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<u>LLOYD DEAN</u>	<u>SENIOR CONTRACTS</u>
	<u>ADMINISTRATOR</u>
NAME	AND/OR POSITION

<u>E-MAIL</u>	<u>PHONE</u>
<u>lloyd.dean@siemens.com</u>	<u>407-736-3386</u>

Organization: Siemens Westinghouse Power Corp

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Cooperative Research and Development for Advanced Materials in Advanced Industrial Gas Turbines

FINAL REPORT

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Prepared by:

Dr. Ramesh Subramanian

Siemens Westinghouse Power Corporation
4400 Alafaya Trail
Orlando, Florida 32826

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Cooperative Research and Development for Advanced Materials in Advanced Industrial Gas Turbines

1.0 ABSTRACT

Siemens Westinghouse Power Corporation has evaluated the performance of innovative thermal barrier coating systems for applications at high temperatures in advanced industrial gas turbines. Improved thermal barrier coating systems are essential to meet the increased performance and reliability requirements of advanced industrial gas turbines.

It was identified that, with an increase in TBC surface temperatures, failure of the coating system can be initiated by sintering of the ceramic top coat – 8 wt. % yttria stabilized zirconia, long before the failure of the bond coat. This program built upon on new thermal barrier coating concepts, already laboratory proven to be superior to the current state-of-the-art coatings, in overcoming the above limitations and culminate in an engine test with components coated with advanced TBC systems by October 2003.

After optimizing the manufacturing processes, the new thermal barrier concepts and materials were proof-tested at a world-class, high heat flux rig. Once proven here, the coatings were deposited onto components utilizing standard manufacturing processes and demonstrated in a commercial environment. The customer site, thus, provided a low risk, commercial testing environment prior to testing in an advanced industrial gas turbine and a fleet wide acceptance of the technology. The progress in the program has utilized a minimum risk, demonstration path for a high pay-off technology and is expected to enable a successful infusion of this technology back into our fleet of engines with significant benefits in improved reliability and performance.

This report summarizes the work performed in 5 tasks in this program. Task 1 – advanced materials development - evaluates additional compositions and concepts that will allow for determination of additional promising candidates for thermal barrier coatings. Task 2 – Advanced Materials Optimization – Optimization of the initially promising materials for application during the production process. Evaluate the production feasibility by performing initial trials. Task 3 – Turbine preparations – In preparation of the engine rainbow test, steps such as component purchase, coater availability and design reviews and production sequence were planned. Task 4 – Sub-system rig testing – Advanced coatings deposited on to specimens were tested under accelerated engine test conditions to determine the improvement over standard bond coat and TBC coating systems. Task 5 – engine demonstration – A customer was identified for the final engine test to validate the coating system performance during engine operation.

2.0 TASKS STATUS

Task 1 – Advanced Materials Concept Development

1.1 Alternate sinter resistant coatings:

Literature evaluation of alternate compositions was initiated to determine any additional classes of materials which could potentially have superior properties compared to 8YSZ. Completion of ongoing lab tests will also direct the conclusion from this task. The details are discussed in section 1.1.1.

1.1.1. Identification of additional sinter resistant compositions:

Additional compositions were selected based on the initial positive results to identify classes of materials which could potentially have superior properties compared to 8YSZ. These compositions were deposited by APS and then evaluated for sintering resistance qualitatively by observation of fracture surfaces. The results are shown in Fig. 1. A comparison of fracture surfaces before and after sintering shows that the intersplat boundaries are still existent after sintering. This indicates a potential higher strain tolerance compared to 8YSZ.

Fig. 1: Comparison of as-sprayed microstructure with aged microstructures of additional compositions in the class of sinter resistant compositions.

These compositions were then evaluated for their thermal cyclic life and surface temperature capability in Tasks 1.4.

The surface temperature capability and failure behavior of the new compositions were evaluated in the high heat flux rig. These results are discussed in Section 4.1.1. The superior sintering resistance of the new compositions showed a higher temperature limit than baseline 8YSZ.

1.1.2. Deposition parameter optimization of new oxides:

New oxides were successfully deposited by EB-PVD at the Operhall research center at Howmet Corporation. The new oxides were identified to be promising from lab scale evaluation tests of phase stability and sintering tests. The deposition trials clearly showed that melt pool stability was maintained consistently during the deposition trials. Columnar microstructures were obtained in all the three new oxides that were deposited (See Fig. 2).

Fig.2. SEM micrographs of the as-deposited microstructures of the new chemistries.

Sintering resistances of the EB-PVD TBCs were evaluated by annealing the microstructures at high temperatures and observing the changes in the microstructure. Optimization of the deposition parameters was also performed and cyclic furnace testing was utilized to screen the best parameters.

