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Technical Note

Modern Industrial Approach to Qualification of Spray Booths and Laboratory Personnel

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Reproducibility, repeatability, and reliability are key words in current development of thermal spraying. This article addresses examples of means and procedures, from an industrial point of view, for use during introduction (or recertification) of new spray equipment and new lab personnel. Modern statistical tools, such as two-sample *t*-tests, ANOVA-analyses and run charts are being applied and defined rules for acceptance/rejection of new machines and personnel are being discussed. It needs to be pointed out that the article gives an industrial approach, rather than a scientific approach, for securing the quality in a modern spray shop over time.

Keywords coatings for gas turbine components, diagnostics and control, properties of coatings

1. Introduction

As thermal spraying is growing to become a more mature process, new sophisticated approaches are being adapted to the process (Ref 1-5).

What was outlined a decade ago by Pejryd et al. (Ref 6) is being realized slowly. Repeatability, reproducibility, and reliability are becoming key words for thermal spray suppliers.

The study done at Metcut in the early part of the 1990s by Sauer (Ref 7) clearly indicated a need for standardization and an approach leading toward an upgradation of the world's laboratories through extensive laboratory round robins and training programs. Volvo Aero (Ref 8, 9) added various experiences about different epoxies, procedures during tensile and hardness testing, thickness measurements, and microstructure evaluation techniques. In 1997, a subcommittee of EACMT (European Airline Committee for Materials Technology) started to work with thermal spray coatings, and some standardization recommendations were published in 2002 and 2003 (Ref 10, 11).

Altogether, thermal spraying is now slowly approaching a level where standard statistics can be applied to calculate capabilities, to improve reliability. and even to go as far as reducing test frequencies.

A software (such as the PKR (Ref 12), developed by Volvo Aero) is a necessity for any statistical treatment of data. This software must be associated with a database where all data (production and lab results) are stored for every spray occasion. Through this software, current capability can always be calculated and displayed for each part. Furthermore, reduced test frequencies can be correlated to current capabilities (PpK) and constantly be displayed with run charts and control levels. Finally, all out of controls (outside control levels ±3 standard deviations (σ)) must be displayed and acted upon, including documentation requirements.

In recent years, Volvo Aero has been installing two new equipments in the workshop. Being in the conservative aero engine field, where safety is of the highest priority, there is a necessity to prove no difference in coating properties between the old and new machines (using same spray parameter), or otherwise, a thorough substantiation of the change would be needed at a cost of ~$25000 per coating and parameter.

The previous equipment was 20-25 years old, and many things have been changed or upgraded, process wise, such as

- Rectifiers
- Robots
- Mass flow controllers
- Air cooling/ventilation
- General environmental requirements
- Laboratory procedures and their requirements

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2. Equipment and Procedures

2.1 Laboratory Procedures

Tensile strength tests were performed per ASTM C633 (Ref 13), using FM1000 epoxy (Ref 14). One reported data point is the average of three individual measurements. Macro hardness tests were performed per ASTM E18 (Ref 15), and each reported data point is an average of 15 and 10 indentations for HR15Y and HR15N, respectively.

2.2 Shop Equipment

The new equipments, Sulzer Metco Multicoat (F4 gun) were compared to the 20-year-old equipments, Sulzer Metco A3000S (F4 gun).

2.3 PKR Database

A computer-based process control system developed by Volvo Aero Corporation was used (Ref 12). All spray data (each spray occasion) since 1997 has been stored in this database, and any data of any group of parts, parameters, or powder lots etc. can be charted instantly.

3. Approach

The approach was first to verify, through internal (and external) round robins (correlation tests) that the lab procedures were accurate and that all the laboratory personnel could be involved in the qualification.

Second, the properties measured must not show a difference to 95% confidence level, when compared.

Guidelines from Six Sigma literature and courses (Ref 16) combined with internal experience tests as described below were set for variables (attributes were handled differently or image analysis was applied) for laboratory and spray equipment, respectively.

3.1 Test of Laboratory Data

Round robins (external and internal) have been performed for many years (branch requirements), but typically in the aero engine field, the only requirement is that all the operators need to be within the specification, which is NOT a harsh enough requirement in this case. Volvo Aero needed to show that the operators evaluate the same. Further, round robin data are also used for determination whether the correct measurement method is applied. Thus, statistical rules were developed/applied to satisfy these needs.

