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Simulation of Hardness Testing on Plasma-Sprayed Coatings

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A plasma-sprayed thermal barrier coating consisting of a NiCoCrAlY bond coat and Ce-stabilized zirconia ceramic coating was heat-treated at 400°C for 1000 h. Microhardness measurements were used to evaluate microstructural variations throughout the coating. One hundred and twenty measurements were performed at both the bond coat and ceramic coating positions within the thermal barrier coating system. Both data sets were analyzed to assess whether they could be described as Gaussian (i.e., "normal") or Weibull distributions. The influence of the sample size, i.e., the number of microhardness tests for a group, on the mean hardness value was also evaluated by a Monte Carlo simulation procedure. The mean value, the standard deviation, the coefficient of variation, and the Weibull modulus for the subsets of data were calculated to assess these effects. The confidence for the mean value was also considered. The results indicated that the reliability of the microhardness test improved as the sample size increased. At least 20 measurements were needed to distinguish differences in microhardness between the bond coat and the ceramic coating at a 95% confidence level.

I. Introduction

THERMAL barrier coatings consisting of a plasma-sprayed ceramic coating and a bond coat are widely used to protect components which are subjected to elevated temperature environments.[1-3] The intrinsic properties of the bond coat and the ceramic coating govern the performance of the coating system. These properties, however, may change after use in high-temperature environments. Investigations to understand the failure mechanisms during operation have proposed that oxidation of the bond coat and residual stress changes within the ceramic coating play important roles in degradation of the coating system.[4,5] The reliability of the coating system before and after operating conditions can be assessed by mechanical property measurements, especially by microhardness methods.[6]

A microhardness test[7]. is defined as "using a calibrated machine to force a diamond indenter of specific geometry, under a test load of 1 to 1000 gf, into the surface of the test material." The diagonals of impression are optically measured, and usually 5 or 10 measurements are made to obtain the desired hardness number.[7] The repeatability for the test can be approved if the variation of the mean values of two or more groups of five indentations is less than 5%. However, because of the composite and heterogeneous nature of thermal spray coatings, often 10 measurements are not sufficient to ensure the repeatability of the mean microhardness. Thus, the reliability of the microhardness test for plasma-sprayed materials, when performed according to the ASTM standard, is questionable.

In this study, the variability of data was assessed by the Gaussian and Weibull distributions. Important statistical parameters including the mean (μ), the standard deviation (a), the coefficient of variation (CV = $100(\sigma/\mu)$, in %), and the Weibull modulus (m) will be used. The Gaussian[8] and Weibull distributions[9,10] have been discussed and are not detailed here. The influence of the sample size of a group of microhardness tests on the mean hardness value was evaluated by a Monte Carlo simulation method[11] which randomly generated subsets of data for specific sample sizes. The mean value, the standard deviation, the coefficient of variation, and the Weibull modulus for the subsets of data were calculated to assess these effects. The objective was to improve the practical applications of the microhardness test when used for testing of thermal spray coatings and to understand the significance of test errors due to material variability.

II. Experimental Procedure

A thickness of 200 μm NiCoCrAlY bond coat (Metco 461) was vacuum plasma sprayed onto a metallic substrate, followed by air-plasma-sprayed 1300 μm ceria-stabilized zirconia (Metco 205) to produce the thermal barrier coating system which was then aged at 400°C for 1000 h.

A MICROMET II tester was used to obtain the Vickers hardness number (HV) with a load of 300 g for 15 s. Microhardness data for the specimen were measured within the bond coat and at three different locations within the ceramic coating, i.e., 300 μm from the bond coat-ceramic coating interface, in the middle, and 300 μm from the free surface (Fig. 1). For the ceramic coating, 40 readings, which were taken randomly along each location of interest, were measured, and 120 measurements were performed in the middle of the bond coat. A total of 120 measurements were taken for both the bond coat and the ceramic coating and analyzed using Gaussian and Weibull statistics.

