

DOE/OR/22242--T16

**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

June 16, 1998

Submitted By

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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 being performed in Phase II and III of the program.

Technical Progress Report

Task II.2 Bond Coat Development

New Bond Coat Chemistries

- In the previous report, it was mentioned that three of the new bond coat chemistries showed sufficient improvement over the baseline system to warrant further investigation. A test matrix was derived to replicate previous LPPS bond coat / APS TBC performance, as well as evaluating the new chemistries as a bond coat for EB-PVD TBC's. Coating and testing are scheduled for July.

- Additionally, two of the three new bond coat chemistries will be evaluated using HVOF for the bond coat deposition. Previously fabricated bond coat powders have been sent for resizing to accommodate the HVOF equipment requirements. Delivery of the resized powder is expected in late June or early July at which time the coating optimization will be started.

Diffusion Barriers -

- Interdiffusion between the bond coat and the substrate superalloy can add to the depletion of aluminum from the bond coat and can result in superalloy elements diffusing through the bond coat to the thermally grown oxide where they have a detrimental effect on the protective capacity of the alumina. This phenomena is particularly pronounced for bond coats on superalloys such as IN-939 which have a low aluminum content (1.9%) and high concentrations of detrimental elements such as titanium (3.7%). The end result is a significantly shorter coating life when compared to the same coating over a higher aluminum superalloy such as MarM002 (5.5% aluminum).

- Four series of diffusion barriers were exposed to accelerated cyclic furnace testing as a means of evaluating the effectiveness of the barriers to substrate side aluminum depletion and superalloy / bond coat interdiffusion. In each of the four series, eight coupons were coated, each with a distinctive chemistry from one of two alloy systems. The coating systems consist of a substrate, diffusion barrier, bond coat, and TBC. The system chemistries were devised to either prevent interdiffusion and/or react with diffusing species such that they should not enter the bond coat. The cyclic furnace test results are shown in figure 1.

- Most notable is the 755 series coatings which vary in a systematic manner with chemistry. The difference in the coating performance as a function of barrier chemistry can readily be attributed to the reduced beta phase depletion in sample 755-8 as compared to 755-1. An optical image of one of the 755 series coatings is shown in figure 2. The poor performance of samples 752-7, 752-8, 753-7, 754-7, and 754-8 can be attributed to poor bond coat adhesion and is being investigated. Metallography and EDAX analysis continues to be performed in order to understand the manner in which the diffusion barrier functions.

Task II.3 Analytical Lifting Model

- The Analytical Lifting Model task was put on hold during most of the current reporting period to allow for additional thermal gradient test rig data to be generated using the Westinghouse Waltz Mill HHFTR. This rig most closely simulates the surface and bond coat temperatures and the heat flux expected in the ATS engine and is therefore critical to demonstrate the utility of the TBC life prediction model (TBCLPM) under ATS conditions. The additional information has been compiled and was sent to SwRI for analysis. SwRI will refit the model constants using the HHFTR data. Due to the delay in getting this final set of test data to SwRI, the delivery of the final report on the has been postponed until August.

Task II.4 Manufacturing Process Development - Task Complete

Task II.5 NDE, Repair and Maintenance

- Cyclic furnace testing of TBC patch repairs under nominal conditions continued throughout the quarter. During the reporting period, all repaired and unrepaired (control) samples remained in test.

- The favorable results from accelerated furnace testing of TBC patch repairs have prompted further work on patch repairs at Westinghouse. An internal program has been initiated to gather additional test data on patch repairs and transfer the technology from laboratory to production-scale components.

- The potential for recrystallization of single crystal substrates due to coating refurbishment was evaluated using flat coupons and machined creep bars. The single crystal samples were subjected to coating, creep testing, stripping, recoating, and diffusion heat treatments in order to simulate the anticipated thermal and mechanical history of a component. A range of temperatures were evaluated for the diffusion heat treatments to establish allowable limits, Table 1. A post aging heat treatment of 1000 hours at the ATS nominal temperature was added to allow for growth of any nuclei which may have formed during the earlier diffusion heat treatment. Metallography was used to identify recrystallization near the substrate surface, and a summary of the results is given in the table below. Creep bar samples which received a diffusion heat treatment and post aging contained near-surface features which may be construed as recrystallization. More detailed metallographic analysis of the features is in progress to confirm whether or not recrystallization has occurred.

