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FOR THE DEPARTMENT OF ENERGY
ORNL-27 (4-00)



C/ORNL04-0698

CRADA Final Report
for
CRADA Number ORNL04-0698
**Structurally Integrated Coatings for Wear and Corrosion (SICWC):
ARC LAMP, INFRARED (IR) THERMAL PROCESSING**

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U.S. Department of Energy

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FINAL CRADA REPORT

**Structurally Integrated Coatings for Wear and Corrosion (SICWC):
Arc Lamp, Infrared (IR) Thermal Processing****ABSTRACT**

The primary goal of this Cooperative Research and Development Agreement (CRADA) between UT-Battelle (Contractor) and Caterpillar Inc. (Participant) was to develop the plasma arc lamp (PAL), infrared (IR) thermal processing technology 1.) to enhance surface coating performance by improving the interfacial bond strength between selected coatings and substrates; and 2.) to extend this technology base for transitioning of the arc lamp processing to the industrial Participant. Completion of the following three key technical tasks (described below) was necessary in order to accomplish this goal. *First*, thermophysical property data sets were successfully determined for composite coatings applied to 1010 steel substrates, with a more limited data set successfully measured for free-standing coatings. These data are necessary for the computer modeling simulations and parametric studies to: A.) simulate PAL IR processing, facilitating the development of the initial processing parameters; and B.) help develop a better understanding of the basic PAL IR fusing process fundamentals, including predicting the influence of melt pool stirring and heat transfer characteristics introduced during plasma arc lamp infrared (IR) processing; *Second*, a methodology and a set of procedures were successfully developed and the plasma arc lamp (PAL) power profiles were successfully mapped as a function of PAL power level for the ORNL PAL. The latter data also are necessary input for the computer model to accurately simulate PAL processing during process modeling simulations, and to facilitate a better understand of the fusing process fundamentals. *Third*, several computer modeling codes have been evaluated as to their capabilities and accuracy in being able to capture and simulate convective mixing that may occur during PAL thermal processing. The results from these evaluation efforts are summarized in this report. The intention of this project was to extend the technology base and provide for transitioning of the arc lamp processing to the industrial Participant.

BACKGROUND

Caterpillar, at its private expense, has been involved in developing hard-facing materials and coatings since the early 1970's. As part of their on-going efforts to optimize these coatings for use in abrasion resistant, mining applications, a prior CRADA agreement between Contractor and Participant (initiated under CRADA No. ORNL-02-0634) provided the initial proof-of-concept for using structurally integrated coatings (SIC) for improved wear (see Appendix 1, Figures A-1 through A-3). This current follow-on CRADA project evolved as a result of this prior, successfully completed CRADA. The primary goal of this current CRADA is to expand the PAL technology base and provide the first step in transitioning arc lamp processing to our industrial partner for implementation into industrial processes. In this collaboration, these partners successfully demonstrated that HDI processing of Caterpillar's functionally graded (FG) and thermally sprayed metal-matrix composites exhibited a 5X's improvement in abrasive wear performance, under heavily loaded wear conditions. These coatings are targeted to improve the wear performance for mining equipment and machines. The implication of these results is that by implementing PAL processing of these steel metal matrix composites and thermally sprayed coatings, equipment downtime could be reduced by up to 50%. Therefore, significant energy savings could be realized because of: 1.) the longer part life-cycles (resulting in reduced numbers of replacement parts being

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needed and processed); and 2.) Improved tool efficiencies and associated weight savings achieved via optimally placing coatings and hard secondary, composite particles.

Although it is well known that high amounts of convective stirring occur during high thermal flux processing (e.g. laser processing or electron beam processing), initial IR processing results seemed to indicate that HDI processing was a laminar process and produced no stirring effects. However, this concept was contradicted when tests were performed to produce functionally graded coatings and the ceramic additions were found to be dispersed within the coating. Consequently, another goal for the current CRADA was to investigate/determine whether convective mixing occurs during IR processing of these coatings.

STATEMENT OF OBJECTIVES

The primary objective of this Cooperative Research and Development Agreement (CRADA) between UT-Battelle (Contractor) and Caterpillar Inc. (Participant) was two-fold: First, to expand the PAL IR technology base and second, to facilitate transitioning arc lamp processing for implementation into industrial processes. Another important project objective was to develop cost effective processes that permit the implementation of advanced abrasion resistant materials. Further, based on prior wear testing results, the benefits anticipated by increasing wear component life were: 1.) a 50% reduction in scheduled downtime; 2.) increased energy efficiency achieved due to the: a.) Reduced need for processing as many replacement parts, and b.) Increased tool efficiency via abrasion resistant material selections; and 3.) Reduced operating costs. A better and more fundamental understanding of the PAL IR processing technology is necessary to achieve these goals. Therefore, the project approach/strategy was to 1.) Determine the thermophysical properties of these coatings, 2.) Establish the thermal energy input distribution/footprint of the PAL, and utilize these data to develop the modeling tools and a modeling methodology that would facilitate optimizing the infrared processing technology. This approach was anticipated to facilitate enhancing surface coating performance by improving the interfacial bond strength between selected coatings and substrates, and by producing fused coatings suitable for abrasion resistant, mining applications.

