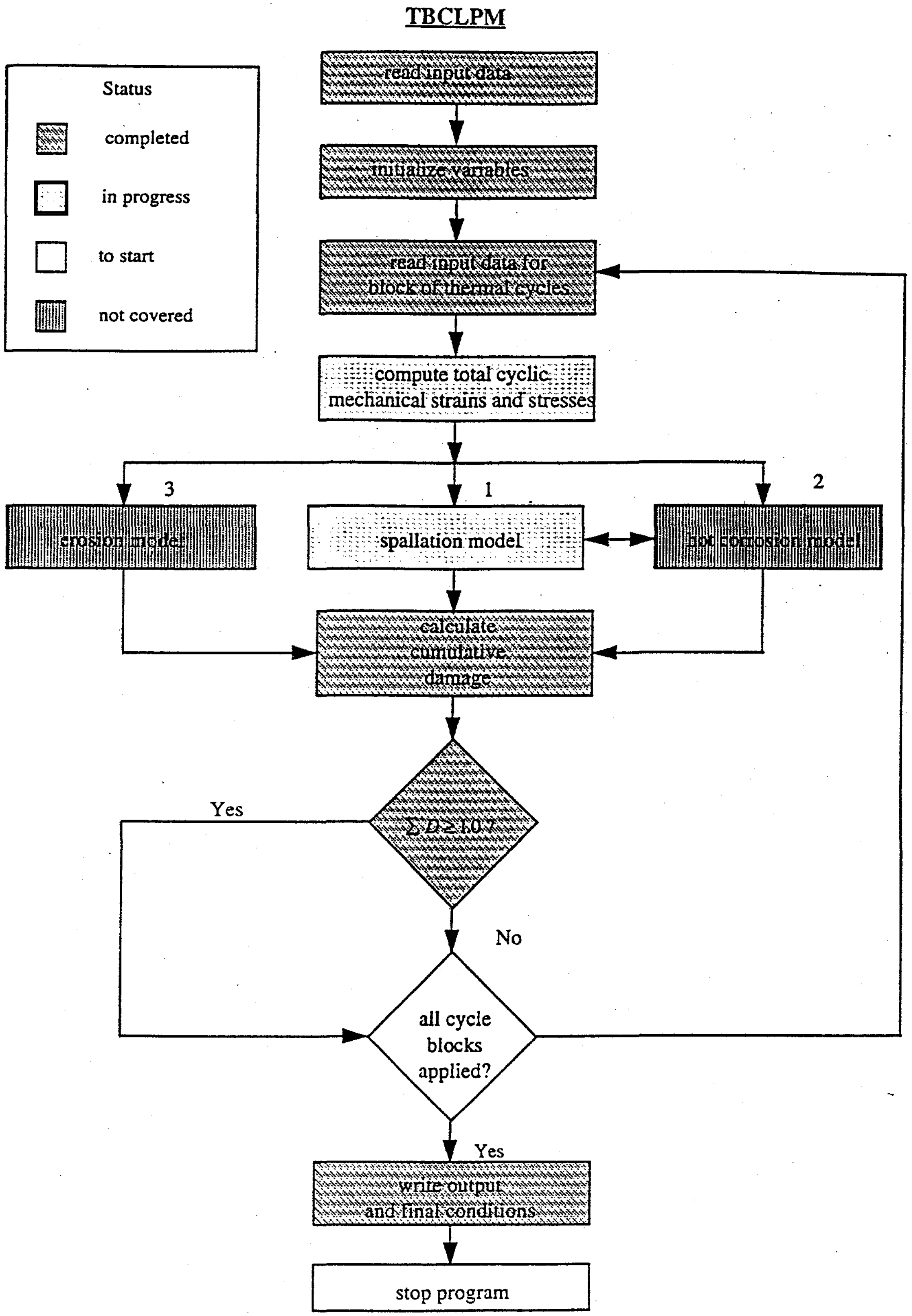


Figure 3: A summary of the status of the development of the TBC Life Prediction Model (TBCLPD) computer code