Table 1: Test matrix to evaluate recrystallization limits during coating refurbishment

	as rec'd	+ diffusion HT	+ diffusion HT + post aging
coupons - as cast	none	none	none
coupons - coated	none	none	none
creep bars - as machined	none	none	possible RX
creep bars - coated	none	none	possible RX

Task II.6 New TBC Concepts

- Lab scale tests were conducted to identify new chemistries with a potential to perform better than 8YSZ as thermal barrier coatings. These lab-scale tests were directed towards evaluating long term phase stability, reactivity with the bond coat (and TGO) and sintering behavior of the new TBC ceramic candidates under ATS relevant and accelerated conditions.

- Of the first batch of fourteen new chemistries, seven of them were eliminated from further consideration due to long term phase instability. Evaluation of the sintering characteristics was performed at high temperatures with soak times up to 40 days. As shown in Fig. 3 (A-C), results clearly indicated that some of the chemistries show a noticeable tendency to densify with increasing time from A to C. Of the seven phase stable chemistries, one was eliminated due to excessive sintering as shown below. None of the chemistries were found to react with either the bond coat or TGO. Based on the above results, six new ceramic chemistries have been moved into evaluation as coatings on test pins.

- Due to the superior strain tolerance of columnar microstructure of coatings deposited by EB-PVD, the new chemistries will be evaluated for the ease of deposition via EB-PVD. Melt pool stability, coating chemistry and microstructure will be the important criteria before proceeding for further evaluation. Determination of the process parameters specific to each new chemistry is the key to reproducibly obtaining a columnar microstructure, similar to that of 8YSZ. To identify these parameters, a systematic parametric study is planned. Thermal fatigue life of the new chemistries will be evaluated relative to that of 8YSZ and the best candidate will be pursued for extensive microstructure and process optimization.

- Different PVD facilities are being considered for coating the test pins. The facilities under consideration are expected to simulate the coating conditions similar to that of commercial vendors. This will allow a smooth transfer of technology upon the identification of a successful coating. Based on the availability of machine time and capability of the facilities, a final decision is expected to be made by the end of June.

- Six promising candidates identified from the results of the lab-scale evaluation discussed above, are planned to be fabricated into ingots suitable for the EB-PVD process. The constituent powders for the new chemistries are already ordered and schedules are being planned for timely delivery of ingots for the deposition process. Upon identification of a deposition facility, first deposition trials are expected by first week of July.

- Lab evaluation of an additional twenty eight chemistries are also underway to determine their phase stability, sintering characteristics and reactivity with the TGO. All of the lab-scale evaluation is expected to be completed by the end of June. This will provide additional candidates for the evaluation as PVD coatings.

Phase III Bench Test

- Testing of Thermal Barrier Coatings under high thermal gradients to establish lifing curves for baseline TBC systems proceeded during the past quarter. The baseline APS systems has been tested at a fixed bond coat temperature, over a range of surface temperatures providing a broad range of coating life conditions. Observed failures can be ascribed to sintering and oxidation/fatigue regimes. Metallographic analysis of the test materials is on-going. Several failures associated with sintering of the APS TBC under high surface temperature conditions have been verified, Figure 4. Testing of the EB-PVD system is also proceeding. The superior performance of the EB-PVD system over APS system under ATS temperature conditions has been clearly demonstrated in the test facility.

- The test facility is currently being modified to increase the sample cooling and plasma torch power output to expand the operational temperature range of the test.

- Additional CMSX-4 substrates have been provisioned in preparation for testing of alternate TBC systems being developed under Task II.6.

III.6 Blade and Vane TBC Monitor - Feasibility Study

Discussions continue to identify a host engine suitable for demonstrating the Blade TBC monitor. While there is conceptual agreement, a detailed statement of work and engine risk assessment has to be worked out prior to begin the demonstration. Agreement on the work scope and a detailed path to demonstrating the technology is expected during the next quarter.