SCOPE OF WORK

The overall technical objectives of this CRADA were to: 1.) investigate/determine the melt pool stirring and heat transfer characteristics of the arc lamp infrared processing, utilizing ORNL's high density infrared (HDI)/transient liquid coating (TLC) process; and 2.) provide the data required for process simulation development including residual stress states of processed coatings. To achieve a better fundamental understanding of the PAL IR process technology that would facilitate transitioning the PAL IR processing technology to Caterpillar's Technical Center for further development of coated components, the following global tasks were identified and proposed to be conducted at ORNL.

- Task 1: Determine initial processing parameters.
- Task 2: Develop a better understanding of the fundamentals behind the fusing process.
- Task 3: Submit Final Report.

BENEFITS TO THE FUNDING DOE OFFICE'S MISSION

The key mission of the US DOE, Office of Energy Efficiency and Renewable Energy (EERE), Industrial Materials for the Future Program is to improve energy efficiency and energy utilization in energy intensive US industries. The overall goal of this project is focused at developing the energy

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efficient plasma arc lamp (PAL), and the infrared (IR) thermal processing technology to enhance surface coating performance by improving the interfacial bond strength between selected coatings and substrates. The intention of this agreement was to extend the technology base and provide for transitioning of the arc lamp processing to the industrial Participant. A major technical goal of this project was to advance the state of the technology and develop coating materials that can be applied to components that will yield the desired performance increase of 4-8x wear improvement over carburized and hardened steels. It was determined that this could be accomplished by determining the melt pool stirring and heat transfer characteristics associated with the arc lamp infrared processing, utilizing ORNL's HDI/TLC process, and provide data required for process simulation development including residual stress states of processed coatings.

TECHNICAL DISCUSSION OF WORK PERFORMED BY ALL PARTIES

The primary goal of this CRADA was to advance the state of the technology and develop coating materials that can be applied to components that will yield the desired performance increase of 4-8x wear improvement over carburized and hardened steels. The following objectives were identified to accomplish this goal: 1.) To develop the analytical data to allow for development of process modeling simulation and optimization; 2.) To define and map the thermal footprint of the PAL that would expedite the development of the initial PAL processing parameters for the materials of choice; 3.) to demonstrate a modeling methodology to facilitate the determination of the processing space related to melt pool stirring that can be used by Caterpillar to guide the optimization of the coating structure; and 4.) to determine the residual stress to allow for an understanding of the resulting component strengths. Unforeseen coating material shortages and extended delays in these material availabilities and shipments adversely impacted this CRADA, although the partners creatively sought and devised and fabricated alternate/surrogate coating materials to simulate the actual coating materials and their anticipated response to IR processing.

Thermophysical Properties Determinations

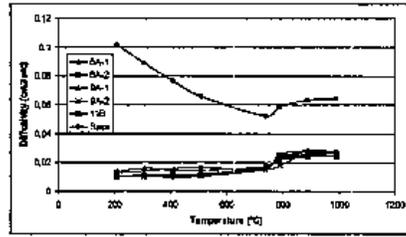
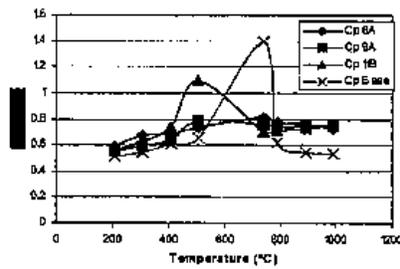
At the beginning of this project, Caterpillar provided ORNL with several different coating compositions (coatings were thermally sprayed onto steel substrates) for which we determined the thermophysical properties for the substrate (as a baseline) and each coating-substrate pair. One objective of this effort was to determine the thermophysical conductivity of the baseline/substrate steel and the coatings selected. An Anter Flashline 5000 laser flash system was used to determine the thermal diffusivity of the substrate and the coatings. A two layer method was used to calculate the thermal diffusivity of the coatings. The thermophysical properties results are summarized below in Figure 1. The thermal conductivities were calculated from the thermal diffusivity, specific heat, and density of the coatings. Using the specific heat (C_p), density (ρ) and thermal diffusivity (α), thermal conductivity was calculated using the following relationship:

$$k = \alpha \rho C_p \quad (1)$$

Figure 1(c) shows the thermal conductivity vs. temperature plots of the 5 coatings and the substrate (labeled BASE) material. The large increase for the substrate is attributed to the large C_p value available from the existing database. In reality the "hump" actually would be a straight line connecting the two nearest neighboring points. As can be seen in Figure 1(c), the thermal diffusivity of the substrate material drops to ~ 50% approximately at the eutectic transition temperature (~740C), followed by an increase as it passes through the Curie transition. The densities were determined for both the substrate and the coatings and are summarized in Table 1.

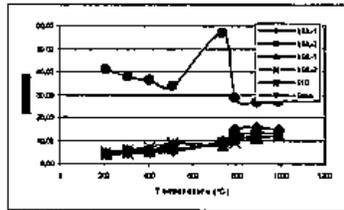
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(a)

(b)



(c)

Figure 1 Thermophysical properties determined for the substrate (labeled "BASE" in this figure), and for the composite coatings as thermally sprayed onto the substrate - a) Specific heat, (b) thermal diffusivity, and c.) conductivity.

Table 1 Density determined for the selected coatings and the baseline/substrate materials.

Sample	6A-1	6A-2	9A-1	9A-2	11B	Base
Thickness-Sub (cm)	0.192575	0.193125	0.19255	0.1897	0.1635	
Thickness-Coating (cm)	0.059875	0.061675	0.057525	0.0546	0.074625	
Coating Density (g/cm ³)	7.304	6.628	6.355	6.922	6.718	7.74

Analytic Data Determination of FREE STANDING Coatings for model input

In an effort to gain a more fundamental understanding of the heat flow/transfer across the coating - substrate interface, and eliminate uncertainties, Caterpillar attempted to produce and provide free standing coatings ("splats"). This latter effort was intended to provide independent TP property data for the substrate and the coatings, so that the thermophysical properties of the coating and substrate could be decoupled to more accurately model the heat flow across the coating-substrate interface providing more accurate thermal processing modeling simulations yielding more accurate predictions for the HDI/TLC processing parameters. These capabilities in conjunction with determining the presence of any convective stirring would significantly extend the technology base and facilitate the transitioning of the PAL processing to industrial practice.

Therefore, Caterpillar made several attempts at thermally spraying free-standing splat samples and provided several splats with the coating compositions of interest (see Figure 2). The sample sizes and dimension specifications for these samples for some of the analytic data (densities, thermal diffusivities and conductivities) are shown in Figure 3.

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The intention was to use these *free-standing steel coatings* to provide the best possible simulation results by determining and capturing the actual influence of the coating-substrate interface on predicting the heat transfer from the coating to the substrate, and hence, determining the HDI PAL processing parameters. Several attempts were made using both EDM (to cut out the rough dimensions) and special handling grinding equipment to carefully grind these analytic sample blanks to, or at least close to, the final dimensions required. Unfortunately, all of the samples provided were so badly distorted (dished like potato chips) during the "splat" spraying process that when these samples were finally flat and parallel enough to be suitable for obtaining the analytic data from them, these samples were much too thin to obtain accurate analytic measurements.

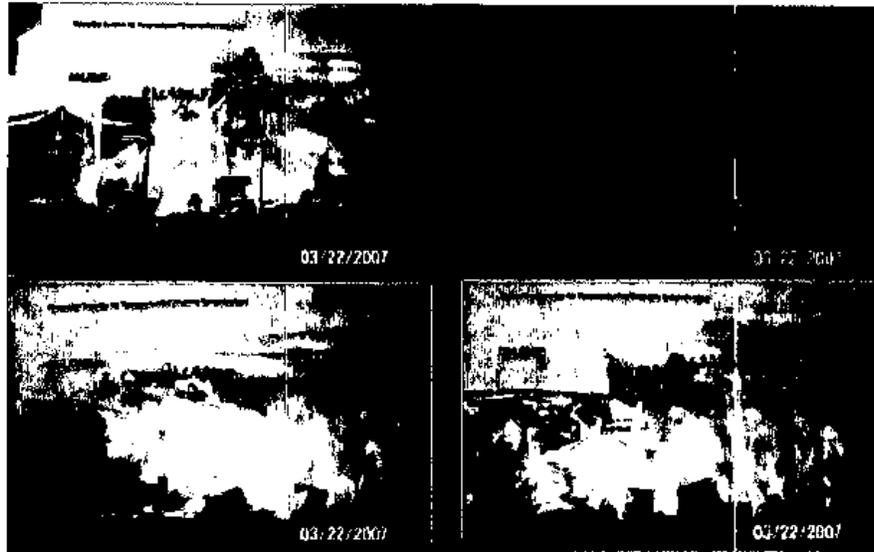


Figure 2 Macro-photographs of free-standing coating samples provided to ORNL to determine analytic data for modeling input and process simulations. Even after several attempts, suitable analytic samples could not be obtained from these badly distorted free-standing coating samples