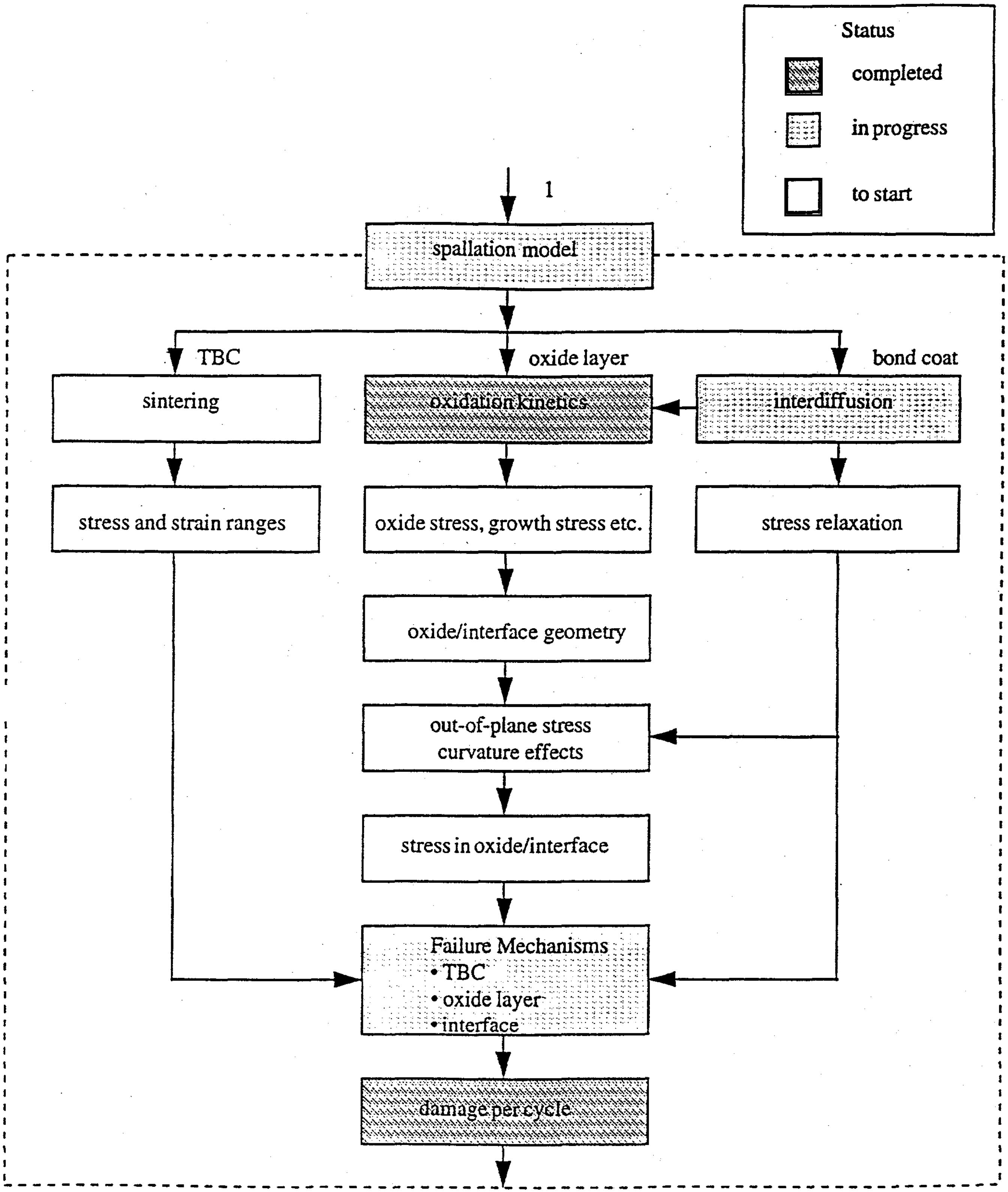


Figure 4: A summary of the status of the development of the spallation model in the TBCLPM computer code.