Rules (tools) 1 and 2 below were applied for evaluation of correlation of laboratory operators, whereas Rule 3 is applied to judge the measurement method itself.

3.1.1 Rule 1: 2 Sigma Test. Each operator's average shall be within $\pm 2\sigma$ of all individual measurements (Ref 17).

3.1.2 Rule 2: ANOVA or Two-Sample *t*-Test. The comparisons are made through either ANOVA or two-sample *t*-test (Ref 16), depending on the number of laboratory technicians involved. $p < 0.05$, i.e., 95% significance for being different, was used as failure or indication of an operator-dependent result.

As always in industrial environment, data are never perfect, i.e., statistical outliers (such as an individual point outside $\pm 3\sigma$ of a normal distribution) need to be identified and often excluded if a natural cause can be identified.

3.1.3 Rule 3: Measurement Scatter Versus Tolerance Width or Total Scatter. Rule 3 is applied to analyze whether the measurement method is appropriate, i.e., ensuring that the measurement uncertainty of the method does not use too much of the tolerance width or production scatter (Ref 16).

There are two possibilities:

(i) <10% of tolerance width (double-sided tolerance, i.e., both minimum and maximum requirements)

(ii) <25% of production scatter (single-sided requirement, i.e., either minimum or maximum requirement)

3.2 Test of Production Data

Four new spray tests in the new spray equipment were compared to initial qualification data (16 runs).

After drawing a standard run chart, a statistical analysis, such as a two-sample *t*-test (Ref 16), was made for each property, for example, thickness, deposition efficiency, voltage, and online particle properties in the flame. $p < 0.05$, i.e., 95% significance for being different, was used as the failure criterion.

Special care needs to be taken regarding deposit efficiency and voltage, due to the new equipment, which are discussed below. Powder lot must also be taken into account for those coatings where powder lot is crucial to the results.

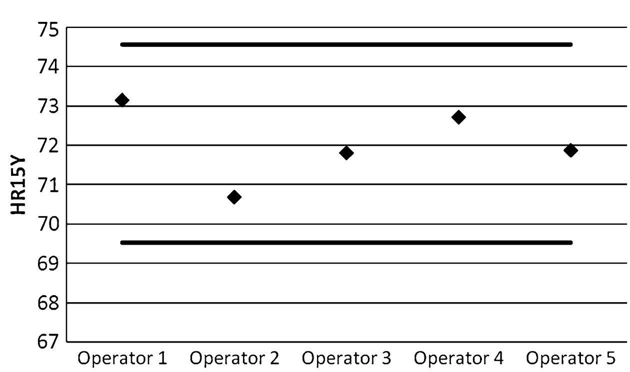

Fig. 1 Test whether each operator's average is within $\pm 2\sigma$ (solid black lines) of all values for HR15Y

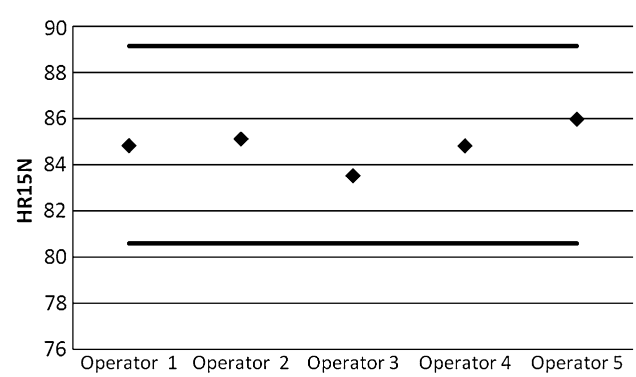

Fig. 2 Test whether each operator's average is within $\pm 2\sigma$ (solid black lines) of all values for HR15N

4. Results

4.1 Laboratory Example: Hardness Test

Macro hardness per ASTM E18 using HR15Y and HR15N was applied to aluminum-polyester coating and WC-Co coating, respectively. Five operators performed hardness measurements on the same coating thickness 1040 and 310 μm, respectively. Each measurement consisted of 15 and 10 indentations for HR15Y and HR15N, respectively.