In an attempt to determine which splat samples were the best candidates for the analytic test samples, these samples were sorted first to be sure that their lengths and widths were large enough to obtain the required analytic sample sizes. From these down-selected samples, further attempts were made to sort these samples to select those that appeared to have sufficient thicknesses to obtain suitable analytic samples. One of these thickness profile examples of the non-uniform thicknesses of these samples is shown in Figure 4. Unfortunately, only after grinding these samples (so that their surfaces were flat and parallel), was it discovered that only a couple of these samples met the analytic specimen dimension specifications. Consequently, only a few of these splat samples proved useful for "coating only" analytic properties to be determined from them, and so the specific heats for two of these splat samples are shown in Figure 5. The difficulties in obtaining uniform thicknesses TP property samples from thermally sprayed splat coatings prevented most of the analytic TP measurements from being determined for the free-standing coatings.

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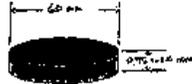
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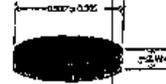


Figure 3 Schematics demonstrating specific dimension specifications for the (a) heat capacity and (b) thermal diffusivity analytic specimen samples.

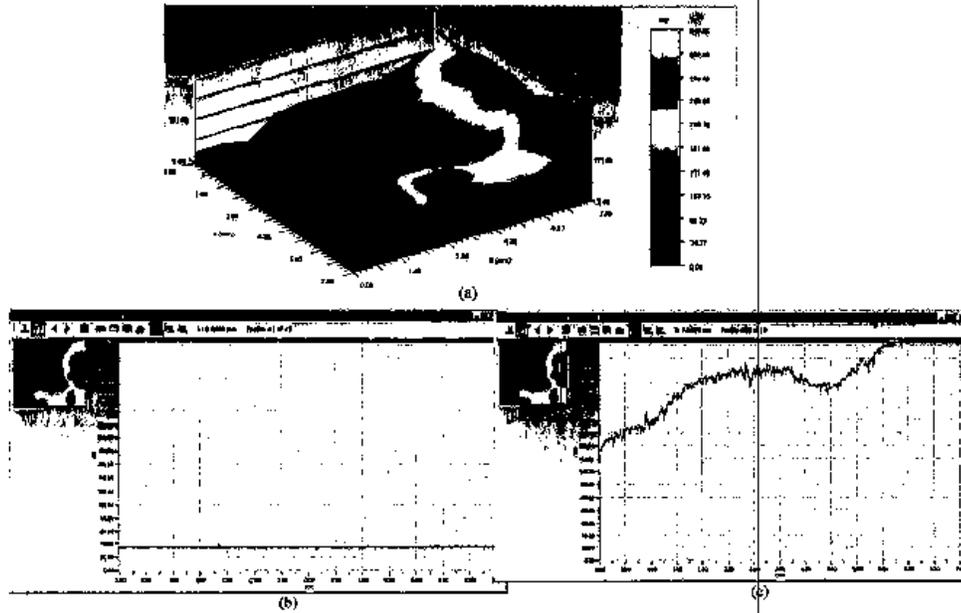


Figure 4 Example of laser profilometry scans determined for splat samples demonstrating non-uniform thicknesses (a) 3D thickness representation, (b) 2D thickness profile measured on test block, and (c) 2D thickness profile measured on splat sample number pac8292f 1a.

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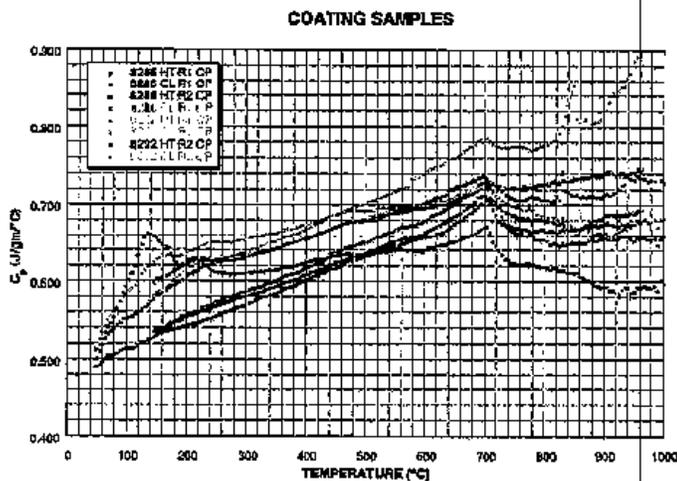


Figure 5 Specific Heat data plots determined for comparing stand-alone coating materials for two (2) of the coating compositions of interest.