After the superior sintering resistance was identified for a few compositions, optimization of deposition parameters is necessary to meet the minimum requirements of cyclic thermal life. Fig. 3 shows the initial results that did not meet the baseline cyclic life. Upon modifications of the deposition process, cyclic furnace tests showed that the adhesion of the ceramic coating to the substrate was substantially improved. These modifications affected the adhesion at the bond coat/TBC interface and consequently the cyclic furnace life. The process parameters which have resulted in this improved adhesion need to be obtained reproducibly and also cost-effectively.

Fig. 3: Graph showing the effect of the modifications in the process conditions on the thermal cyclic life of coatings

Based on the optimized parameters identified for the new TBC compositions, further modifications were done to make the advanced coatings more cost-effective. The results from this study are shown in Fig. 4. The microstructures are very similar to each other, in spite of the differences in deposition processes. The average spallation life is also equivalent to each other. The number of specimens was increased to determine the distribution in failure time. Some early failures are observed and the reasons for this are planned to be evaluated by using a high resolution TEM. The results are discussed Task 2.3.

Fig. 4: Comparison of deposition parameters on TBC spallation life. The second set of parameters reduce the total processing cost of the coatings.

1.2. Materials property evaluation

1.2.1. Thermal conductivity:

Thermal conductivity of all the new compositions were measured for coatings deposited both by EB-PVD and APS. All the measurements of thermal conductivity were done at HTML at ORNL using their laser flash diffusivity approach. The samples were typically about 1 cm square and 300 microns thick.

Since TBCs sinter when exposed to high temperatures, the coatings were aged to different temperatures and times and then evaluated for changes in conductivity.

Initial trials on the feasibility of utilizing alumina substrates for the evaluation of thermal conductivity of the EB-PVD TBCs are complete. The results are shown in Fig. 7. The relative benefit provided by the new compositions relative to 8YSZ is clearly shown.

Fig. 5: Thermal conductivity of EB-PVD new compositions relative to EB-PVD 8YSZ. The samples were all aged at 1400 C prior to measurement.

Thermal conductivity of air plasma sprayed (APS) coatings were also evaluated. However, these were performed by using free standing 300 μm thick coatings, in two conditions – as-deposited and after aging at 1400 $^{\circ}\text{C}$ for 500 hours. Results are shown for one of the new compositions in Fig. 5. Clearly, this new chemistry provides almost about 30 % reduction in thermal conductivity. This is an added benefit over the improved phase stability and sintering resistance.

Additional compositions that were identified for their superior sintering ability were also evaluated for their thermal conductivity. For all these compositions, the coatings were aged at 1400 C. After aging, the thermal conductivity was measured and the values show that the new compositions can provide a reduction in thermal conductivity of about 20 to 50 % (See Fig. 6).

Fig. 6: Thermal conductivity of APS new compositions relative to APS 8YSZ. The samples were all aged at 1400 C prior to measurement.

It was expected that as new deposition parameters are finalized, additional data may be required. However, after several optimization trials, the new porosity values did not affect the thermal conductivity significantly.

1.2.2. Dilatometry:

Dilatometry will provide a quantitative comparison of the sintering resistance between the new compositions. Specimen preparation involved the deposition of thick APS coatings and then machining the samples with the dimension appropriate for dilatometry (3 mm by 4 mm cross-section and 25 mm long). The sintering shrinkage was measured at different temperatures and the trend in sintering behavior with temperature and time was fit to an Arrhenius equation.

After deposition of additional compositions by APS, they were all evaluated for thermal conductivity by the laser flash methodology. A compilation of all the data is shown in Fig.7. The results show that all the new compositions have clear improvement in sintering resistance. The sintering shrinkage of seven new compositions were tested at 1400 C and compared with 8YSZ shrinkage at similar temperatures. Clearly, almost a 200 C improvement was possible with the new compositions, based on improvement on sintering resistance. This improvement in sintering manifests itself as an improvement in temperature capability.

Fig.7: Comparison of sintering shrinkage relative to 8YSZ. The new compositions were evaluated at 1400 C.

Dilatometry can also be used for the determination of thermal expansion of the materials. Matching of thermal expansion of ceramics is very important for the adhesion of the ceramic coatings to the metallic superalloy substrates. The thermal expansion of the new compositions was measured using a high precision dilatometry at HTML in ORNL. The results are summarized in Fig. 8. The results show that the thermal expansions are close to that of 8YSZ and, in some cases, also exceeds the baseline thermal expansion. Therefore, an adverse reduction in spallation life is not expected due to thermal expansion mismatch. Compositions that did possess a slightly lower thermal expansion are being evaluated by cyclic furnace testing to determine the potential negative effect of the lower thermal expansion.

Fig. 8: Comparison of thermal expansion of new compositions relative to 8YSZ.

As discussed above, new compositions have a sintering resistance superior to that of 8YSZ. An improvement in surface temperature capability is definitely expected, however, the improvement is dependent on the sintering kinetics at different temperatures and time. To obtain this data, samples 3 mm x 3mm x 2.5 cm were utilized for dilatometry at 3 different temperatures 1100 C, 1200 C and 1400 C. An Arrhenius equation was fitted to the data (See Fig. 9). This now provides dependence of sintering behavior on time and temperature. This equation can now be utilized to determine the decrease in strain tolerance and when it is below a critical value, then TBC spallation could be initiated. The dependence of strain tolerance on temperature and time is essential for life prediction of TBC spallation.

