INEL/CONF-97-00532
INEL/CONF-97-00532
CONF-9709200--

Flame Spraying of Polymers

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Abstract

Statistical design-of-experiment studies of the thermal spraying of polymer powders are presented. Studies of the subsonic combustion (i.e., Flame) process were conducted in order to determine the quality and economics of polyester and urethane coatings. Thermally sprayed polymer coatings are of interest to several industries for anticorrosion applications, including the chemical, automotive, and aircraft industries. In this study, the coating design has been optimized for a site-specific application using Taguchi-type fractional-factorial experiments. Optimized coating designs are presented for the two powder systems. A substantial range of thermal processing conditions and their effect on the resultant polymer coatings is presented. The coatings were characterized by optical metallography, hardness testing, tensile testing, and compositional analysis. Characterization of the coatings yielded the thickness, bond strength, Knoop microhardness, roughness, deposition efficiency, and porosity. Confirmation testing was accomplished to verify the coating designs.

A PLASTIC (1) MAY BE DEFINED as a synthetic organic polymer resin capable of being formed into load-bearing shapes and possessing properties that enable it to be used in the same manner as traditional materials. An engineering plastic is artificially made from a carbon base material (i.e., polymer), which may also contain various additives, thus making it a resin rather than a pure polymer. One very important classification scheme for polymers is based on the response of the polymer to heat and solvents. Thermosets cannot be melted once they are cured, whereas thermoplastics can be heated and melted. Thermoset plastics are formed by processing raw materials that contain monomers, fillers, and catalysts. The curing reaction is ini-

tiated at temperature and the material is converted into an insoluble and infusible three-dimensional network. The curing process introduces cross links between molecules, after which the materials can no longer flow. The properties of a thermoset are substantially affected by these cross links. Once reacted and cured, thermoset plastics cannot be reformed by any means except to break the covalent bonds and destroy the polymer. Examples of thermosets include epoxy, phenolic, polyester, and urethane resins.

By the turn of the century, applications for polymer coatings will abound in many industries for corrosion prevention, electrical insulation, wear resistance, and general coating applications (i.e., consumer markets). Plastics will represent a \$350 billion industry by the year 2000, with some 3.5 billion pounds of engineering and specialty plastics needed to meet the U.S. demand alone. The strong demand for plastics supports growth in business. Plastics used in the packaging industry is expected to double over the next 10 years, with comparable growth in the transportation, aircraft, aerospace, industrial, electronics, and consumer markets (construction, sporting goods, general usage, etc.). Significant new development activity that will accelerate in the years ahead include development of rapid, low-cost manufacturing methods and improved high-temperature polymer coatings.

Unsaturated polyesters are the most extensively used type of thermoset resin because of their low cost, ease of processing, and good performance characteristics. Unsaturated polyesters are generally combined with glass fibers, as well as fillers and additives, to alter the properties for specific applications. Thermoset polyesters are widely used in transportation, construction, electrical, and consumer products.

Polyurethane coatings are used in applications requiring abrasion resistance, skin flexibility, fast curing, good adhesion, and chemical resistance. Polyurethane resin systems are used for applications requiring superior toughness

and resistance to tear and abrasion, and cold-temperature impact and flexibility. Their major shortcoming is low resistance to steam, fuels, strong acids, and bases.

Environmental effects on plastics are unique and sometimes crucial because plastics are generally more sensitive to environmental factors than are other engineering materials. Ultraviolet light causes degradation through a breakdown of molecular weight. Plastics are also sensitive to chemicals. Alkalies and acids drastically affect them through hydrolysis of polymer chains.