Figure 6 In establishing melt temperature data in preparation for determining thermo-physical property data for free-standing coatings, a low melting eutectic phase resulted in this equipment being shut-down for months. This is an example of the slower heating segment exhibiting the exotherm for sample #292.

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Prior to determining analytical data on an experimental alloy composition, it is common practice to verify these alloy melting temperatures to facilitate the experiments on and the use of the actual analytic equipment. The platinum sample holder/tray was selected based on the melting temperatures anticipated for these coatings as well as the purported coating chemistries. In fact, Caterpillar provided an estimate of the melting temperatures of these coating compositions as predicted by ThermoCalc. While the coatings were being tested and evaluated at ORNL, one of these coatings suddenly reacted with the Pt tray and melted onto the DSC heat sensor below damaging it, and making this analytic equipment unavailable for further testing.

It has been postulated that this unanticipated melting event could have resulted from the introduction of Si into the coatings by one of two plausible events. First, a day earlier (unknown to ORNL), during some analytic SEM material evaluations on their coatings, Caterpillar inadvertently had discovered that silicon (Si) was present. Knowing that silicon was not part of their intended coating chemistry, they deduced that it must be present because it was a constituent in the binder that was used to thermal spray these coatings. Being unaware that Si may be able to be present in these coatings, the Pt pan was used to hold the free-standing coatings. Consequently, if Si were present, it could have chemically reacted with the Pt pan resulting in the formation of the low melting eutectic that is known to form when Pt combines with Si. An alternate scenario is that the equipment operator had used silicon based grit papers to smooth the surface of these coatings just prior to his thermophysical data collection. It therefore is also possible that some of this Si could have become embedded into the porous coating resulting in a low temperature eutectic during heating in the Pt pan. However, to conserve funding, we did not determine the definitive source of the Si, but we postulate that it came from the grinding papers, since the Si present in the water-based binder is less than 1%. However, regardless of what caused the Si melting reaction observed to occur, the unexpected low melting phase that formed dripped onto the sapphire crystal in the thermophysical testing instrument, rendering it out-of-service. Consequently, the heat capacity data were determined for only two (2) free standing coatings (see Figure 5). Figure 6 demonstrates an example of the heat flow (y-axis) versus temperature (x-axis) curves measured and recorded for sample 8292 (prior to equipment damage) during two sequential heating and cooling runs exhibiting the various endo- and exo-therms associated with the various liquidus and solidus for compounds within the composite coatings. The remainder of these free-standing coating samples could not be measured due to the equipment damage, and the lack of budget available to repair this equipment within the timeframe of this project.

Understanding the Fusing Process Fundamentals*Surrogate Coating Materials Development*

Due to unanticipated coating materials delays by the supplier, the CRADA partners agreed to proceed by identifying alternate/surrogate coating materials and conducting experiments using these coating material compositions to gain more insight into the convective stirring aspect of this CRADA. These experiments were conceived to provide an experimental basis for tuning the convective mixing modeling aspect of this project, and to help expedite the investigation and evaluation of whether convective mixing occurs in the coating during PAL processing. Another important aspect of modeling these convective currents and the development of these experiments is knowledge of the range of anticipated sizes and volume fractions of the composite particles contained in these coatings.

Nickel was selected as the primary binder material constituent for the surrogate coating. The

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ceramic addition was selected based on the following four constraints: 1.) it had to have a density similar to nickel; 2.) Ni must be able to wet the ceramic addition selected; 3.) the ceramic selected had to be easily identifiable in photomicrographs; and 4.) the reaction between the ceramic additions had to be limited or non-existent. Tantalum (Ta_2O_5) met these requirements and was used as the ceramic addition for the first series of tests. E.g., Ta_2O_5 has a density of 8.2 g/cm³ compared to the 8.9 g/cm³ of Nickel; and it is wet by Ni without reacting; Ta_2O_5 is easily distinguishable from Ni in optical micrographs. Therefore, in preparation for the convective mixing trials, the nickel and Ta_2O_5 material powders were procured and techniques were investigated to determine the best method to prepare these materials as coatings. Figure 7 shows an experimental design concept (a) and conceptually depicts the resulting dispersion of particles that might result after IR processing due to convective stirring currents.