The influence of the sample size on the mean hardness value and data scatter was evaluated by a Monte Carlo simulation procedure. A flow chart of this procedure is shown in Fig. 2. A specified number of readings were randomly selected from the

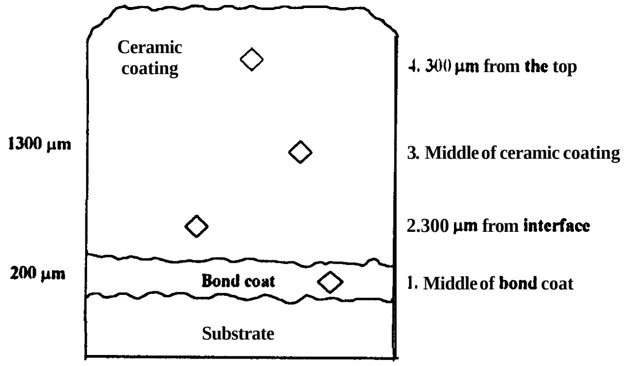

Fig. 1. Schematic representation of testing locations within the thermal barrier coating.

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parent distributions (that is, the data sets for the bond coat and the ceramic coating) to create a subset of data and then the mean value, the standard deviation, the coefficient of variation, and the Weibull modulus for these subsets of data were calculated to assess the effect of sample size.

III. Results and Discussion

Earlier studies suggested that complex processes such as stress relaxation, growth of oxide layers within the bond coat, and phase changes occur within the coating system.[6] In the present study, a prime purpose was to distinguish the difference (if any) in microhardness between the bond coat and the ceramic coating. Also, the influence of sample size on the average hardness could be better understood.

Table I shows the microhardness results for both the bond coat and the ceramic coating, and Fig. 3 shows Weibull plots for both coatings. The Weibull plots regarding different testing locations within the ceramic coating are shown in Fig. 3(b). With regard to the ceramic coating, a slight increase in average hardness according to the different positions is observed; however, the Weibull modulus does not change significantly. The increase in hardness from the bottom of the ceramic coating may be due to the grain growth induced by heat transfer during the spraying process.[12] Figure 4 shows the histograms as well as the probability density functions (PDFs) for both data sets. In Fig. 4(b) and other occurrences where $n = 120$ for the ceramic coating, the data for the three test locations have been pooled. The PDF for the Gaussian distribution (as shown by the dotted line) is a bell shaped curve; however, the PDF for the Weibull distribution is skewed to the left. The Weibull distribution may be a better representation of the microhardness within the bond coat.

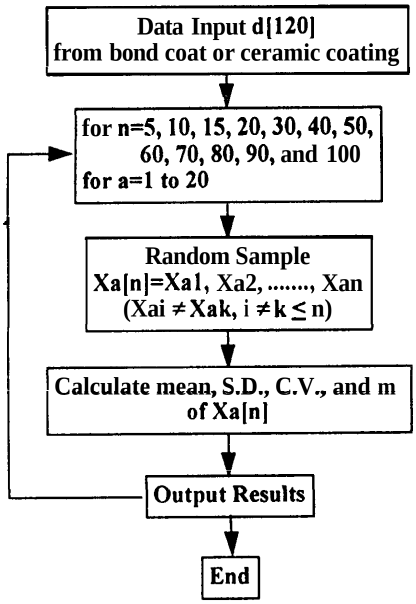

Fig. 2. Flow chart for Monte Carlo procedure (n is the specific sample size for the simulation procedure and a is the number of the simulation cycle).

Each subset of data from the simulation procedure can be considered as an individual group of tests for the microhardness measurements. The mean hardness for the subset is identified as μ_i (i = 1 to 20) and forms a new distribution of the mean hardness. For each specific sample size (n = 5, 10, 15, 20, 30, etc.), a corresponding distribution of the mean hardness is generated.‡ The mean, the standard deviation, and the coefficient of variation for these new distributions (i.e., μ_i, i = 1 to 20 with different n) are identified as $\overline{\mu}$, $\overline{\sigma}$, and $\overline{CV}$, respectively. Figure 5 shows $\overline{\mu}$, which is the average value of the means, with respect to sample size n, where $\overline{\sigma}$ is represented by error bars. It can be noted that at least 20 measurements are needed to distinguish differences in hardness between the bond coat and the ceramic coating. The $\overline{\mu}$ value fluctuates around the mean value of the original parent data set. The larger the sample size n, the smaller the fluctuation; therefore, the uncertainty for a material property test can be abridged by increasing the number for the test. This can be verified by plotting the coefficient of variation for the distribution of the mean (i.e., $\overline{CV}$, which implies the relative variation of the mean from the parent data sets) with respect to the sample size (Fig. 6). It is also noted that the $\overline{CV}$ decreased to below 5%, which is a general limit for repeatability, if 15 measurements were selected for the bond coat and 20 measurements for the ceramic coating. Though 120 tests are not sufficient to represent the real microhardness distribution within the material, this plot provides information about the variation of the mean hardness for different sample sizes. The confidence intervals, which can be used to confirm the trend shown by the coefficient of variation, for the sample size effect were also considered.[8,13] The deviation of μ_i from the mean value fitted into the 95% confidence lines if the sample size was larger than 20 is shown in Fig. 7.