Fig. 9: Dependence of sintering behavior on time and temperature for a new TBC composition.

1.2.3. Susceptibility to hot corrosion:

The specimen dimensions used for dilatometry can be used for evaluation of the effect of corrosives on the mechanical properties of the new ceramic as well. The test matrix for the hot corrosion conditions and mechanical testing are being discussed. Due to increased focus on determining the thermal conductivity, thermal expansion and erosion characteristics of the new compositions, the effect of corrosion products on new composition was not investigated. Also, it was expected that for the hot section components (especially in first stage components) the temperatures are so high that hot corrosion is not expected to be a major failure mode for the coatings.

1.2.4. Erosion testing:

Erosion bars were machined from CMSX4 slabs to fit into the erosion test rig at the University of Cincinnati. The erosion bars were then deposited with a bond coat using low pressure plasma process (LPPS) and the surface was appropriately prepared prior to TBC deposition by EB-PVD.

Erosion testing was performed at University of Cincinnati high temperature erosion rig. All the erosion testing was done at a temperature of 900 C. All the test results shown here were with a particle size of 27 microns and 120 m/s velocity. These are the extreme conditions that could be obtained within the limit of the rig capabilities. The effect of the erosion on coating thickness is summarized in Fig. 10 and 11. The effect of column angle and erodent impact angle show that there is a complex relationship between the erodent angle and the PVD column angle.

Fig. 10. Comparison of erosion rates with change in the PVD column angle to the substrate

Fig.11: By change in the PVD column angle and the erodent impact angle, the erosion rates are significantly higher than the 90/90 case. Also, note that the angle of impact can also affect the erosion behavior of the coating.

Based on the previous results, erosion testing of EB-PVD coatings established the baseline for erosion rate with change in the PVD column angle and the particle size. The erosion testing was performed at 900 C with a Al_2O_3 as the erodent and with particle sizes at 10 microns and 27 microns. Similar systematic evaluations of the new compositions were also completed and they showed a similar behavior with regards to the effect of the column angle and erodent impact angle.

1.4. Substrate/TBC compatibility

The substrate/TBC compatibility is necessary so that the adhesion of the coatings to the different substrates is established as a system. The evaluation as a system establishes the complex relation between bond coat oxidation and thermal expansion mismatch. Test pins of the substrate alloys – MM002, CMSX4, IN939, IN738 – were selected due to their use as substrate alloys in the current and advanced engines. These were coated with CoNiCrAlY bond coat (RT122) and were deposited with new TBC compositions. The cyclic furnace data generated here will establish a database for the life prediction of TBC spallation as a result of bond coat oxidation. Some of the TBC spallation results are also discussed in Task 2.3.

SUBTASK A

A report on the market analysis is attached at the end of this report.

Task 2 – Advanced Materials Optimization

2.1. Initial optimization trials

Based on the experimental results obtained in the DOE-ORNL TBC program (DE-AC05-95OR22242) it was determined that deposition at a high temperature and a slow feed rate provides growth of slender columns with negligible formation of secondary columns. By decreasing the temperature and increasing the feed rate more than standard production values, significant secondary columns were observed.

By modification of the deposition conditions, the thermal cyclic life of the coating was significantly improved. The new improved cyclic life is even better than the baseline, but this could be the effect of the bond coat quality, since the predominant mode for failure is by oxidation for the cyclic furnace testing.

Further optimization with EB-PVD coatings required some fundamental understanding of the deposition process, especially the interaction between the ingot composition and the condensate composition. This correlation between the ingot composition and the condensate composition is shown in Fig. 12.

The figure clearly shows the changes in composition during the various stages of the melt pool formation. During the initial stages, the pool is significantly away from the ingot composition. As the equilibrium between the ingot and melt pool and that of the melt pool and vapor gets closer, the condensate composition is similar to the ingot composition – the desired composition of the coating. Based on this knowledge, the

deposition trials were modified to obtain the best cyclic furnace life, coating composition and microstructure.

Fig. 12 Figure shows the effect of the ingot composition relative to the condensate composition.

After the initial optimization trials, preliminary process parameters for deposition were identified. Additional deposition results however showed that in addition to temperature and feed rate, the total chamber pressure could play a role on the microstructure. Insufficient degassing and consequently a higher pressure could result in a microstructure with more secondary columns. Additional trials under controlled conditions of low pressure were evaluated. Next deposition trials were continued to determine the effect of different vendors on the performance of coatings. This is discussed in section 2.3.

After the optimization trials with EB-PVD, deposition trials were focused on using APS as the deposition approach. The results for these attempts are discussed in detail in task 2.7.3.