In this study, the polymer coating designs have been optimized using Taguchi-type fractional-factorial experiments. Statistical design of experiments (SDE) has been rigorously developed for over 60 years by numerous scientists, including Sir Ronald Fisher (2). The main reason for designing an experiment statistically is to obtain unambiguous results at minimum cost. Thus, SDE methods are important tools to an engineer wishing to achieve the best possible design for a product. As such, SDE strategies are rapidly becoming invaluable resources in many industries (3). These experimental designs represent a plan for constructively changing the input parameters in order to determine their effect on the attributes of the product. A variety of SDE strategies are available (e.g., Box, Taguchi) to obtain statistical information within the selected test matrix. This methodology provides an exacting technique to optimize thermal spray processes and products.

Flame Spray Process

Combustion spraying was the first thermal spray process developed for applying coatings (4). The process uses combustible gases as a heat source to melt the coating materials, which are in powder, wire, or rod form. The commonly used combustion spray gases are oxygen, acetylene, propane, and hydrogen. The most common fuel for the Flame process is acetylene, since it can obtain the maximum flame temperature when burned with oxygen (3350°C). The materials are introduced into and melted in the combustion gases, and the subsequent molten particles/droplets are accelerated to the substrate by the gas flow. Flame temperatures and characteristics depend on the stoichiometric combustion temperatures of the oxy-fuel mixtures. The process is temperature limited and generally used for materials with low melting points.

The ratio or stoichiometry of fuel to oxygen is important in determining the final coating structure. Stoichiometric combustion for acetylene requires a 2.5:1 ratio of oxygen molecules to acetylene molecules ($2C_2H_2 + 5O_2 = 2H_2O + 4CO_2$). This combustion ratio produces a neutral flame (i.e., each oxygen and acetylene molecule is consumed during combustion). If the combustion ratio is lean, there are unconsumed oxygen molecules in the flame, creating an "oxidizing" flame environment. A fuel rich flame creates an oxygen

depleted "reducing" flame temperature. This is of concern in spraying metals, since it results in increased unmelted particles and porosity in the coating structure (5). For most oxidizer/fuel combinations, the maximum flame temperature typically occurs in a slightly fuel-rich composition. For oxygen/acetylene flames, this maximum flame temperature occurs at an oxygen/acetylene ratio of approximately 2.0, more fuel-rich than any of the conditions in this study. Accordingly, the flame with the highest adiabatic temperature used in this study was the most fuel-rich. The stoichiometric flame had a lower temperature, and the oxygen-rich flame had a lower temperature still. However, the greatest flame power (i.e., heat measure per unit time) was achieved at the stoichiometric ratio.

There is a definite safety advantage to using metal and polymer thermal spray coatings to ensure complete containment of hazardous waste, and the extended development of this technology has many future applications in industry. The use of polymer thermal spray coatings to refurbish waste containers protects the containers from corrosion and extends lifetime. The coatings research of this study is also applicable for many generic applications in U.S. industry and government relative to transportation (e.g., automotive) and infrastructure (e.g., bridges) applications. The material systems are commonly used for anticorrosion infrastructure applications. Smooth coatings with low porosity are desired for most applications.

Requirements for Coating Radioactive Waste Containers

The objective of this work was to develop a capability to solve corrosion problems in radioactive waste storage containers using thermal spray coatings (6). For this application, the coating system for the waste containers had to be compatible with the waste container materials and to withstand the normal operating environment experienced by the containers. These requirements were thermal (service temperature, thermal shock) and chemical (corrosion), which dictated the choice of the materials for the coatings. The coatings for the waste containers must withstand a low- to high-humidity air atmosphere for the application. The coatings also must maintain integrity against temperature cycling from freezing to a maximum temperature of 100°F. The Flame subsonic combustion spray process was chosen because it can produce high-purity, low-porosity, polymer coatings.

The role of a polymer for this application is to act as a barrier (i.e., sealant) coating for a metal coating (e.g., zinc, aluminum) which is applied to mitigate corrosion of metal surfaces. The original barrier action of the polymer coating layer and secondary sacrificial action of the metal layer afford separate portions of the protective ability of the total coating system. The adhesion between the polymer and the

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metal coating is critical for maximum durability of the polymer coat.