The data scattering within each subset of data is assessed by the coefficient of variation (CV_i, i = 1 to 20), which fluctuates with respect to the CV of the parent data sets (i.e., CV = 16.6 and 27.4 for the bond coat and the ceramic coating, respectively). The coefficient of variation for each subset of data is related to the Weibull modulus, which is an alternative way to describe the data scattering. The larger the Weibull modulus, the smaller the coefficient of variation. The Weibull modulus for each subset of data is identified as M_i (i = 1 to 20).§ Figure 8 shows the Weibull modulus with respect to the sample size. It can be noted that the central tendency of the Weibull modulus increases with increasing sample size. The variation of the Weibull modulus decreases to 10%, if 40 readings are selected. The Weibull modulus, however, depends more on the parent sample size. According to the Monte Carlo simulation by Ritter *et al.*,[14] the coefficient of variation for 100 specimens is 10%. Thus, when considering the scatter in the data, the Weibull modulus with confidence intervals may be a better way of expressing the results.[10,15]

Whatever material property is tested, the uncertainty of a material property test depends on the number of measurements. Every five measurements can be considered as an individual

‡For Tables II and III, giving the Monte Carlo simulation results of the Gaussian distribution for the bond coat and the ceramic coating, respectively, order ACSD-221 from the Data Depository Service, American Ceramic Society, 735 Ceramic Place, Westerville, OH 43081.

§For Tables IV and V, giving the Monte Carlo simulation results of the Weibull distribution for the bond coat and the ceramic coating, respectively, order ACSD-221 from the Data Depository Service, American Ceramic Society, 735 Ceramic Place, Westerville, OH 43081.

Table I. Microhardness Results for Both the Bond Coat and Ceramic Coating

Testing location	Number of tests	Mean	SD	CV (%)	m	X_o
Bond coat	120	229.8	38.1	16.6	6.8	246
Ceramic coating	120	206.7	56.7	27.4	4.2	227
300 μm from BC-CC interface†	40	189.4	53.8	28.4	3.7	210
Middle of ceramic coating	40	207.0	54.0	26.1	3.8	230
300 μm from the free surface	40	218.8	62.5	28.6	4.0	241

†BC: bond coat. CC: ceramic coating.