2.2. Component coating trials

Blades and vanes were the target components for evaluation of component feasibility. Component coating trials were conducted at TACR (Turbine Airfoil and Coatings Repair) Middletown, NY using 501 F vanes and blades. Along with the component coating trials, deposition was achieved both on the components and test pins during a single deposition trial. For this arrangement special holders at the edge of the platform was set-up (See Fig. 13). Both the blades and vanes were deposited with EB-PVD one at a time in this trial. Metallography was performed to determine the change in the

microstructure and thickness across the airfoils and platform. These results are shown in Figs. 14 through 16.

Fig. 13: 501 F components (vanes and blades) after deposition of the advanced coatings in a production coater. Metallic tabs, ceramic substrates and metallic pins are shown attached on the surface of the component trials. Each of these tabs can then be evaluated for microstructure and thickness.

Results:

- TBC Successfully Deposited
- 7 to 10 mils across Inner Shroud
- Column angles from 34° to 40°

Fig. 14: Results from the metallic tabs on the inner shroud of the coated vane. The microstructures show that they are inclined at an angle to the platform with a maximum of about 34 to 40 degrees. The thickness of coatings ranged from 175 to 250 microns (7 to 10 mils).

Fig. 15: Results from the metallic tabs on the airfoil of the coated vane. The microstructures show that the PVD columns are almost perpendicular to the platform with a maximum of about 8 degrees tilt. The thickness of coatings ranged from 225 to 300 microns (9 to 12 mils).

Fig. 16: Results from the metallic tabs on the outer shroud of the coated vane. The microstructures show that they are inclined at an angle to the platform with a maximum of about 30 to 40 degrees. The thickness of coatings ranged from 150 to 250 microns (6 to 10 mils).

After the completion of component coating trials with EB-PVD of new compositions, the focus was shifted to application of the same compositions with another production process, air plasma spray, to increase the applicability of the advanced compositions. Initial trials were focused on establishing the appropriate parameters for APS TBCs and then followed by component coating trials.

Advanced TBC coatings were deposited on 501F Row 1 vanes to establish production feasibility. Microstructure and thickness profile across the components were evaluated by metallography. Tabbed vanes, shown in Fig. 17, were utilized to establish the microstructure and thickness.

Fig. 17: Typical picture of a vane with tabs to establish the microstructure and thickness of the TBCs.

The thickness distributions clearly show that for the first attempt on deposition trials, the thickness is consistent across the component. For the airfoils, the thickness consistency differs as a result of the composition variation. Further deposition trials and optimization is required to improve the thickness distribution due to the difference in parameters. A thickness distribution on the platforms and fillets is observed, where the deposition angle could affect the TBC thickness and microstructure. However, in these deposition trials, the thickness and microstructure were very similar to that on the airfoil indicating a very homogenous coating property across the component surface.

The typical microstructures obtained at different locations on the component and they meet the required porosity level in the coatings.

2.3. Final Optimization trials

After identification of the initial process window for the deposition of the advanced coatings, further deposition trials were completed to determine the differences in the coatings performance from different vendors. Fig. 18 shows that several modifications (designated as A, B and C) for the new compositions were evaluated at vendor #1 and

vendor#2. The effect of the modification “C” showed that they had similar coating performance. This is a very important result since this allows the use of both vendors for production of the advanced coatings.

Fig. 18: Comparison of the effect of modifications on TBC spallation life between different vendors.

The fundamental understanding was established by utilization of a FIB/STEM analysis of the TGO/TBC interface. Figs. 19-22 show that the spallation life is correlated to the local microstructural interface between the TGO and TBC. After optimization, the spallation life is significantly better than initially obtained.

Fig. 19: A typical STEM microstructure of the bond coat/TBC interface showing the formation of a mixed oxide zone between the bond coat and TBC

Fig. 20: STEM microstructures are being utilized to understand the differences in the performance of TBCs from different vendors and also batch to batch to variations.

Fig. 21: Modifications to the deposition processes yielded significant improvements in TBC spallation life. The reasons for this are expected to become available by a detailed analysis using STEM.

Fig. 22: Improved results of TBC spallation life based on a fundamental understanding of TBC failure

For the APS process, based on the initial results on the microstructures obtained from the component coating trials, further optimization trials both for test coupons was performed. A systematic design-of-experiments was planned based on the deposition parameters of the plasma gas such as H₂, N₂, carrier gas, voltage and current. The mils/pass and the porosity was determined for these deposition trials. Typical microstructures are shown during the optimization trials. Fig. 23 shows the microstructures and the corresponding porosity values from the image analysis software. The porosity values are then correlated back to the TBC spallation properties and surface temperature limits.

Fig. 23: Comparison of the porosity levels within the TBCs by image analysis for the different compositions.

2.4. Production feasibility

By demonstration of the component coating trials in section 2.2 the feasibility of utilizing a production EB-pVD coater was considered successful. The next step was to establish the logistics of the production capability of the advanced coatings. This was achieved by considering the differences in process parameters and any additional variations from the standard production process. No significantly different arrangements were necessary to realize the deposition of the new compositions. Ingot preparation is key to obtaining the homogenous microstructure and thickness.