Experimental Procedure

A Metco 6P combustion spray system and commercially available polymer powders were used for the experiments. Classical experiments were first conducted (7) at the equipment manufacturers recommended process parameters. Once reasonable coatings were obtained, a Taguchi-type (8) L9 fractional-factorial experiment was conducted to determine the parameter space for optimization of the coatings. This statistical analysis was accomplished with Sadie (9) and Design-Expert (10) software. The designs evaluated the effect of the processing variables on the quantitatively measured responses. The design-of-experiment approach for the thermal spray process is ideal because it displays the range of measured coating characteristics attainable, and it statistically delineates the impact of each process parameter on the measured coating characteristics across all combinations of the other parameters. This information is extremely useful in establishing coating specifications.

Table 1 illustrates the SDE experiments conducted for the polyester (experiments P01-P09) and urethane (experiments U01-U09). The experiments were primarily conducted to investigate the stoichiometry at three levels (i.e. stoichiometric = 2.5, fuel rich = 2.125, fuel lean = 2.875). The total flow, spray distance, and powder feedrate utilized the ranges suggested by the process equipment manufacturer. Based on the results of the characterization and statistical results, confirmation runs were made to verify the optimum process parameters for the two powder systems (i.e. P0, U0).

The following process parameters were used for the SDE experiments: oxygen to acetylene flow ratio, total flow, spray distance, and powder feedrate. The working gases were acetylene (C_2H_2) and oxygen. The axial powder injection was external to the torch. An x-y servo-manipulator ensured

the standoff distance and repeatability in the experiments. The traverse x-motion rate was 15 inches per second. A y-step of 0.125 inches was used. Sixteen traverses per pass, four passes per sample, were used to fabricate the coatings. The powder was sprayed onto light carbon steel substrates ($4 \times 6 \times 0.125$ inches) cooled by air jets on the back side. Each coupon was grit blasted with No. 36 alumina grit prior to spraying to obtain a surface roughness (i.e., amplitude) of 3 mils.

Coating Characterization

The coatings were characterized and evaluated by a number of techniques. These included hardness tests, optical metallography, image analysis, roughness, composition, deposition efficiency, and tensile testing. Characterization of the coatings yielded the thickness, bond strength, Knoop microhardness (25-gram load), roughness amplitude, porosity, and deposition efficiency. Attributes were measured on metallographically prepared cross-sections of each coating. Results of the thickness, porosity, microhardness, bond strength, and roughness measurements are listed in Tables 2 and 3 for the polyester and urethane coatings for the Taguchi experiments and the confirmation experiments (i.e., P0, U0).

Coating thickness was measured by optical metallographic observation at 200 $\times$ magnification. Average thicknesses from 10 measurements of the coatings are presented. The polymer deposits, reflecting influences of spraying parameters, ranged from 4.4 to 29 mils for the polyester coatings, and 4.2 to 33 mils for the urethane coatings. Thickness buildup is calculated by dividing the average thickness by the powder feedrate. The thickness buildup ranged from 1.47 to 4.83 mils-hr/lb for the polyester coatings, and 1.5 to 5.5 mils-hr/lb for the urethane coatings.

Deposition efficiency for the materials was determined with conventional techniques by measuring the amount of

Table 1. Polymer thermal spray experiments 01-09, P0, U0.

Exp. No.	Flow Ratio	Total flow (scfh)	Spray Dist. (inches)	Feedrate (lbs./hr)
01	2.125	75.0	3.0	2.0
02	2.125	100.0	4.5	4.0
03	2.125	125.0	6.0	6.0
04	2.500	75.0	4.5	6.0
05	2.500	100.0	6.0	2.0
06	2.500	125.0	3.0	4.0
07	2.875	75.0	6.0	4.0
08	2.875	100.0	3.0	6.0
09	2.875	125.0	4.5	2.0
P0	2.75	96.3	3.5	3.8
U0	2.875	75.0	4.6	5.4

Table 2. Polyester coating characterization results.