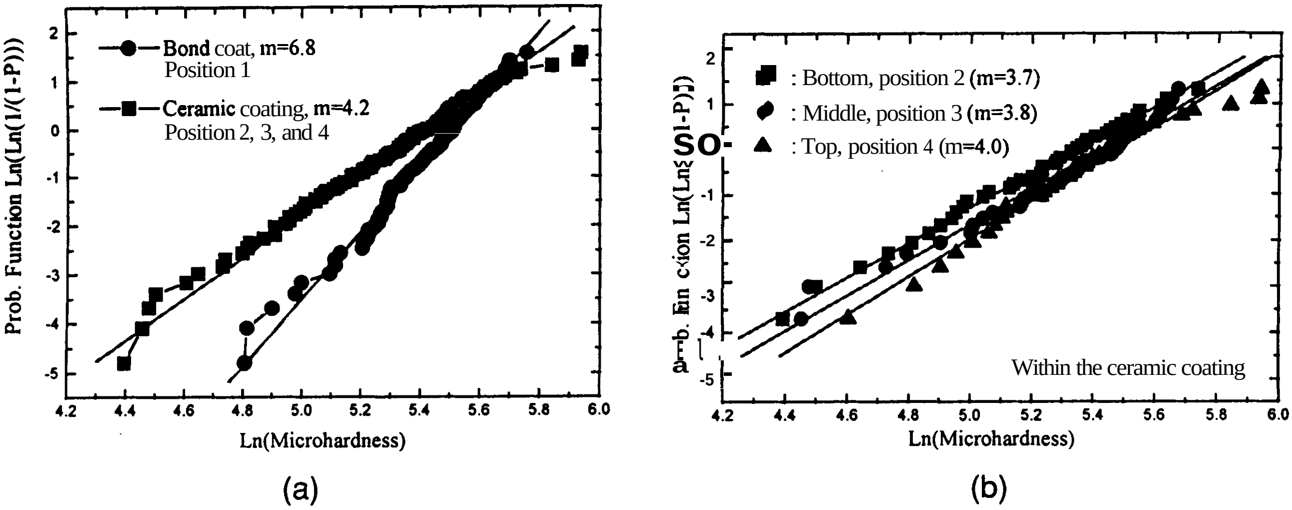

Fig. 3. Weibull analysis results for bond coat and ceramic coating (complete data sets used).

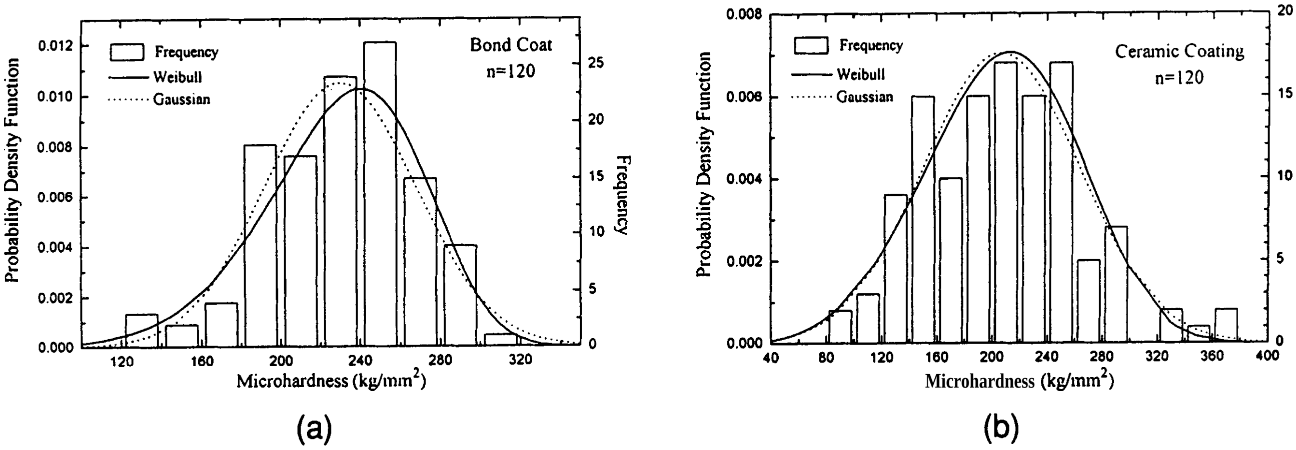

Fig. 4. Histogram and probability density functions plots for the parent data sets of the bond coat and ceramic coating ($n = 120$).

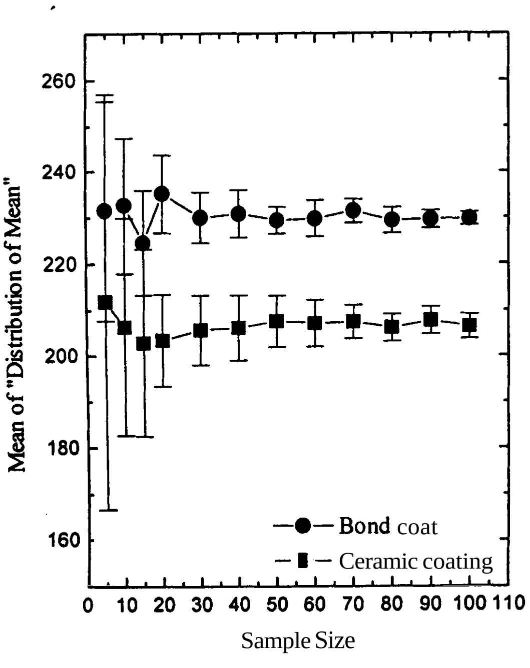

Fig. 5. Mean **hardness** of 20 **cycles (i.e., $\overline{\mu}$)** for bond coat and ceramic coating versus **sample** size (standard deviation $\overline{\sigma}$ **was** represented **by** error **bar).**

