Statistical Analysis of Microhardness Variations in Plasma Sprayed Cr_3C_2-NiCr Coatings

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ABSTRACT

The microstructure and properties of plasma-sprayed coatings depend on a great number of spraying parameters, random factors, which lead to vibration in these spraying parameters, may in some degree influence the microstructure and properties of the coatings. Therefore, the property values appear certain distributions, and the description and comparison of the properties of plasma-sprayed coatings should be performed employing statistical analysis. In this paper, Cr_3C_2-NiCr coatings of different thickness were sprayed onto stainless steel using atmosphere plasma system and adopting three kinds of gun translation speeds. Then the microhardness measurements were performed on polished surface of the coatings. Forty readings were taken and statistically analyzed by calculating the characteristic values, estimating and comparing the means, and assessing whether they belonged to the Normal or Weibull Distribution. This study has found that statistical analysis could discriminate influence of spraying parameters and coating design on microhardness of the Cr_3C_2-NiCr coatings from random vibration, which showed that the microhardness of the Cr_3C_2-NiCr coatings were related to gun translation speed and coating thickness.

Keywords: Statistical analysis, plasma spraying, microhardness, Cr_3C_2-NiCr coating

1. Introduction

Chromium carbide coatings are widely used in high-temperature wear-resistant and corrosion-resistant applications in aerospace, automobile and transportation industries [1-3]. There have been many reports on the properties of plasma-sprayed Cr_3C_2-NiCr coatings. These property parameters appeared rather scatter [1-5]. For example, the microhardness of plasma-sprayed Cr_3C_2-NiCr coating even varied from 454 to 752 under the identical process parameters [5]. However, the phenomenon about property scatter of plasma-sprayed coatings has not been paid enough attention. In fact, the microstructure and properties of plasma sprayed coatings depend on many spraying parameters such as plasma power, plasma gases, powder feed rate, spraying distance, spraying angle, gun translation speed and substrate cooling [6-8]. Random factors, which lead to vibration in these spraying parameters, may in some degree influence the microstructure and properties. Therefore, evaluation and comparison of the properties of plasma sprayed coatings should be performed employing statistical analysis.

Industry trends within the past several years have required denser and harder coatings [9]. In order to improve the properties of plasma-sprayed coatings to the desired levels available, it is necessary to understand fully the statistical characteristics of their properties so that it can investigate the relationships between the properties and spraying parameters for optimizing coating process.

During plasma spraying, various thickness of coating and gun translation speed may result in change in heat transfer between molten or heated particles and substrate to be coated and in the solidification rate of the molten particles, which are closely related to the microstructure and properties of the coatings [8]. The objective of this paper is mainly to study the statistical characteristics of microhardness of plasma sprayed Cr_3C_2-NiCr coatings, and to investigate whether statistical analysis can discriminate effects of thickness and gun translation speed on the microhardness the Cr_3C_2-NiCr coatings from random vibration.

2. Experimental procedure

Cr_3C_2-NiCr powder (Metco 82VF-NS) was sprayed onto stainless steel using atmosphere plasma system consisting of Sulzer Metco F4-MB plasma spray gun mounted on ABB S3 robot. Spraying parameters were adopted the recommended data by Sulzer Metco except for gun translation speed, which was regarded as an investigated factor influencing properties of the coating in this paper. Coating samples of three gun translation speeds and three thickness were applied on stainless steel plates of 50mm×30mm×2mm. Codes of the samples are listed in Table 1. For every set of spraying parameters, two identical coating samples were applied.

Table 1 Identification codes for coating samples

		Gun translation speed (m/min)		
		50	75	100
Thickness	50	A11,A12	A21,A22	A31,A32
(μm)	100	B11,B12	B21,B22	B31,B32
	300	C11,C12	C21,C22	C31,C32

Microhardness were measured on polished surface of the Cr_3C_2-NiCr coatings by indenting at a load of 50 g for 15 s using a HX-1000 Vickers hardness tester with an optical microscopy which has a magnification of 400. Each measurement series comprised 40 readings, which were randomly located on each sample. Data were then adjusted by subtracting the two largest and two smallest readings to discriminate against results that would be atypical of the overall coating properties. The statistical analysis of the adjusted data were performed by calculating the characteristic values, estimating and comparing the means, and testing whether they belonged to the Normal or the Weibull distribution.