Coating No.	Thickness (mils/FR)	Porosity %	Micro-hardness HK25	Bond Strength psia	Roughness mils
P01	4.6 (2.3)	5.1	16.5	203	3.8
P02	10.0 (2.5)	1.6	15.0	243	3.6
P03	8.8 (1.47)	3.3	17.7	162	5.2
P04	15.0 (2.5)	2.6	16.2	243	6.4
P05	8.4 (4.2)	2.4	17.6	162	3.7
P06	7.2 (1.85)	2.0	17.1	203	4.0
P07	9.0 (2.25)	2.7	15.6	243	3.7
P08	29.0 (4.83)	3.6	14.9	284	5.5
P09	4.4 (2.2)	1.2	16.7	162	4.9
P0	14.0 (3.7)	7.6	12.3	243	6.0

Table 3. Urethane coating characterization results.

Coating No.	Thickness mils (mils/FR)	Porosity %	Micro- hardness HK25	Bond Strength psia	Roughness mils
U01	4.2 (2.1)	1.9	18.5	162	4.3
U02	12.0 (3.0)	1.6	18.0	162	4.7
U03	6.3 (2.72)	1.1	15.9	81	4.7
U04	33.0 (5.5)	3.8	14.2	325	4.3
U05	5.0 (2.5)	1.1	22.4	81	4.9
U06	10.0 (2.5)	4.4	17.0	162	5.3
U07	17.0 (4.25)	0.7	17.3	203	5.3
U08	23.0 (3.83)	2.0	15.8	243	5.6
U09	3.0 (1.5)	1.1	23.2	81	4.3
U0	27.0 (5.0)	2.6	16.4	325	4.7

sprayed polymer deposited for an allotted time for selected experiments. The deposition efficiencies for the optimum process parameters were 43% for the polyester powder (run P0) and 57% for the urethane powder (run U0). Note that the DE experiments were conducted on 4-in. by 6-in. coupons cooled by air on the back. It is estimated that the reported deposition efficiencies could be 15 to 20% higher if sprayed on larger substrates (e.g., 12 in. by 12 in.).

Porosity for the coatings was determined using image analysis techniques. An NIH image analysis program, a Power Macintosh personal computer, and a Nikon Epiphoto metallograph were used for the metallurgical mounts. The metallographic image is captured in gray scale using the video monitor in the computer, which is connected to the camera in the metallograph. The image is then imported to the NIH image analysis program where the image is "seen as" 320 × 240 pixels (video squares), each with a level of gray between 0 and 255 (0 = white, 255 = black). An area of interest is outlined. The gray scale image is converted to a binary image (black and white) by manually setting the threshold gray value. All pixels with a gray value less than the threshold value are set to 0 and all pixels with a gray value larger than the threshold are set to 255. This process makes the porosity appear black and the coating white. The computer program counts the number of black pixels and the total number of pixels in the region of interest. The percent porosity is then calculated by dividing the number of black pixels by the total number of pixels in the area of interest. Ten regions were evaluated to determine the percent porosity in the polymer coatings, which ranged from 1.2 to 5.1% for the polyester coatings and 0.7 to 4.4% for the urethane coatings.

Owing to the soft nature of the polymer coatings, microhardness measurements were performed using the Knoop hardness scale with a 25-gram load on the specimens. By using the Knoop indenter, the test was performed within the boundaries of ASTM E384-89 (i.e., keeping impressions within two and one half indentation diameters from any

edges). As illustrated in Tables 1 and 2, the microhardness measurements ranged from 14.9 to 17.7 for the polyester coatings and 14.2 to 23.2 for the urethane coatings.