For air plasma spray process for advanced TBC deposition, the production feasibility has clearly been demonstrated, as shown in Section 2.2. The components were coated with current production hardware. Deposition parameters require adjustment as a result of differences in compositions; however, they did have an acceptable thickness and microstructure distribution.

The production feasibility for deposition of advanced coatings was demonstrated on vanes and blades. Figs. 24 and 25 show the typical component and the thickness obtained across the midspan of a blade airfoil. The thickness was uniform for a first attempt for deposition across the airfoil. Further optimization of the TBC thickness is required to obtain a tighter thickness control.

Fig. 24: A 501 F blade coated with a new TBC composition.

Fig. 25: Typical TBC thickness obtained across the midspan of the airfoil

2.5. Ingot/powder preparation and optimization

The new compositions, due to their different sintering behavior, require a different heating cycle for the obtaining a uniform density across the ingot height and diameter. Several different ingots cold pressed to various densities were heat treated to multiple sequences to determine the effect on ingot density and homogeneity. Due to the higher temperature capability of these ingots and different thermal conductivity, targets for density were initially considered to be different than the standard 8YSZ. With this target, the heating cycles were established to meet the target density.

For reproducible microstructures of APS TBCs, the particle size and powder preparation were optimized. After identification of the process window for a given particle size

distribution, this will be varied to determine the optimum range. The powder preparation process also allowed for a cost-effective approach for the new compositions. The particle size is currently maintained within a tight window to allow for reproducibility in results.

2.6. CVD/solution Deposition process scale-up

The scale-up of the CVD/solution deposition equipment was evaluated after the initial deposition trials, which are discussed in tasks 2.7.1 and 2.7.2. The deposition trials using CVD showed that the results were consistent with previous coupon tests. Therefore, the current set-up for deposition trials used for the deposition of the coatings on to components was sufficient without any further modifications or scale-up.

2.7. Cost effective processes

2.7.1. Evaluate solution deposition trials:

Solution deposition trials were utilized for improving the sintering capability of EB-PVD coatings. These were done by utilizing the standard viscosity and deposition conditions already well established by COI. SEM observations showed that coating modification was inappropriate. Additional deposition trials with a focus on reducing the viscosity of the sol and utilizing different approaches, such as by ultrasonic vibration or use of high pressure are being considered. Initial results are shown in Fig. 26.

Fig. 26: The superior sintering resistance of EB-PVD 8YSZ coatings after a solution based modification.

Based on comparison with ease of deposition of the CVD process, the solution based deposition process is not considered for future development for cost-effective coating of components.

2.7.2. Evaluate the potential of a CVD process:

Initial trials for CVD modification of EB-PVD coatings were performed. This involved the deposition of three different thicknesses of the CVD coatings. Each of the samples was then aged in a furnace at 1400 °C. SEM observations after the sintering step showed that the deposition of CVD coating to the thickest value leads to the most sinter resistant microstructure.

Additional trials were completed to optimize the deposition parameters. Initial results are shown in Fig. 27. There was no significant difference found in the deposition temperature of the additional coating step on the final sintering resistance.

After exposure to 1400 C

Fig. 27: The superior sintering resistance of EB-PVD 8YSZ coatings after a vapor based modification. The result is shown for 3 different temperatures. Sintering performance is pretty even across the coatings.

The CVD process for the advanced coatings was evaluated based on the results from deposition trials. The details of the success on the component coating trials were determined in each zone of the airfoil (See Fig. 28). Both metallic tabs and ceramic tabs were evaluated for deposition by CVD.

Fig.28: Different zones on the airfoil were deposited with CVD and the microstructure reproducibility was determined.

Deposition trials were completed on a component using the CVD process. Fig. 29 provides an overview of the set-up holding the component and the deposition trials. Thermocouples are attached to the component to aid in the measurement of temperatures during the deposition trials.

Fig. 29: Set-up for the CVD deposition on to gas turbine components.

Deposition trials were completed on a component using the CVD process. The results matched those from the coupon tests. These results show that the current set-up can be used without any scale-up.

2.7.3. APS coating evaluation

One of the new compositions was manufactured for APS by spray drying and sintering of the mixed oxides. Standard process parameters were used for the deposition of the new composition by APS. The resultant coatings were evaluated for the sintering resistance when used as free-standing coatings. These samples will then be evaluated for changes in thermal conductivity and microstructure by evaluating changes in thermal conductivity and microstructure (See Fig. 30)

Fig.30: SEM observations of a fracture surface of an APS coating before sintering and after sintering at high temperatures

During the APS deposition trials of the new compositions, an observation was made regarding the effect of particle size on the coating composition. The preliminary results showed that the reduction in particle size resulted in more deviation from the desired composition. The differences in the vapor pressure which is typically considered in EB-PVD coating composition variation also should be considered for APS coatings. See results in Fig. 31 & 32.