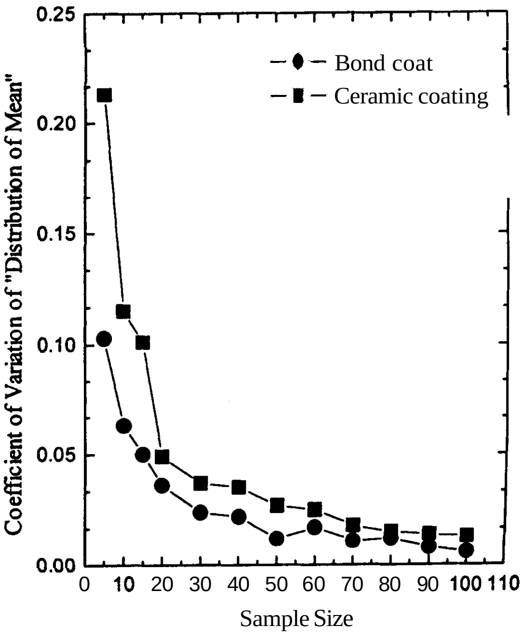

Fig. 6. **Coefficient** of variation for 20 cycles **(i.e.,** $\overline{\sigma}/\overline{\mu}$) versus ***sample*** size.

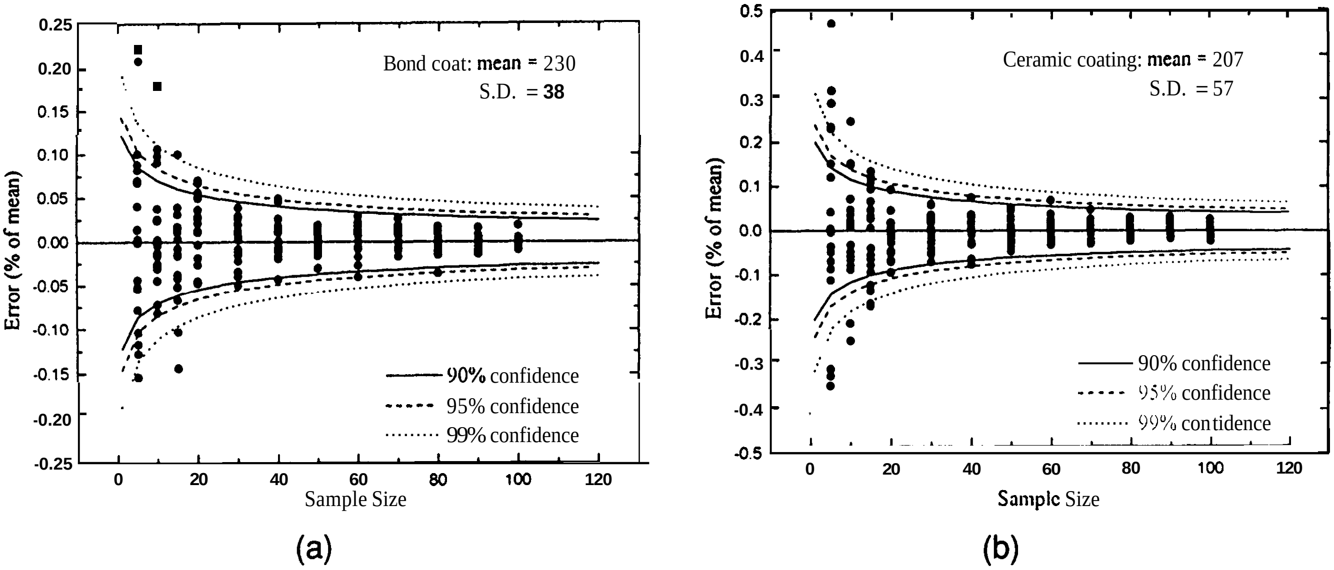

Fig. 7. Confidence intervals versus sample size.