Bond strength studies used a portable adhesion tester (PATTI, pneumatic adhesion tensile testing instrument) following the test procedure described by ASTM standard D4541. The polymer powder was sprayed onto light carbon steel substrates (4 × 6 × 0.125 inches) grit blasted to a surface roughness of 3 mils. An adhesive was then used to bond a pull stub to the coatings. This method is reported to generate quantitative tensile strength data with a 2% or better accuracy. The bond strength ranged from 162 to 284 psia for the polyester coatings and 81 to 325 for the urethane coatings.

Surface roughness was measured with a dial depth micrometer. The coating amplitudes (i.e., peak to valley readings) were nominally 4.5 mils for the polyester coatings and 4.8 mils for the urethane coatings.

Certain deposited coatings and starting materials were analyzed for composition using IR transmission analysis (FT-IR) and inductively coupled plasma atomic emission analysis (ICP-AES). A sample was prepared for IR transmission analysis by chipping small amounts of the coating and grinding this into a KBr pellet. This sample was then analyzed using a Bomem MB 102 FT-IR spectrometer. For atomic emission spectrometry, the urethane coating was abraded off using a razor blade. The coating was carefully weighed and transferred into a 200-ml beaker. An acid decomposition procedure using concentrated sulfuric acid, nitric acid, and 30% hydrogen peroxide was used to get the urethane and filler completely into solution. After the digestions were complete, the remaining acid in the beakers was transferred to 100-ml volumetric flasks. Hydrofluoric acid was added to dissolve the titania and silica. The instrument used for analysis was a calibrated Thermo Jarrell Ash, model 61E, inductively coupled plasma atomic emission spectrometer.

Metallographic examination and image analysis revealed differences in the microstructures of the polymer coatings. The quality of all the coatings produced in this study can be graded from good to very good. The photomicrographs indicate very dense coatings, with homogeneously dispersed porosity. All the microstructures exhibited a totally melted coating matrix (i.e., no unmelted particles). Surface examinations revealed splat-like formations, indicating the predominance of molten conditions upon deposition. The lamellar morphologies are very similar, perhaps with a tendency for slightly rounder splats in the urethane coatings. Many of the polymer coatings evidenced layering, possibly due to the method in which the coatings were fabricated with 4 passes.

Sample P0 (Figure 1 at 100×) reflects the optimized design for the polyester system. Relative to the other polyester coatings, this coating had the highest porosity, the lowest hardness, intermediate deposition buildup, and very high bond strength and roughness. No macroscopic cracking (seg-

Figure 1. Photomicrograph of polyester coating UO (100x).

mented cracking appearing either parallel or normal to the substrate) was apparent in the body of the coating or at the interface. Three or four distinct polymer layers are evident in the microstructure. The porosity ranged from 5 to 15 micron size pores and was dispersed homogeneously throughout the coatings.

Sample U0 reflects the optimized design for the urethane system. Relative to the other urethane coatings, this coating possessed low porosity, intermediate hardness and roughness, and very high bond strength and deposition buildup. Figure 2 illustrates the coating (100x). No macroscopic cracking (segmented cracking appearing either parallel or normal to the substrate) was apparent in the body of the coating or at the interface. Random pores from 3 to 20 microns were discernable at a magnification of 200x. Three or four distinct polymer layers are evident in the microstructure.

Figure 2. Photomicrograph of urethane coating UO (100x).

Coating Composition. The urethane-based coatings developed in these experiments were analyzed by FT-IR and ICP-AES. The urethane-based starting material (Coating Powder UFW551S9 available from Herberts-O'Brien Inc., Houston) was described as having the following inorganic constituents: 15–40% titanium dioxide, 1–5% amorphous silica, and 1–5% aluminum hydroxide. Table 4 shows the percentage of titanium and silicon atoms in the deposited urethane-based coatings and the urethane-based starting material, as measured by ICP-AES.