4.1.1 Rule 1: 2 Sigma Test. All operators passed the test per Rule 1, Fig. 1 and 2.

4.1.2 Rule 2: ANOVA Analysis. The ANOVA-analysis of the HR15Y method, Fig. 3, shows that one operator was an outlier ($p < 0.0005$). The interesting point with this is that this operator was new and under training. He was included just for curiosity. The test, thus, failed regarding Rule 2 for HR15Y, and the conclusion was that the operator under training needed further training before he could be certified.

The test per Rule 2 for HR15N, Fig. 4, resulted in $p = 0.14$, i.e., > 0.05, which means acceptable results (non-difference could not be verified to 95% confidence).

4.1.3 Rule 3: Measurement Scatter Versus Tolerance Width or Total Scatter. In the case of HR15Y, there are double-sided requirements, which mean that Rule 3(i) can be selected:

- Standard deviation (1σ) for the operator's average is 1.26
- Tolerance width is 20HR15Y (60-80)
- 1 standard deviation divided by tolerance width is 6% (Requirement: <10%)

When comparing the total scatter with the tolerance width, it shows that only 6% of the width is used for measurement scatter. Method is OK.

In the case of HR15N, there is a single-sided requirement, where Rule 3(ii) needs to be used:

- Standard deviation (1σ) for the operator's average is 0.88
- Production scatter from the last 15 spray occasions showed a σ of 1.2
- 1 standard deviation divided by production scatter is 0.73, i.e., 73% (Requirement: <25%)

This method is NOT good enough for this purpose, and needs to be improved. Knowing that the number of indentations is crucial for coatings, a second round robin was performed, where each operator was told to perform 30 indentations per measurement;

- Standard deviation (1σ) for the operator's average is 0.38
- Production scatter from the last 15 spray occasions showed a σ of 1.2
- 1 standard deviation divided by production scatter is 0.32, i.e., 32% (Requirement: <25%)

The method has now substantially been improved, and comes close to established recommendations. (It must, however, be noted that just adding number of indentations to improve the measurement method, may not be approved by all OEM's (original equipment manufacturer).)

4.1.4 Conclusion to Laboratory Examples. HR15Y is an appropriate method, but the round robin shows that the results are operator dependent. One operator needs further training before being certified for HR15Y measurement.

HR15N is a borderline method if 30 indentations are being used per measurement, but the round robin shows no operator dependence.

4.2 Production: Example

4.2.1 Tensile Strength Comparison. Four spray runs (plasma spraying of Inconel 718) were made in the new spray equipment, and then compared to the initial qualification (16 spray runs) made 10 years ago. First, a run

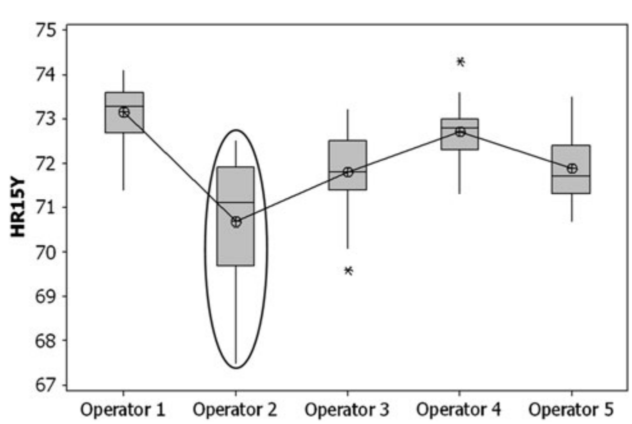

Fig. 3 ANOVA analysis of the five lab operators involved with HR15Y measurements. Asterisk (*) identifies one individual indentation being an outlier

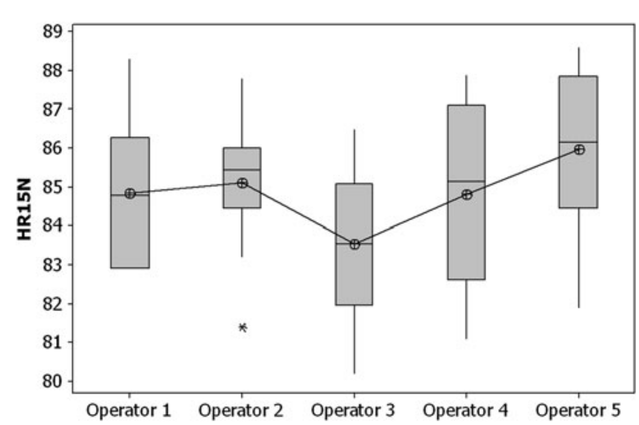

Fig. 4 ANOVA analysis of the five lab operators involved with HR15N measurements. Asterisk (*) identifies one individual indentation being an outlier

chart, Fig. 5, was created, showing a comparison of the tensile strength test data (coating thickness was 1550-1610 μm).