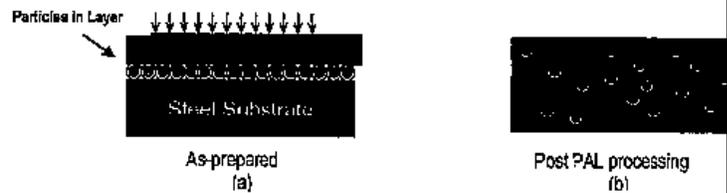


Figure 7 Schematics representing the concept to test whether convective stirring is a factor during PAL IR processing. (a) depicts a simplified version of this concept for the as-prepared samples, and (b) depicts a hypothetical case for dispersed particles due to convective mixing.

Coating preparation constraints are that: 1.) the overall coating should be no thicker than coatings previously applied using the HDI method (i.e., $\sim <750\mu\text{m}$ (0.030") in thickness); and that, 2.) there must be no mixing of the Ni and the Ta_2O_5 during the application process. Coating preparation and application methods were investigated and both the *dip method* and the *layered tape method* were determined to be plausible. The most economical Ta_2O_5 material powders were only readily available in a very fine powder size. Consequently, Ta_2O_5 powder was initially prepared as a dip coating onto much smaller pieces to establish whether these powders would work for the larger pieces that actually are needed for the HDI processing trials. Once initial feasibility studies demonstrated that these smaller samples could be successfully fabricated using the dip coat method, six larger (3-inch x3-inch x 0.5-inch thick) pieces were tape cast and pressed onto the steel substrates using a Carver press to warm press (80°C) the tapes onto the steel coupon. Initial plans for subsequent experimental trials were to create multi-layers using the tape caster and the Carver press to warm press these layers. However, project plans were altered before HDI processing trials were conducted to experimentally investigate, evaluate and ultimately validate modeling predictions for the extent of convective mixing that would occur during HDI processing. Due to budgetary constraints and Caterpillar's preference, the experimental portion of this CRADA was postponed to ensure sufficient funding would be available to do the modeling.

Modeling the PAL Fusing Process - Fundamentals

The main goal of this effort was first two-fold. ORNL first investigated the availability and applicability of various casting computer modeling codes (e.g., Flow3D, and ProCAST) to determine whether these could potentially provide a quick turn-around in evaluating convective mixing. ORNL determined that for FLOW 3D, although it is very accurate, there most likely would

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be compiler incompatibilities between this code and the convective subroutine needed. For ProCAST, although it would be fast to set-up, melting is not handled well, and the accuracy would most likely not be good enough to predict optimum processing parameters for fusing coatings. The conclusion reached was that these casting codes are not ready at this point for a quick application to model convection during IR processing. An assessment of the new version of the Telluride code revealed that it seems that it can be customized to investigate convective mixing; however, since this new version previously has not been tested at ORNL, it would require an extensive effort to describe the spatial and temporal variation in heat flux on the top surface of the sample during high density infrared processing using the plasma arc lamp. All the modules would: 1.) first, need to be tested to verify that they operate as advertised; then, 2.) the applicability of this code would need to be assessed and validated for its applicability for combined heat transfer/ fluid dynamics/ phase change issues; next, 3.) the user subroutines would need to be implemented for heat flux evolution (spatial and temporal); then, 4.) an appropriate model would need to be developed to capture the convective mixing aspect; and 5.) simulations would need to be run for static positions of the PAL to validate the computer model based on experimental validation (temperature and/or liquid pool depth); then, the level of fluid convection and ensuing mixing would need to be verified in the molten region; and finally, the experimental program data and computational program would need to be coordinated intimately to assist in an accurate model being developed.