Fig. 31: The variation in particle size with different batches can result in composition variation.

Fig. 32: Vapor phase calculations which show the difference in vapor pressure although lower between the constituents, the absolute vapor pressures are higher.

Based on the results on the effect of particle size and vapor pressure on the TBC composition, further deposition trials are underway to determine the appropriate process window and particle size.

Two deposition trials were conducted with the new compositions to determine the TBC spallation life relative to 8YSZ. A cyclic furnace testing technique was utilized to determine the extent of ceramic adhesion to the substrate. Five new compositions were evaluated in these trials. This provides an additional information regarding the stability of the coating life, as deposited by APS.

The results show that there is a significant improvement in TBC spallation life from the first trials to the second trials. The deposition parameters were changed to adapt to the material properties such as thermal conductivity, specific heat and density of the new compositions. The results from the second trials still need some improvement relative to the baseline data. Metallographic evaluation of the coatings showed that the sprayed density of the new coatings is slightly higher than the baseline coatings. (See Fig. 33). Porosity and intersplat boundaries are not as predominant in the new coatings as in the baseline 8YSZ.

Fig. 33: Comparison of density of as-sprayed coatings of new compositions versus 8YSZ.

The spray parameters were changed to introduce additional porosity in the coatings and also result in an increased spallation life. (Fig. 34)

Fig. 34: Comparison of spallation obtained in the initial trials versus the second trials. The second trial has resulted in increased porosity.

These coatings with the new parameters were evaluated at the high heat flux test facilities and the initial results are discussed in section 4.1.1.

Task 3 – Turbine preparations

TBC performance data was necessary to initiate the risk assessment and procurement of parts for the rainbow engine test. The work was initiated upon completion of initial sub-system rig testing (Task 4) to enable a risk analysis.

Based on the results of the component coating trials and the superior performance of the TBCs during high heat flux testing, the risk level was clearly expected to be low. Design reviews were initiated with the preparation of purchasing department specification (PD Spec) with the description of the product with the coating thickness and microstructure.

Based on the preliminary surface temperature limits of the TBC, an appropriate TBC thickness could be calculated based on the heat flux for the component. The drawings need to be changed to reflect the different TBC thickness.

Both PD specifications and drawings were released. A customer was identified for the engine test in the spring outage of 2003. Further discussions and negotiations were required to determine the feasibility of installation of the components with the new coatings at this customer site.

A design review was held to evaluate the risk of introduction of the advanced coating systems in an engine at a customer site. There were no risks identified. The benefits from these coatings would be very significant in terms of increasing part life and component performance.

All tasks under Task 3 are complete. The coated parts were installed in an engine to meet the spring outage opportunity. Follow-up of the performance of the coatings will be completed in Task 5.

Task 4 – Subsystem rig testing

4.1.1. High heat flux rig testing of advanced TBC

Advanced TBC composition deposited by EB-PVD was tested under a very high thermal gradient and cycled every hour. The testing results in an extremely high cooling and heating rates resulting in transient stresses which could significantly affect the TBC spallation. The surface temperatures of the TBC were adjusted so that the TBC surface was expected to sinter considerably and result in a significant increase in the driving force for TBC spallation. Fig. 35 shows a typical picture of the specimen utilized in testing at the Westinghouse Plasma Center. The sample is internally cooled by compressed air and externally heated by a plasma torch. The thermal gradient is controlled by monitoring the cooling flow and the plasma torch power. The surface temperature is maintained by a infrared pyrometer.

Fig. 35: Picture of the internally cooled high heat flux test rig specimen during operation.

A specimen, designed for testing at the high heat flux rig testing facility, was coated with EB-PVD 8YSZ TBC and then followed by a CVD process. This sample was then tested under a high heat flux. The specimen was subjected to surface temperatures above the phase transformation limit of 8YSZ and the bond coat temperature where oxidation was not a failure mode with 60 minutes hold time at the surface temperature. The sample was subjected to more than 1000 cycles without failure. In comparison, baseline specimens coated by EB-PVD 8YSZ failed in half the time. This is an improvement of at least a factor of two in EB-PVD life.

SEM analysis of the failed specimen showed that the columns had deteriorated at the surface, similar to the failure of 8YSZ coatings. XRD of the surface indicates the formation of monoclinic phases. Excessive amount of porosity is also evident on the surface – this is possibly due to the rearrangement of the pores within the columns. Additional modifications to this coating are required prior to another test.

A specimen, designed for testing at the high heat flux rig testing facility, was coated with a modified EB-PVD 8YSZ TBC system. The sample did not fail after exposure to twice the time of baseline TBC.

SEM analysis of the failed specimen showed that the columns had deteriorated at the surface, similar to the failure of 8YSZ coatings. XRD of the surface indicates the formation of monoclinic phases. Excessive amount of porosity is also evident on the surface – this is possibly due to the rearrangement of the pores within the columns. It is clear from this examination that further modification of the coating is necessary to improve the TBC performance.