Accelerated Cyclic Furnace Test Results for Diffusion Barrier Samples

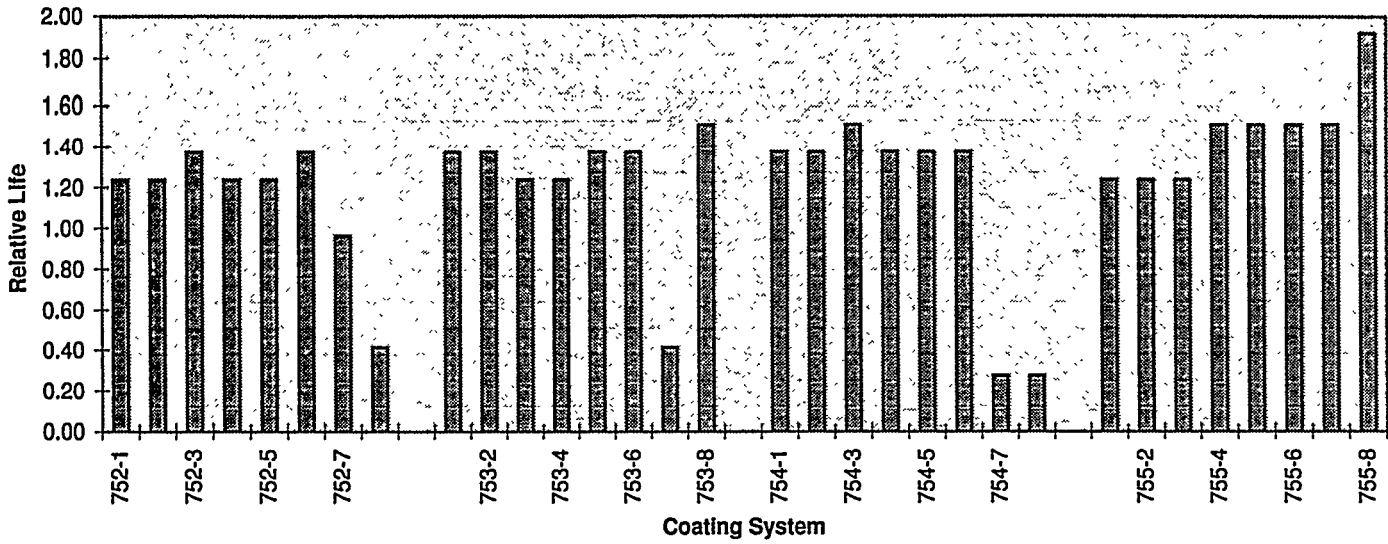


Figure 1: Normalized cyclic furnace life of coatings incorporating diffusion barriers. Each line represents a distinct composition with one of four series of diffusion barriers.

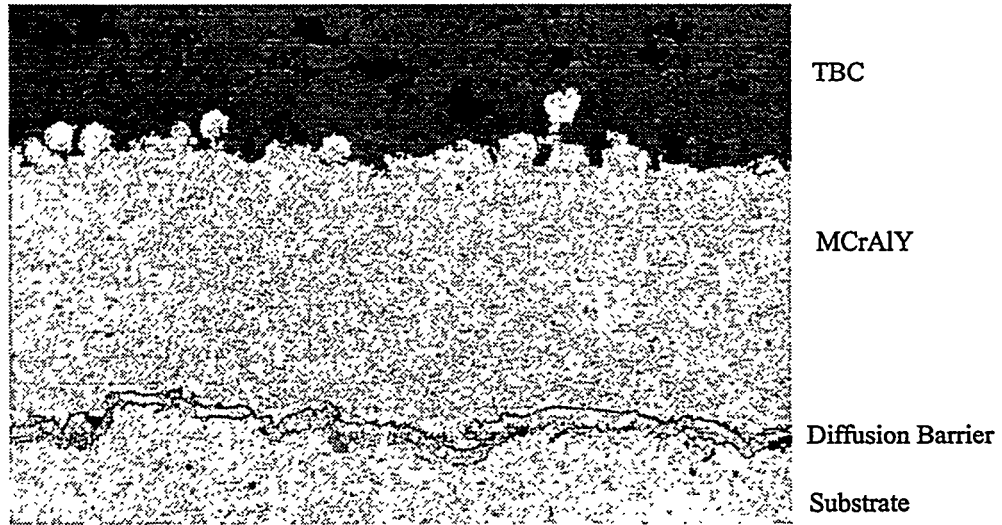


Figure 2: Optical micrograph of as processed coating system incorporating a substrate, diffusion barrier, bond coat, and thermal barrier coating layers.