These same urethane-based samples were analyzed by FT-IR. This analysis examined the level of the urethane functionality response (NHCO, 1530 cm⁻¹). Based on the intensity of the 1530 cm⁻¹ urethane band relative to the aromatic band at 1500 cm⁻¹, the urethane level in the samples is as follows:

Powder > U08 = U04 = U05 = U02 = U01 > U06 > U03 = U09

In addition, this analysis indicates the level of urethane functionality in the powder is approximately twice the level in samples U02 and U05, and essentially no urethane functionality is evident in U03 and U09. Also, the C=O region (1740–1650 cm⁻¹) is broader in the flame-deposited samples, indicating a probable increase in urea or other C=O formation resulting from the deposition process. All flame-deposited samples have higher levels of the inorganic materials and lower levels of urethane compared to the starting material. This result indicates that the flame deposition process both decreases the relative amount of the organic component and alters the structure of the organic material deposited, probably by oxygen incorporation.

Analysis of the composition results by the same statistical method used to examine the porosity, hardness, and thickness responses suggests that increases in the relative amount of the inorganic component correlates directly with total gas flow and inversely with powder feed rate. In addition, samples made with a lower total flow tend to retain more of the

Table 4. Percentage of Ti and Si in urethane coatings.

Coating Number	Ti weight %	Si weight %
U01	28.1	1.5
U02	29.4	1.6
U03	29.3	1.6
U04	24.2	1.3
U05	32.7	1.9
U06	29.8	1.7
U07	26.7	1.4
U08	27.9	1.5
U09	36.1	2.0
Powder	22.5	1.0

urethane functionality. An explanation of this relationship is difficult because as the total gas flow of the flame increases, so do the flame power and the carrier gas velocity. This means that dynamic, thermal, and chemical interactions of the gases and the particles in the deposition system could all contribute to the changes observed in the deposited coatings. Regardless of the mechanism, in this flame deposition system a higher gas/powder ratio will tend to form a coating that has a higher percentage of inorganic materials, and a higher oxidation of the organic materials that are deposited.

Statistical Design of Experiments. In most experimental programs, a natural evolution occurs. In the early stages, classical screening experiments are conducted to identify potentially influential process parameters. In the latter stages, the experimenter knows which parameters influence the responses, but he or she requires a more quantitative understanding of the main effects, the possible interactions between the effects, and experimental error. In this stage, fractional-factorial and factorial designs are used. In this study, the polymer coating designs have been optimized using this sequential methodology. Once classical experiments were conducted (6) to ascertain important process parameters, the aforementioned Taguchi-type L9 fractional-factorial experiment was conducted to optimize the coating. Once the experiments were conducted, the data were analyzed with the Design-Expert and Sadie statistical programs. Table 5 presents the general design for a fractional factorial L9 experiment. Four factors (variables) are studied, allowing three levels for each factor. A full factorial experiment (i.e., 3^4 , 81 experiments) would involve running all combinations of conditions of the independent variables. For this case, the use of nine experiments, a $1/9$ fractional experiment, allows complete evaluation of all four factors (i.e., A = stoichiometry, B = total flow, C = spray distance, D = feedrate) and their quadratics (e.g., A²). However, all interactions (i.e., AB, AC, BC, ABC) between the process variables are aliased.

Results of the Statistical Design of Experiments.

Table 5. Factor level combination for L9 fractional factorial experiment.

	Factors			
	A	B	C	D
Test 1 Levels	-1	-1	-1	-1
Test 2 Levels	-1	0	0	0
Test 3 Levels	-1	+1	+1	+1
Test 4 Levels	0	-1	0	+1
Test 5 Levels	0	0	+1	-1
Test 6 Levels	0	+1	-1	0
Test 7 Levels	+1	-1	+1	0
Test 8 Levels	+1	0	-1	+1
Test 9 Levels	+1	+1	0	-1

Effects analysis was first conducted for the responses listed in Tables 2 and 3. A typical example of the analysis is illustrated in Figure 3, showing the effect of the four process parameters on the polyester porosity. As illustrated, the quadratic design is required to predict the minimum porosity for all four process parameters for the polyester porosity. The quadratic model was then used for all of the remaining coating attributes for both the polyester and urethane systems, as dictated by the effects analysis.