Tests under a thermal gradient have shown that the EB-PVD 8YSZ TBCs undergo significant changes within the microstructure. A typical microstructure for 8YSZ TBC upon failure under a high heat flux is shown in Figs. 36 and 37.

Fig. 36: Typical spallation of EB-PVD TBC due to sintering of columns

Fig. 37: Metallographic cross-section of the TBC next to the spalled area. Clearly the effect of phase transformation and sintering together results in severe degradation of the TBC

Fig. 37 clearly shows the degradation of the coating due to sintering and phase transformations on the microstructure. The top surface, exposed to the high temperatures, has become very porous perhaps due to the volumetric changes between

the monoclinic and tetragonal phases during thermal cycling. This probably results in a very highly porous area. Below this area, columns are seen to be sintered. These reduce the strain tolerance of the coating and can result in early spallation.

In comparison, for the same conditions that were used to test the baseline EB-PVD 8YSZ coatings, new compositions were tested under a thermal gradient. The advanced TBC lasted for a duration twice as long as the baseline EB-PVD 8YSZ. Upon spallation, the sample was broken in half and evaluated for any microstructural changes. The fracture surfaces clearly show the existence of columnar features. The intercolumnar spacing is clearly intact with minimum damage to the surface of the TBC. Only 25 microns of the 300 micron TBC is affected due to the high temperatures (See Fig. 38).

The success of the new composition to show very negligible sintering and microstructural change is very promising. The severity of the thermal cycling establishes the very low risk level of applying these coatings on to gas turbine components.

Fig. 38: Fracture surface observations of the EB-PVD new TBC composition tested under a thermal gradient

Advanced TBC compositions deposited by APS were tested under a very high thermal gradient and cycled every hour. The testing results in an extremely high cooling and heating rates resulting in transient stresses which could significantly affect the TBC spallation. The surface temperatures of the TBC was adjusted so that the TBC surface was expected to sinter considerably and result in a significant increase in the driving force for TBC spallation

The different TBCs were tested at equivalent surface temperatures to allow for a comparison of the TBC spallation time relative to 8YSZ. The objective was to fail the

TBC within the ceramic as a result of the TBC sintering. The spallation lives of the new compositions were at least 5 times that of the standard APS 8YSZ TBC. The impact of this improved TBC spallation life on the temperature capability of the coating is very significant and could potentially allow for a higher temperature capability.

Spallation sizes and locations vary for each of the materials. Metallographic cross-section of these coatings is pending and it is expected to provide an insight into the failure modes of these coatings. Changes in microstructure will also be evident from these tests.

The superior TBC spallation life from the initial trials already indicates that the superior sintering is important. Although these results are from the first deposition trials, the survival of the coatings for the severe thermal cycling indicates a very low risk level of applying these coatings on to gas turbine components.

The time to spallation showed a strong correlation to the surface temperature and enabled the determination of a preliminary surface temperature limit for operation for defined component lives. These surface temperatures provide a key parameter for the component designers to utilize for their calculations.

The microstructures show that the spallation is localized both on the surface of the coatings. No additional delamination is seen at the bond coat TBC interfaces. The spallation behavior is similar to 8YSZ in that the failure occurs within the TBC with no contribution from the bond coat oxidation. The change in temperature also does not affect the spallation behavior of the coatings.

A closer observation of these microstructures also shows the existence of inter-splat boundaries which exist even after exposure to very high temperatures. This is a clear indication of the superior sintering characteristic of the compositions. Although these results are from the first deposition trials, the survival of the coatings for the severe thermal cycling indicates a very low risk level of applying these coatings on to gas turbine components.

Several additional tests were done for the different compositions and all have shown an improved TBC spallation life and consequently a higher TBC surface temperature limit. The testing provided a comparison of the performance of the different compositions relative to baseline 8YSZ coatings. TBC surface temperatures along with the thermal conductivities provide the key parameters for the designers to define the heat transfer characteristics of the coating.

Task 5 – Engine demonstration

Gas turbine components with the advanced high temperature coatings were installed in a 501 F engine at a customer site in the June 2003. The engine is operational at full load and generating power. The parts are incorporated in the first stage of the turbine section – the hottest parts in the engine.

The parts were shipped to the qualified coating vendors for deposition of the advanced coatings. The parts were coated and assembled according to specifications. The parts were shipped to the customer site and installed during an outage. The location of the

parts was ensured such that there were statistical distribution within the hot and cold zones to obtain a good comparison with the baseline coatings.

A combustor inspection was conducted in the fall of 2003 and the initial observations showed that the advanced TBCs were performing successfully at these high operational temperatures. (See Fig. 39).

Fig. 39: Boroscope inspection of an advanced TBC composition tested in a 501FD engine. Pictures show successful operation with no failures.

A cover lift after 8000 hours showed the same result and provided a validation of the technology (See Fig. 40). It emphasized that the application of TBCs with superior sintering resistance can increase the component integrity significantly and provides the customer benefits of reduced repair costs and also better engine performance.