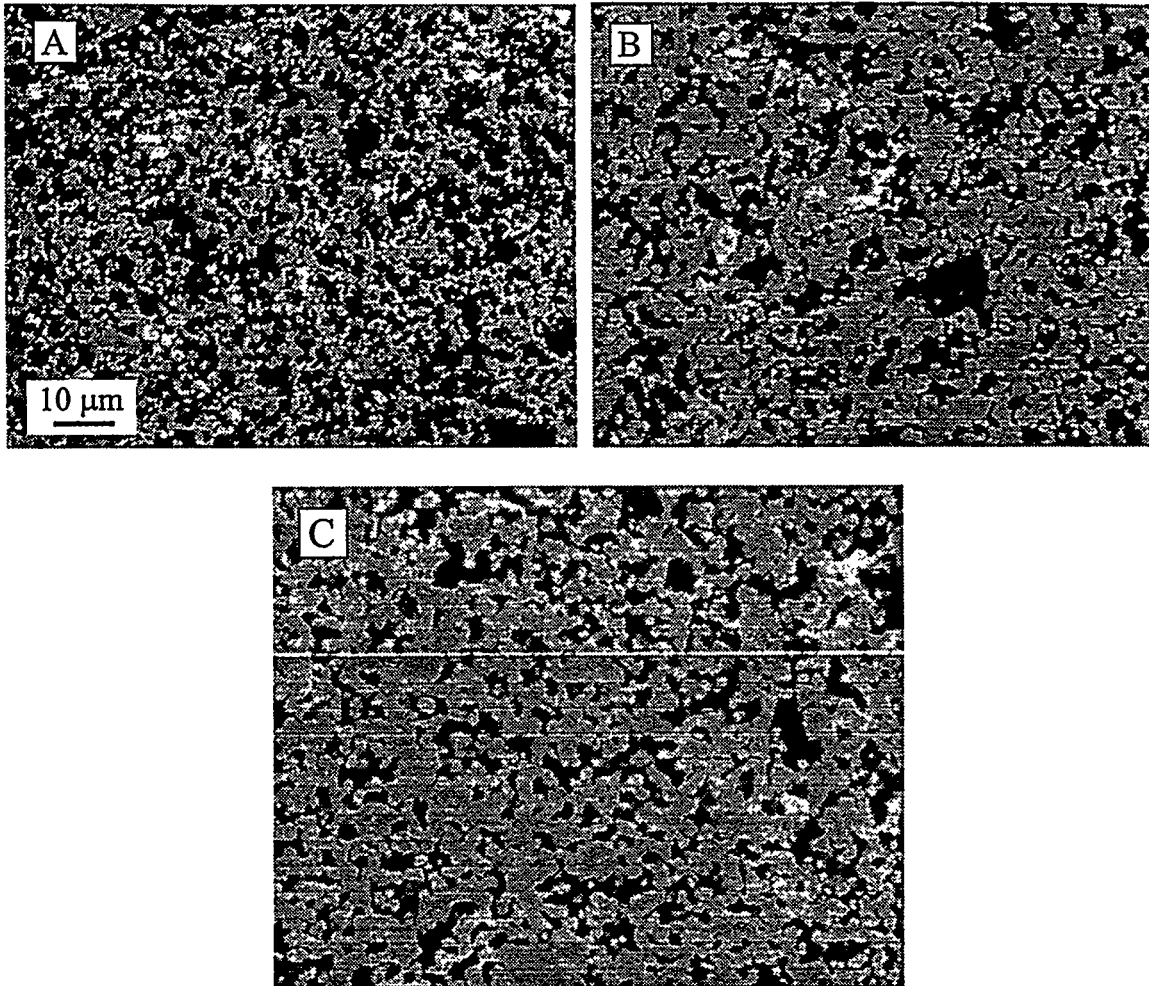


Figure 3: Micrographs showing increased densification of a phase stable candidate TBC chemistry

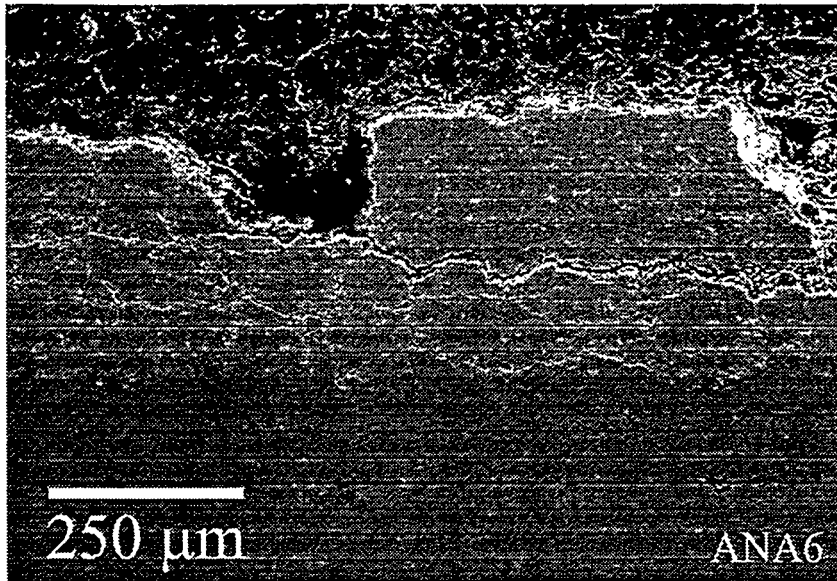


Figure 4. Microstructure of sintered APS coating after exposure in the Waltz Mill test facility.