Each data point is an average of three tensile strength measurements.

The box plot, Fig. 6, is from the two-sample *t*-test (between the four spray runs in new machine and the 16 original spray runs), which concluded that the difference is between −11.03 and 4.75 to 95% confidence.

The conclusion is that to 95% confidence a difference can not be verified, and that the data thus are acceptable.

However, this is the simplest case. For many powders, there is a strong powder lot correlation to the coating properties, meaning that a direct comparison to initial data will prove a difference. Thus, powder variations often must be taken into account. Figure 7 shows a chart of tensile strength results for a typical Ni-5Al plasma sprayed coating, where it is clearly shown that the initial qualification data cannot be used directly.

The graph shows the first 16 qualification runs, all receiving inspection tests being made over the years (represented by different markers) and finally, the four new data from the new spray equipment. All are sprayed in pristine condition, which means spraying on a cylindrical shaped fixture with a diameter of 12 in., and using a simple vertical movement for the robot.

The two-sample *t*-test between the initial qualification data and the four latest data points (the qualification data from the new machine) clearly conclude a significant difference, i.e., $p < 0.05$. In this case, a two-sample *t*-test must be made from the data, using the same powder lot rather than the initial qualification data to make a fair

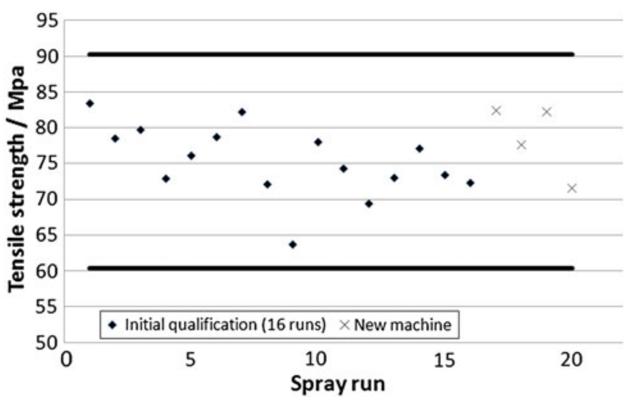

Fig. 5 Run chart showing initial qualification data (16 runs) vs. new machine data (4 runs) for plasma sprayed In 718 (solid black lines are ±3σ as calculated from the initial 16 runs

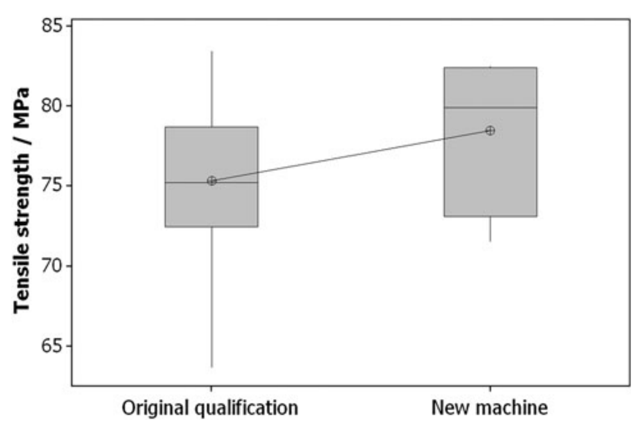

Fig. 6 Box plot from Minitab (Minitab Inc, State College, Pennsylvania, US) illustrating the two-sample *t*-test results between initial qualification data and new machine data for plasma spraying of Inconel 718