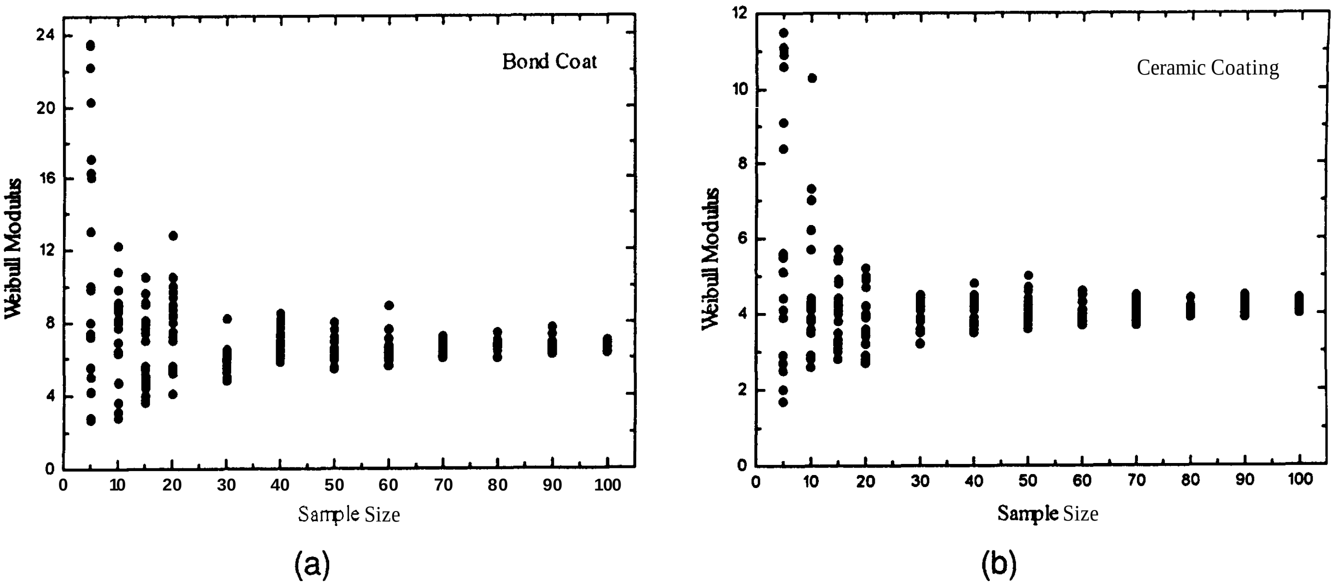

Fig. 8. Weibull modulus for subsets of data versus the sample size.

group. If the variation of mean values for two groups does not exceed 5%, the repeatability of the test is acceptable, or else, 10 or more measurements are necessary for a reliable test result. The engineering reliability for a material test can be improved by increasing the total test number, but at the expense of increasing effort and cost.

IV. Concluding Remarks

The microhardness test was used to assess the homogeneity within the coating system, and the engineering reliability was evaluated by a Monte Carlo simulation procedure.

The distribution of the mean, which is generated by a Monte Carlo simulation procedure, fluctuates around the mean value of the parent distribution; the fluctuation decreases as the sample size increases. At least 20 measurements are needed to distinguish the difference in microhardness between the bond coat and the ceramic coating. Meanwhile, the coefficient of variation for the distribution of the mean decreases to below 5%, if 15 measurements are made for the bond coat and 20 for the ceramic coating. The confidence tests confirm this trend. The errors fit into 95% confidence levels if 20 measurements are made. However, the reliability for data scattering, which is evaluated from the Weibull modulus for each subset of data, requires a larger sample size. If 40 measurements are made, the variation of the Weibull modulus is around 10%. A final point is that the size of the parent distribution in this study was 120 experimental values, whereas it is not unusual for Monte Carlo methods to numerically generate parent sets of thousands of data points. It would be expected that the statistical analysis of a much larger data set (if available) would be slightly different from those reported since a parent distribution consisting of experimental data was used in the present study.

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