A number of techniques for eliminating cooling hole restriction were reviewed. Several of those techniques have been selected for coating trials. Progress was made in the areas of polymer masking, programmed spray automation, and air flow masking. Test samples were fabricated for masking and spray technique development.

Polymer masking trials were performed as discussed previously.* A heat treatment was used to burn off the polymeric masking materials. Ceramic filler particles from the masking materials remained in the holes after heat treatment. The particles were loosely caked into the holes, and they were easily removed using pressurized water wash. A thin residual layer of masking material did remain adhered to the cooling hole walls after the pressurized wash. As an alternative to the filled masks, several unfilled materials are being investigated.

Additional masking trials will be performed on test plates that have sets of simulated cooling holes. The hole diameters and angles to the airfoil surface were selected to simulate those used on typical Westinghouse blade and vane airfoils. Masking materials will include modified Praxair materials and several alternatives.

PVD-TBC samples, used for baseline comparison, have been fabricated.

Customized spray patterns and angles may be used to reduce cooling hole restriction as discussed previously.* Initial coating trials were performed using simulated cooling hole plates. HVOF and plasma sprayed MCrAlY bond coats were used during the trials. An 8%YSZ top coat was applied using air plasma spray. Two angles, 45° and 90°, were used to apply the coatings. The initial findings are: 1) the HVOF bond coat causes less restriction than the plasma spray bond coat, for a 90° spray angle; 2) hole restriction was greater for 45° degree spray than for 90° spray for both bond coats and TBC.

Task II.4.3 Hole Re-Drilling

Three machining techniques have been identified for re-shaping cooling holes after TBC deposition. Table 4 shows a list of techniques that are being evaluated. A vendor has been identified for high pressure water-jet cutting. Initial trials are being performed to verify the minimum practical jet size and the positional resolution of the equipment. Upon sufficient verification of the hardware, Westinghouse will supply coated samples for additional verification of the water-jet method. Issues to be addressed with water-jet machining include loss of tolerance on the cooling holes, and impingement of the jet on the internal cooling passages.

---

* Advanced Thermal Barrier Coating System Development, Contract # DE-AC05-95OR22242, Technical Progress Report, to the U.S. Department of Energy, June 10, 1996, Submitted by Westinghouse Electric Corporation

Table 4: Cooling hole re-machining techniques

Machining Technique	Advantages	Disadvantages	Status
Water-jet cutting	compatible with non-conductive materials	•current resolution •under cut on angles	initial trials being performed at vendor
Laser cutting	allows precise positioning of beam	•thermal stresses •heat affected zone •ceramic to metal transition	discussion with vendor
Rotary sonic milling	readily adaptable to CNC machines	•mechanical stresses	discussion with vendor

Task II.5 NDE, Maintenance, and Repair

Task II.5.1 Repair and Maintenance

Localized repair of a coating system offers the potential for considerable cost savings over general stripping and recoating of a component. Two general types of local repairs, major and minor, have been identified and will be considered. Minor repairs are intended for new or nearly new parts with a chipped TBC, but little if any bond coat degradation. Major repairs constitute a local TBC and bond coat stripping and refurbishment. For both types of repairs, it is assumed that the substrate has not been damaged.

Major and minor repairs will be simulated on flat 1.5 x 1.5 x .125 inch coupons with MCrAlY bond coats and ceramic top coats. For the minor repair coupons, grit blasting will be used to damage the top coats, simulating a chipped TBC. In the case of major repairs, damage to the bond coat will be introduced by localized, non-uniform corrosion of the bond coat, simulating a turbine blade or vane. Both single crystal and polycrystalline coupons have been machined and coated for this effort.