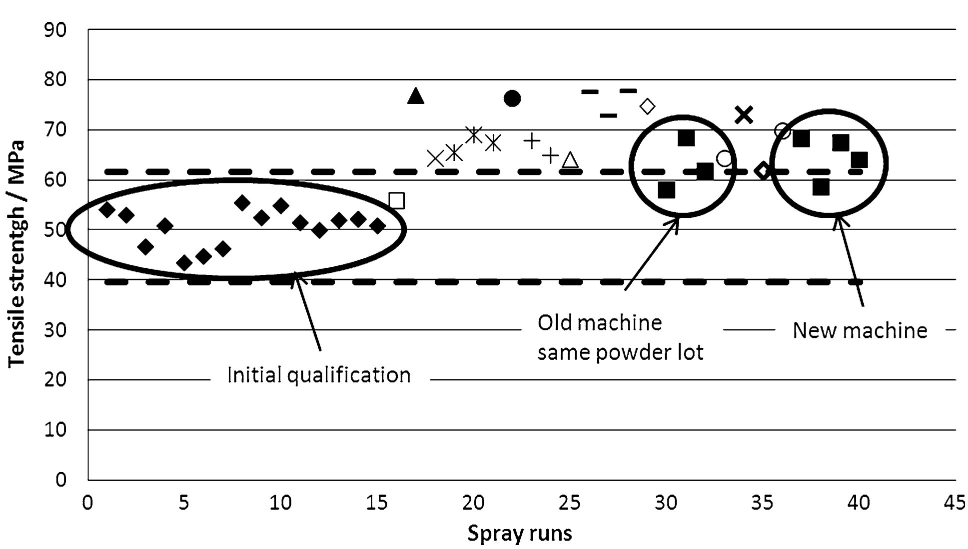

Fig. 7 Chart showing initial qualification data (16), all sprayability tests for different powder lots and finally the new machine data (4 latest) for plasma-sprayed Ni-5Al. Dashed lines are ±3σ for original data. Different markers identify different powder lots, all sprayed in pristine condition

comparison. As per Fig. 7, it can be seen that only three data points existed from this powder lot sprayed in pristine condition (and to the same thickness), which is rather too few for statistical analyses. Thus, it was decided to make the two-sample *t*-test using all individual values, i.e., 9 (3×3) and 12 (4×3) individual points, respectively.

p-Value is 0.436 and the data pass the test, i.e., difference could not be verified to 95% confidence, Fig. 8.

The correlation between powder lots and tensile strength was not further analyzed in this study, but an explanation was already suggested by Svantesson and Wigren in 1992 (Ref 18).

In many cases, there is only one sprayability test per powder lot. In this case, one would have to use production data. However, this increases the risk of failing the test, since thicknesses and geometry may vary too much.

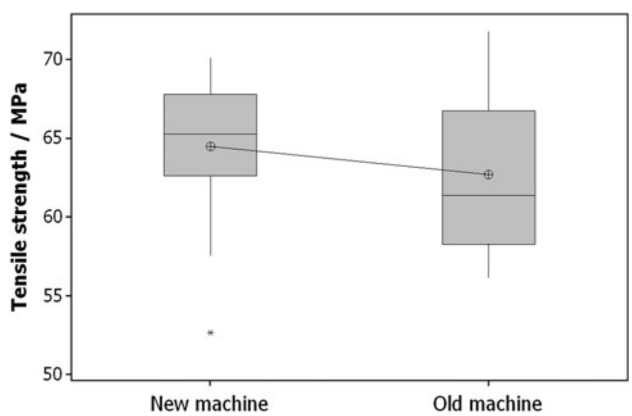

Fig. 8 Box plot from Minitab illustrating the two-sample *t*-test results between initial qualification data and new machine data for plasma spraying of Ni-5Al with the same powder lot. Asterisk (*) identifies one individual pull being an outlier