This model would need to be first developed to evaluate convective experiments to evaluate the experiments designed to first control the deposition (spatially) of the composite particles to evaluate the surrogate (the baseline control sample) and then expanded and tested on Caterpillar's actual composite coatings. To accomplish this, user routines would need to be developed to predict the heat flow/flux evolution. These would be incorporated into the parent code. An example of a model that was developed on the baseline Telluride code for a baseline PAL processed test case provides some guidance for a case that was developed without considering the convective aspect during IR processing. Figure 8 depicts these results that were generated to predict optimum processing parameters for fusing alloy based coatings. In particular, this figure demonstrates that for the baseline case (without convection mixing) (a) excellent agreement has been achieved between the experimental and predicted thermal profiles both at the coating-substrate interface and on the back side of the substrate; and (b) that modeling has enabled the thermal flux/temperature to be predicted across a 3 inch wide sample as a function of HDI processing parameters. These latter results demonstrate that sample temperatures can be predicted based on processing parameters - i.e., as a function of HDI lamp speed, enabling HDI processing parameters to be predicted. Further, this effort has demonstrated that we can predict HDI processing parameters (lamp power and scan speed) to obtain UNIFORM thermal flux for a three inch (3") wide sample, eliminating over/under heating. (This provides the flexibility that would facilitate the use of HDI Processing feedback controls). A newly revised version of the Telluride modeling code (Truchas) was briefly evaluated. This latter code was determined to be capable of being used to implement the user subroutines necessary for describing the spatial and temporal variation of heat flux on/in the sample during HDI processing using the plasma arc lamp.

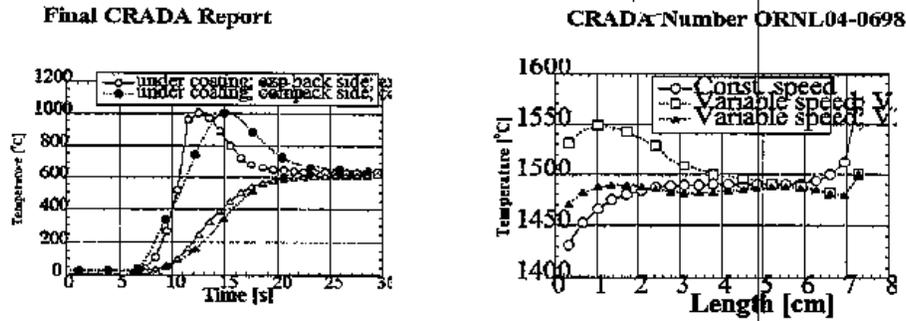


Figure 8 (a) Excellent agreement has been demonstrated between experimental and predicted thermal profiles both at the coating-substrate interface and on the back side of the substrate; and (b) modeling enables the thermal flux/temperature to be predicted across a 3 inch wide sample as a function of HDI processing parameters. These results demonstrate that sample temperatures can be predicted as a function of HDI lamp speed.

High Density Infrared Plasma Arc Lamp Mapping

IR plasma arc data have been collected on ORNL's current IR PAL to provide input data for the initial modeling effort. An example of these data is plotted for a defocused PAL case (see below in Figure 9).

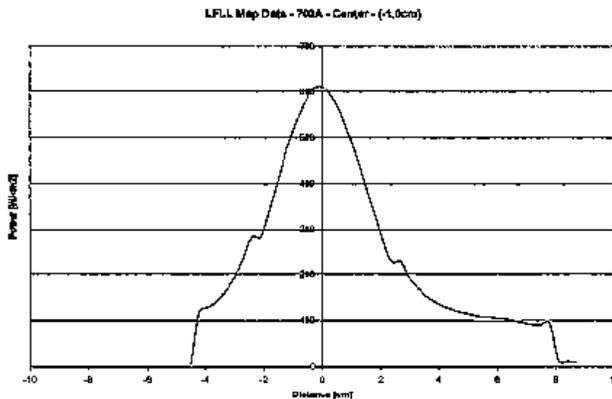


Figure 9 Example of power profile for the 350kW PAL using the long focal length reflector and operating at 700 amps at a distance of 5cm.

SUBJECT INVENTIONS

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(Jason: please fill in)

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COMMERCIALIZATION POSSIBILITIES

Although this work is still considered by Caterpillar to be in the research and development stage, it is considered worthy of future time and monetary investments. An infrared arc lamp has been purchased for further research and development at the Caterpillar Technical Center. The installation of the arc lamp will provide the ability for Caterpillar to optimize processing parameters to maximize product yield while minimizing overall process costs, requirements for industrial viability.

PLANS FOR FUTURE COLLABORATION

None anticipated.

CONCLUSIONS

Analytic thermophysical (TP) modeling input data were determined and provided for the coating-substrate composite case. Although equipment damage and material supply issues limited the determination of TP data for the stand-alone coatings, samples were successfully prepared, and procedures were developed and designed to run experiments to capture salient convective mixing details. Several current casting models were assessed for quick applicability and determined not to be ready nor accurate enough to predict process modeling parameters to handle convective mixing. Both the original and a newly revised version of the Telluride modeling code (Truchas) were evaluated. The latter code was determined to be capable of being used to implement the user subroutines necessary for describing the spatial and temporal variation of heat flux on/in the sample during HDI processing using the plasma arc lamp. A baseline HDI PAL process model was successfully set-up and developed as a precursor case (without incorporating convective mixing) and tested for an alloy coating.