3. Theory of statistical analysis

3.1 Characteristic values of data

The mean value, $\bar{x}$, and standard deviation, s, which are used to indicate the data scatter, of measured data series $x_1,x_2,\ldots,x_n$ are defined as [10,11]

$$\bar{x} = \frac{1}{n}\sum_{i=1}^{n} x_i \tag{1}$$

$$s = \sqrt{\frac{1}{n-1}\sum_{i=1}^{1}(x_i - \bar{x})^2} \quad (2)$$

The standard deviation and the variance, s^2, are measures of the absolute variation of data and depend on the scale of measurement. Thus it is more convenient to use the coefficient of variation, C_V, which gives a measure of relative variation [10,11], to compare several sets of data.

$$C_v = \frac{s}{\bar{x}} \times 100\% \quad (3)$$

3.2 The mean estimation

The mean estimation can give a confidence interval of the actual mean of the coating property, μ, with the characteristic values of measured data set at a certain confidence level. At the certain confidence level, 1-α/2, the confidence interval can be described as [10,11]

$$[(\bar{x} - \frac{s \bullet t_{n,\frac{\alpha}{2}}}{\sqrt{n}}) \le \mu < (\bar{x} + \frac{s \bullet t_{n,\frac{\alpha}{2}}}{\sqrt{n}})] \quad (4)$$

Where $t_{n,\alpha/2}$ is a value of the random variable t whose sampling distribution is approximated very closely by the t distribution.

3.3 Statistical tests

The student's t-test can be used to compare the hypothesis that the means of two data sets are equal if the data sets belong to the Normal distribution. If the t-test value, $P\{|t_n|>t_{n,\alpha}\}$, is unity then the two data sets are identical. If the value is very small (usually less than 0.05) then the means of the data sets, $\bar{x}_I$ and $\bar{x}_J$, are significantly different [10,11].

$$t_n = \frac{\overline{x_I} - \overline{x_J}}{\sqrt{\frac{s_I^2}{n_I} + \frac{s_J^2}{n_J}}} \quad (5)$$

Where s_I^2 and s_J^2 are the variances, n_I and n_J are the sample sizes of the two data sets, $n = n_I + n_J - 2$, α is the confidence coefficient.

The χ^2-test can determine whether a data set has a specified theoretical distribution under a test level. The complete agreement between the data set and the theoretical distribution will give zero for χ^2. The poorer is the agreement between them, the larger will be the χ^2 value.

$$\chi^2 = \sum_{i=1}^{k} \frac{(ei - mi)^2}{ei} \quad (6)$$

Where k is cell number for appropriate statistic and test, ei and mi represent the expected and measured frequencies, respectively, for the ith cell [10,11].

The cumulative density function of probability for measured data set, *F(i)*, is obtained from placing the data in ascending order and letting

$$F(i) = \frac{i}{n+1} \quad (7)$$

Where n is the total number of data points, and i is the ith order in ascending data set [12].

The Normal distribution is widely used by scientists and engineers to assess the degree of regularity of the data [10-12]. As the strength distribution of brittle materials can be highly skewed or broadly distributed and it is different and inadequate to describe the variation by means of the Normal distribution, the Weibull distribution is often considered to be suitful for implementing to accommodate this type of problem [12].

The density function $p(x)$ of the Normal distribution is given as [10,11]

$$p(x) = \frac{1}{s\sqrt{2\pi}} e^{-\frac{(x-\bar{x})^2}{2s^2}} \qquad (8)$$

The Weibull function, in the two-parameter form, is given as [12]

$$F(x) = 1 - e^{-(\frac{x}{x_0})^m} \qquad (9)$$

Where F(x) is the cumulative density function of probability, x_0 is the characteristic value below which 63.2% of the data lie, and m is the Weibull modulus, which reflects the data scatter within the distribution. The Weibull parameters m and x_0 can be obtained using the Weibull plot method. The Weibull plot can be drawn by rearranging the Weibull cumulative density function of probability and taking natural logarithms twice. Thus m and x_0 can be determined by least-square fitting the following equation