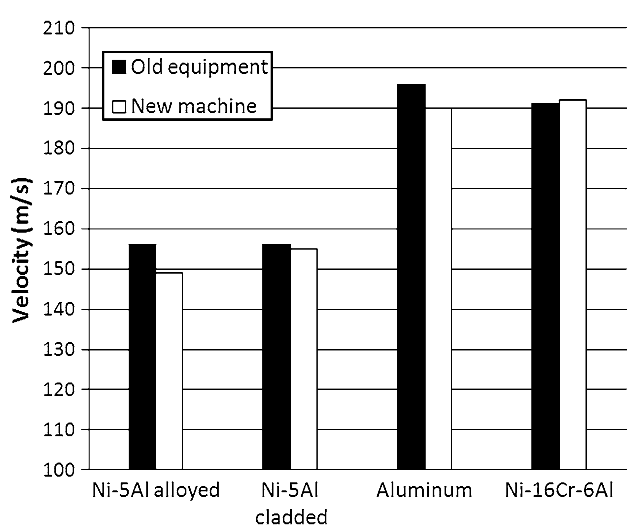

Fig. 9 In-flight particle velocity difference between old and new equipment for various coatings

4.2.2 Particle Properties. In order to finally prove the similarity between machines, the in-flight particle properties were also compared. Both DPV2000 and Accuraspray (Tecnar Automation Ltd, Quebec, Canada) are being used as equipment for particle diagnostics at Volvo Aero, but data below are from DPV2000 measurements.

In this case, not enough old data were available. Instead, a difference of less than ±5 m/s was used as acceptance level. This value is rather derived from experience than from any statistical method. Figure 9 demonstrates that the particle velocities can be considered as the same between the machines.

Figure 10 shows the differences between particle temperatures. As it can be seen (e.g., for the aluminum particles) the absolute values cannot be trusted, but the differences can be trusted. A difference of less than ±50 °C was used as acceptance level.

4.2.3 Deposit Efficiency. The deposition efficiency comparison is often much more inconclusive. Figures 11 and 12

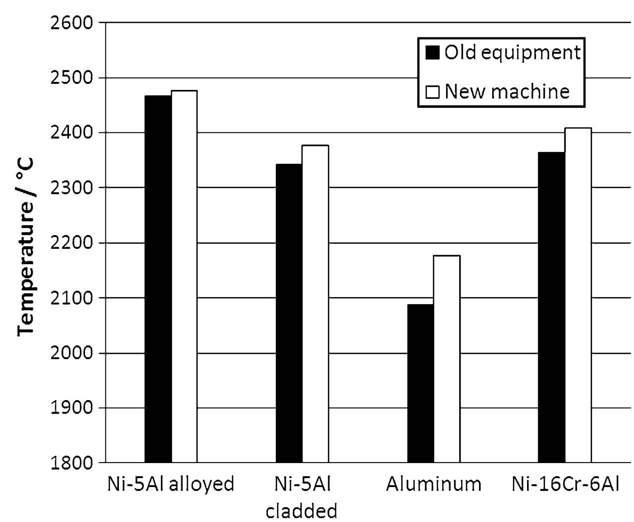

Fig. 10 In-flight particle temperature difference between old and new equipment for various coatings

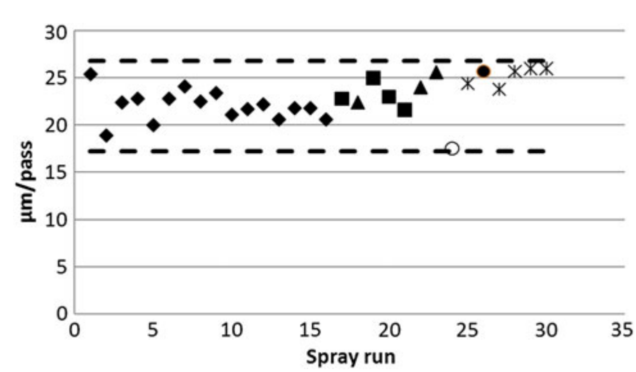

Fig. 11 A chart of µm/pass for plasma spraying of aluminum. First 16 points are the initial qualification. Last four are sprayed with the new machine. Different markers identify different powder lots, all sprayed in pristine condition

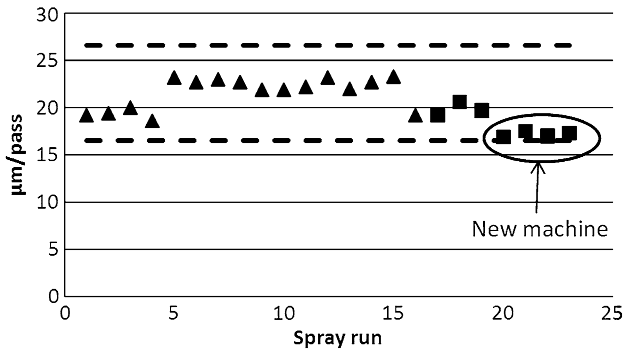

Fig. 12 A chart of µm/pass for plasma spraying of Ni-5Al. First 16 points are the initial qualification. Different markers identify different powder lots, all sprayed in pristine condition. Last four spray runs are from the new machine