Coupons with HVOF bond coats and APS top coats will be used for the initial development of major and minor repair methods. These repairs will be evaluated with thermal wave imaging, metallography, furnace exposure, and bond strength testing. Based on results, the best repair techniques will be selected and further demonstrated on two types of thermal barrier coating systems: an HVOF bond coat with APS top coat and a LPPS bond coat with EB-PVD top coat. Not only will this serve to confirm the quality of the repairs, but also that the repairs are applicable to any bond coat and any TBC.

Task II.5.2 Off Line NDE

Off-line NDE consists of using thermography and eddy current techniques as they apply to TBC ceramic coatings. This work will build on ongoing R&D efforts being conducted as part of the ATS program (DOE contract # DE-FC21-95MC32267) where bond coat NDE tools are being evaluated. Reference coupons are being fabricated and will be evaluated in the ATS effort as well as in the current program. The current program will specifically examine the simple plate and step plate as indicated in the following table. These reference coupons are currently being fabricated as part of this effort.

Table 5: NDE Test specimens and evaluation method

Specimen Type (#)	Measurement	Method	Comments
Simple Plate (16)*	Thermal	IR Imaging/Diffusive	TBC Coat**
Step Plate (3)*	Electro/Thermal	EddyCurnt./IR	TBC Coat**

* Sample fabricated under ATS program
** Ceramic TBC coating applied under current program

Task II.5.5 In-Frame NDE

In accordance with the program schedule, the in-frame NDE effort was initiated in July. This kickoff meeting, attended by SwRI, Pyrometer Instrument Company Inc., and Westinghouse engineers including materials, diagnostics, and design personnel, was held on July 24th, 1996. The meeting resulted in a critical review of the need for on-line monitoring of TBC performance from both a predictive and operational point of view. The need for such monitoring technology was immediately evident and strongly supported throughout the team.

Through the course of the meeting, it became evident that no single NDE technique can address all the TBC monitoring requirements. Therefore numerous approaches to monitoring TBC performance were discussed. These techniques are being reviewed in terms of their cost, benefits and risk of success.

Task II.6 New TBC Concepts

Yttria-stabilized zirconia is the mainstay of the aero and industrial gas turbine (IGT) TBC coating industries. Significant differences in the operating mode of an IGT relative to an aero engine exist. The long time at temperature in an IGT, has created significant concerns as to whether current zirconia TBC's are capable of operating under ATS conditions.

Task II.6.1 New TBC Chemistry

Powders of new TBC compositions are being fabricated. The supplier had difficulties in obtaining the raw materials for several of these compositions which resulted in delayed powder fabrication. Delivery is expected early in August for the first of these compositions.

Task II.6.2 Microstructure: Microcracked / Segmented / Columnar

Plasma spray processing of YSZ can result in a significant range of microstructure through manipulation of the process control parameters and raw materials. Sermatech, in a research effort prior to the current program, has developed a process for applying compliant and thermal shock resistant ceramic coatings for applications that require the coating to have good abrasive properties along with substantial rapid thermal shock resistance. Although primarily being developed for aerospace applications, it is felt that the technoloy is readily adaptable for land-based TBC applications. It has been demonstrated by Sermatech that the process is capable of producing coatings (on coupons) in excess of 3 mm thickness. It is also capable of being applied into a variety of bond coats in which the surface morphology precludes the application of standard TBC coatings. This technology will serve as the basis for Task II.6.2.

The test matrix for application of the baseline technology to TBC applications has been developed. Three coating thicknesses and three densities will be investigated. Substrates have been sent for coating.

Task II.6.3 Process Optimization

Initial spray gun parameters were obtained from the TBC powder vendors and coatings applied to test panels. Various hardware modifications were necessary before spray applications could be done. A total of four top coat powders and five hardware+process parameter configurations were examined. Analyses is currently underway to determine the microstructural aspects of these coatings