Once the effects analysis was completed, the ANOVA (analysis of variance) calculations were then conducted for each response for each material. The ANOVA calculations guide further experimentation by indicating which parameters are the most influential on coating attributes. In general for all of the responses, the comparison of the treatment variance with the error variance was good, the probability that the model terms are null was low, and the coefficient of variation (CV) was low, indicating that the error was relatively small. This analysis yielded the regression equations for each attribute. Equation 1 illustrates the quadratic regression equation for the polyester porosity (i.e., A, B, C, and D use the coded variables = -1 to +1):

$$\text{Polyester porosity} = 0.6 - 0.42*A - 0.65*B - 0.38*C + 0.13*D + 0.58*A^2 + 0.28*B^2 + 1.38*C^2 + 0.93*D^2 \quad (\text{Eq. 1})$$

The statistical evaluation yielded very low model probability values, indicating the validity of Equation 1. The residual analysis indicated low residuals for the calculated data. The cube plot of the predicted (Eq. 1) values for the polyester porosity was constructed. Figure 4 illustrates the polyester porosity as a function of standoff distance and flame chemistry at constant minimum values of total flow and feedrate. As illustrated, the minimum polyester porosity is obtained at a 4.8-inch spray distance, a 2.62 oxygen-to-acetylene flow ratio, a 3.6-lbs/hr powder feedrate, and a total flow of 125 scfh.

Using the Sadie and Design-Expert programs, this statis-

Figure 3. Effects analysis of polyester coating porosity.

Figure 4. Cube plot of polyester porosity.

tical methodology was conducted for all of the coating attributes. The rho percent calculation indicates the influence of a factor or parameter on the measured response, with a larger number indicating more influence. This information is critical in developing new coating specifications. Tables 6 and 7 illustrate the effects analysis results for all of the coating attributes in order of desired priority.

Analysis was then conducted to determine the optimum parameters for the coating designs. This methodology involved numerical optimization to search for a combination of factor levels that simultaneously satisfies the requirements placed on each of the responses. With multiple responses, one must find regions where the process parameters simulta-

Table 6. Results of the polyester effects analysis.

Processing factor:	O2/Acet Ratio	Total Flow	Spray Dist.	Feedrate
Desired attribute	rho%	rho%	rho%	rho%
1 Low porosity	15.7	24.6	42.9	16.9
2 High bond strength	9.3	37.1	16.6	37.1
3 Low roughness	6.2	4.0	11.5	78.3
4 High hardness	26.6	34.7	19.5	19.2
5 High tk buildup	16.9	67.0	5.5	10.6

Table 7. Results of the urethane effects analysis.

Processing factor:	O2/Acet Ratio	Total Flow	Spray Dist.	Feedrate
Desired attribute	rho%	rho%	rho%	rho%
1 Low porosity	44.3	5.5	38.0	12.2
2 High bond strength	8.8	41.3	16.8	33.1
3 Low Ti content	3.7	48.4	2.9	45.0
4 Low roughness	19.6	15.4	36.3	28.7
5 High hardness	3.7	11.7	5.5	79.1
6 High tk buildup	10.3	36.4	3.5	49.8
7 Low Si content	2.2	52.7	2.2	42.9

neously meet the critical properties. By superimposing response contours one can numerically search for a compromise optimum for the coatings. The prioritized attributes illustrated in Tables 6 and 7 were used to weight the analysis.

The optimum derived process parameters for the polyester coating design are an oxygen-to-acetylene ratio of 2.75, a total flow of 96.3 scfh, a spray distance of 3.5 in., and a powder feedrate of 4.0 lbs./hr. Using these process parameters, the predicted polyester coating attributes would be porosity 1.6%, Knoop hardness 14.9, roughness 3.9 mils, bond strength 273 psia, and a thickness buildup of 3.8 mils-hr/lb. The actual polyester coating attributes illustrated in Table 2 from the characterization studies are porosity 7.6%, Knoop hardness 12.3, roughness 6.0 mils, bond strength 243 psia, and a thickness buildup of 3.7 mils-hr/lb (i.e., DE = 43%). It is obvious that this confirmation coating is not optimized for this particular application, and will not be used. Follow-on studies are planned to investigate whether this discrepancy is due to an equipment or material anomaly.