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APPENDIX 1

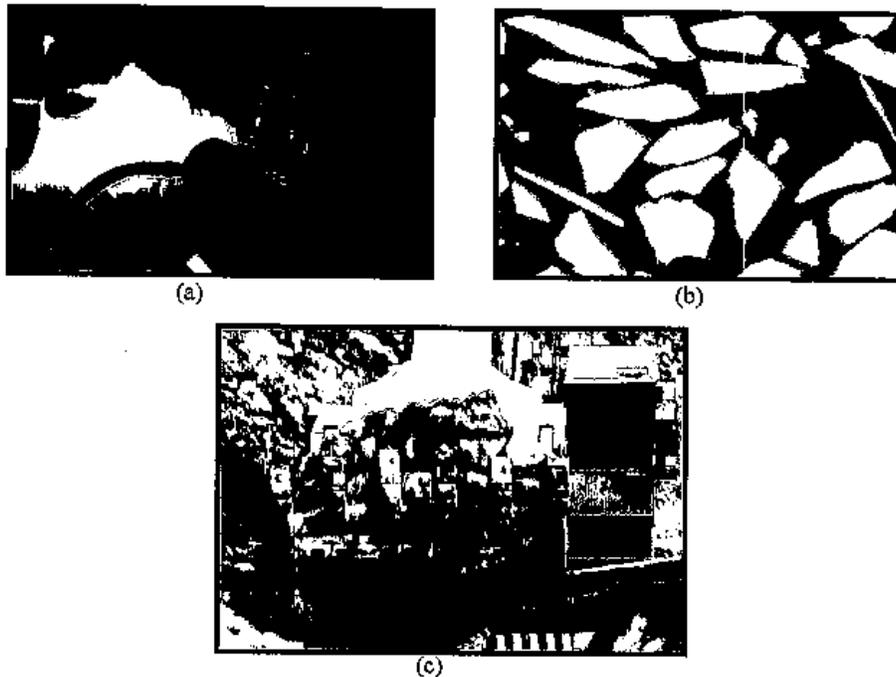


Figure A-1 Industrial laboratory wear testing on (a) thermally sprayed, and IR fused steel (b) matrix composite coatings applied on bearings lasted 5X's longer than current carburized and hardened steels. (c) Front-loader buckets are another possible high-wear application that would benefit from these coatings.



Figure A-2 Example of PAL infrared processing set-up for bonding functionally graded surface coated bearings that were successfully wear tested.

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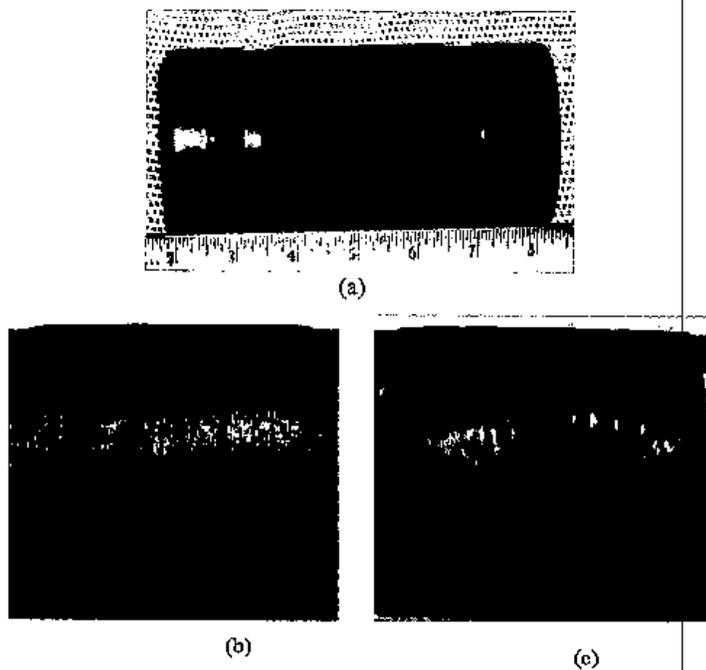


Figure A-3 (a) IR fused coatings on industrial tested bearings at Caterpillar demonstrated a 5X's improvement in wear life performance over the original carburized and hardened steel bearings. These graded coatings were fused on cylindrical substrates using the PAL (b) before high-stress, three-body wear testing and (c) after 7,000 wear testing cycles.

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Technology Transfer and Economic Development

Line and Program Managers (as appropriate)

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