Fig. 40: Part examination after 8000 hours cover lift shows no TBC spallation or degradation. This completes the full validation of the advanced TBC system as outlined in the initial proposal.

3.0 NEAR TERM PLANS

With the successful completion of the program, advanced coatings are available for implementation in our existing fleet for advanced engines. The near term plans include the incorporation of these advanced coatings as a part of component design and gain significant advantages with regards to component integrity and engine performance.

4.0 PROGRAM ACHIEVEMENTS

KEY MAJOR ACHIEVEMENT:

1. Gas turbine components coated with advanced coatings developed in this program performed successfully after 8000 hours of baseload operation in one of our customer sites with a 501F engine. The operating temperatures and conditions were demanding compared to the rest of the fleet. were installed in 501F engine at a customer site.

The tests were monitored after 2000 hours via a boroscope inspection and also after 8000 hours after a cover lift. The compositions performed very successfully and increased component integrity compared to baseline 8YSZ compositions – a major achievement of the program.

The results of this program have enabled the inclusion of these advanced compositions as part of the new component designs. A significant benefit is expected from increased component integrity and engine performance.

The compositions developed in this program have enabled the advancement of the coatings technology critical for the needs of industrial gas turbines.

ADDITIONAL KEY ACHIEVEMENTS:

1. Preliminary demonstration of the superior sintering resistance of the advanced chemistries was demonstrated for both EB-PVD and APS chemistries. This was done both qualitatively and quantitatively by dilatometry.
2. Correlation of the superior sintering resistance to thermal conductivity was clearly demonstrated. The benefit of a lower thermal conductivity is extremely valuable for component life extension.
3. Modification of the sintering capability of EB-PVD coatings was demonstrated by two techniques – solution and vapor based approach. Both approaches yielded very good results in the improving the sintering resistances. High heat flux rig testing of a PVD coating system with modifications lasted twice as long as the standard baseline PVD 8YSZ. This provides a significant influence on the performance capability of coatings

4. For EB-PVD coatings, detailed evaluation of the bond coat/TBC interface for 8YSZ showed that the mixed oxide zone of zirconia and alumina is very critical to the adhesion of the TBC to the bond coat. This was evident also for the new compositions and provided new insights into improving the cost-effectiveness of the deposition process and also the reliability.
5. Erosion testing of the baseline 8YSZ under different erodent angles and PVD column angles was completed. It provided a new insight in the failure mechanisms of these coatings. The variation in PVD column angles and erodent angle is clearly expected in a component and this is a real indication of the erosion expected in an engine exposed to very large particles and high volume of erodent.
6. Significant optimization trials were done for APS of new compositions and they resulted in improved TBC spallation life. Key parameters for consistent deposition quality were identified.
7. High heat flux testing of new compositions deposited by APS was completed successfully. For the first time, the advantage of a superior sintering behavior of a coating was demonstrated under severe thermal gradient. The successful demonstration reduces the risk level for application of coatings on to gas turbine components.
8. High heat flux testing of the new compositions clearly showed a correlation between the TBC spallation life, failure mechanisms and the test surface temperature. This enables the establishment of a preliminary surface temperature limit. This is essential for the heat transfer calculations to determine the appropriate TBC thickness based on the heat flux in that location.
9. Demonstration of the component coating capability of advanced compositions. Coatings were deposited on the 501F vanes and blades. Thickness and microstructure met specifications for production components. Additional modifications are being evaluated for making the process cost-effective and production feasible.

5.0 VARIANCES OF SCHEDULE AND COST PLANS

There were no deviations from the final schedule and cost of the total program. Deviations during the course of the program were detected due to the usual variations in R&D activities. All deviations were overcome by the end of the program.

6.0 OVERALL PROGRESS AND CONCLUSIONS

The project achieved the goals set forth in the initial proposal. The objective of evaluating the performance and temperature capability of advanced coatings by rig testing successfully showed that the coatings had a superior performance to the baseline 8YSZ coatings. After establishing the superior performance and determining the relatively low risk level, gas turbine components were deposited

with advanced coatings and installed in a customer engine. This provides a complete cycle from the development to installation in an engine and determination of the performance in an engine. This low risk demonstration has enabled a fleet wide application for these coatings. An overview of the approach is shown in Fig. 41. The high pay-off is being realized with significant efforts towards design implementation of the advanced coating capabilities both in the existing fleet and new designs.

Fig. 41: Overview of the technical approach utilized for the evaluation of advanced compositions and implementation in the engine.

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The tests were monitored after 2000 hours via a boroscope inspection and also after 8000 hours after a cover lift. The compositions performed very successfully and increased component integrity compared to baseline 8YSZ compositions – a major achievement of the program.

The results of this program have enabled the inclusion of these advanced compositions as part of the new component designs. A significant benefit is expected from increased component integrity and engine performance.

The compositions developed in this program have enabled the advancement of the coatings technology critical for the needs of industrial gas turbines.