The optimum derived process parameters (i.e., U0) for the urethane coating design are an oxygen-to-acetylene ratio of 2.875, total flow of 75 scfh, spray distance of 4.6 in., and a powder feedrate of 6.0 lbs./hr. Using these process parameters, the predicted urethane coating attributes would be: porosity 1.9%, Knoop hardness 15.1, roughness 4.5 mils, titanium content 25.6, silicon content 1.3, bond strength 309 psia, and a thickness buildup of 5.2 mils-hr/lb, DE of 43%). The actual urethane coating attributes illustrated in Table 3 from the characterization studies are porosity 2.6%, Knoop hardness 16.4, roughness 4.7 mils, bond strength 325 psia, and a thickness buildup of 5.0 mils-hr/lb (i.e., DE = 57%).

The statistical methodology employed in this study is significant in that it finalizes coating design studies by optimizing the most important process or coating attributes and the process parameters that affect these attributes. The attributes may differ for the same material in different applications, and the baseline data generated in this study can be used to develop specific confirmation runs that approach other desired application attributes.

Summary and Conclusions

An experimental study of the Flame spraying of polymer coatings has been presented. Taguchi-type statistical experiments were conducted. The objective of the work was to develop a capability to solve corrosion problems in hazardous waste storage containers using polymer thermal spray coatings. The role of the polymer coating is to act as a barrier coating (i.e., sealant) for a cathodic coating. A Metco Flame combustion spray system and commercially available polymer powder were used for the experiments. Samples were produced for urethane and polyester powder systems.

The coatings were characterized and evaluated by a number of techniques, including Knoop microhardness tests, optical metallography, image analysis, and bond strength. All the coatings produced in this study may be graded from good to very good in quality. In general, the coatings had reasonable bond strength, low roughness, and good deposition efficiency. The photomicrographs indicate very dense coatings with low homogeneously dispersed porosity. All the microstructures exhibited a totally melted coating matrix (no unmelted particles).

A statistical effects analysis was conducted for all the coating attributes. Quadratic designs were required to predict the characteristics of the attributes. ANOVA calculations were then conducted for each response for each material. This analysis yielded the regression equations for each attribute. Analysis was then conducted to determine the optimum parameters for the two coatings. This methodology involved numerical optimization. The studies led to final optimum process parameters that were in confirmation runs to establish the coating design for waste applications at the Idaho National Engineering and Environmental Laboratory. The optimum derived process parameters for the polyester coating design are an oxygen-to-acetylene ratio of 2.75, total flow of 96.3 scfh, spray distance of 3 in., and a powder feedrate of 4.0 lbs./hr. Using these process parameters, the actual polyester coating attributes did not correlate with what was predicted by the statistical evaluation. Thus, the confirmation coating is not yet optimized for this particular application, and will not be used. Follow-on studies are planned to investigate whether this discrepancy is due to an equipment or material anomaly. The optimum derived process parameters (i.e., U0) for the urethane coating design are an oxygen-to-acetylene ratio of 2.875, total flow of 75 scfh, spray distance of 4.6 in., and a powder feedrate of 6.0 lbs./hr. Using these process parameters, the actual urethane coating attributes correlated well with those predicted by the statistical analysis, and yielded the best urethane coating of the study.

Acknowledgments

B. Dittmar and A. P. Bohlke (3M Company) completed the FT-IR and ICP-AES analysis for this study. The work described in this paper was supported by the U.S. Department of Energy, Assistant Secretary for Defense, under DOE Contract No. DE-AC07-94ID13223.

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