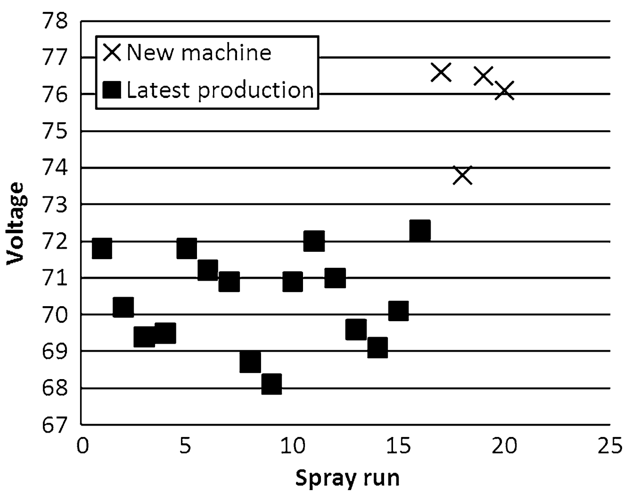

Fig. 13 Gun voltage difference for a Ni-5Al spray parameter between old and new machines

show some charts for deposit efficiency for aluminum and Ni-5Al spraying, respectively.

Four spray runs are generally insufficient in this case. Statistically, it is almost always possible to verify a difference to 95% confidence. In Fig. 11, for Al-spraying, the deposit efficiency is significantly higher, whereas in Fig. 12, for Ni-5Al spraying, the deposit efficiency is lower in new machine.

However, the data are all within $\pm 3\sigma$, with respect to original qualification data.

4.2.4 Voltage. The new rectifier technology automatically leads to a higher voltage in the plasma spray gun, Fig. 13 and 14. In the voltage case, the age of the nozzle is of utmost importance, since it generally drops the older the nozzle gets. The lower voltage tolerance is typically used as indication of maintenance need.

The voltage is not dependent on part geometry. Therefore, any spray configuration using the same spray parameter can be used in the evaluation.

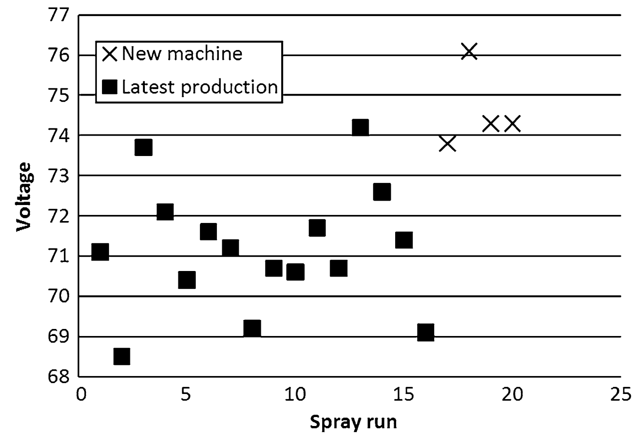

Fig. 14 Gun voltage difference for a Ni-18Cr-6Al spray parameter between old and new machines

Two examples are given below: for a Ni-5Al spray parameter and for a Ni-18Cr-6Al parameter.

Two-sample *t*-tests indicate a significant difference of 3.3-7.4 and 2-5 V (to 95% confidence level) between the old and new machine for the Ni-5Al and the Ni-18Cr-6Al parameter, respectively.

5. Conclusion

Modern statistical analysis tools, such as two-sample *t*-tests, ANOVA analyses, run charts, Six Sigma guidelines, applied to thermal spraying to verify reproducibility, repeatability, and reliability can reduce costs in qualifying new booths and operators. It is a necessity that all data are continuously stored in databases for efficient continuous analyses.

With the use of these techniques, a high capability can be maintained over years, for example, during change of personnel and equipment.

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