$$\ln\{\ln[\frac{1}{1-F(x)}]\} = m[\ln(x) - \ln(x_0)] \qquad (10)$$

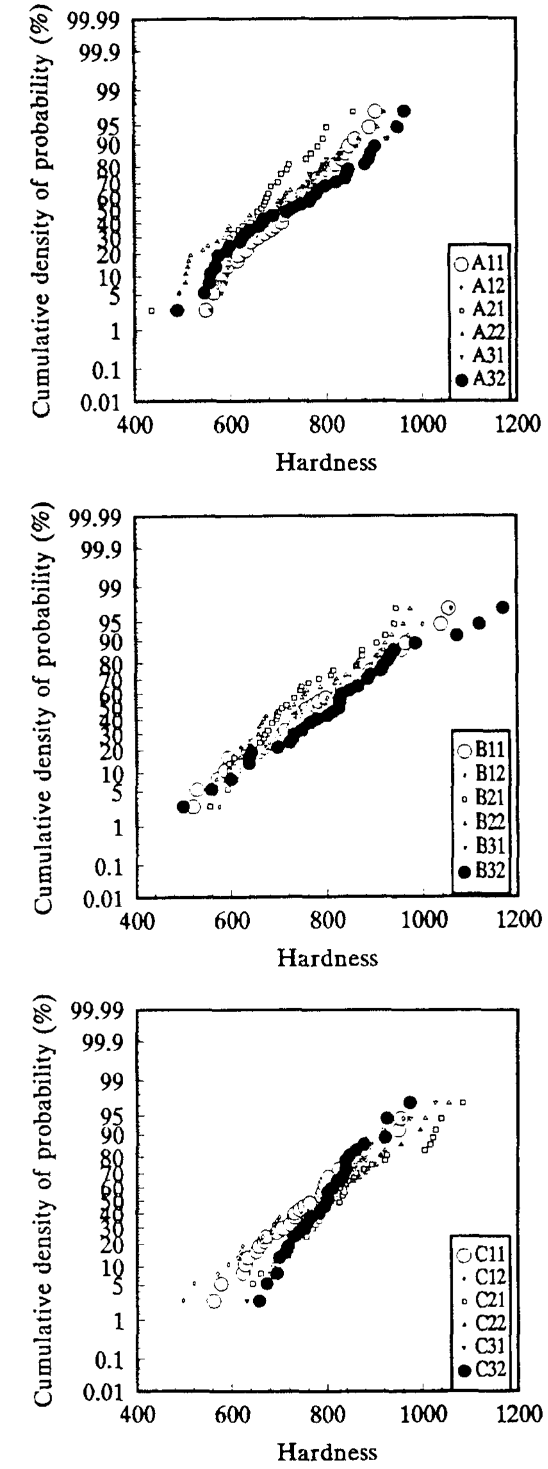

Fig.1 The cumulative densities of probability for microhardness data sets of the samples on the Normal probability paper.

4. Results and discussion

The results of statistical analysis of the measured microhardness data of the Cr_3C_2-NiCr coatings are listed in Table 2-4. In order to assess the χ^2–test, the χ^2 values of various α confidence intervals at 33 degrees of freedom, which is equal to the total number of each data set, 36, subtracted by 3, are also listed in Table 5 [10,11].

Table 2 The characteristic values and confidence interval of the microhardness data sets

Sample	Mean $\bar{x}$	Confidence -90%	Confidence +90%	Minimum	Maximum	Std. Dev. S	Var. Coe. Cv
A11	722	695	750	547	904	97	0.13
A12	716	680	753	476	975	130	0.18
A21	660	636	684	435	857	85	0.13
A22	677	640	713	487	922	130	0.19
A31	714	684	745	559	975	108	0.15
A32	727	689	765	490	965	135	0.19
B11	781	741	821	520	1055	142	0.18
B12	782	748	818	575	1061	124	0.17
B21	735	704	765	555	946	109	0.15
B22	739	703	775	500	975	127	0.17
B31	808	769	846	555	1055	137	0.17
B32	820	781	859	631	1171	138	0.17
C11	767	737	797	562	965	107	0.14
C12	768	732	804	498	985	130	0.17
C21	832	799	864	644	1084	116	0.14
C22	823	795	851	669	1055	101	0.12
C31	801	776	826	631	1027	89	0.11
C32	796	775	818	658	975	75	0.10

From Table 2, it can be seen that the microhardness measurements of all the 18 samples appear rather scatter basing on the minimal and maximal values. Moreover, the coefficients of variation vary from 0.10 to 0.19 for the different samples, which indicates that the relative variations for the different samples are obviously different. Therefore, it is impossible to compare the microhardness only by the

Table 3 The results of the student's t-test to compare the mean of the microhardness data sets

	A12	A21	A22	A31	A32	B11	B12	B21	B22	B31	B32	C11	C12	C21	C22	C31	C32
A11	<u>0.84</u>	00.0	0.01	0.74	0.86	0.04	0.02	0.60	0.53	0.00	0.00	0.06	0.09	0.00	0.00	0.00	0.00
A12	1	0.03	0.17	0.94	0.66	0.05	0.03	0.52	0.46	0.05	0.00	.007	0.09	0.00	0.00	0.00	0.00
A21		1	<u>0.54</u>	0.02	0.01	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
A22			1	0.18	0.11	0.00	0.00	0.04	0.04	0.00	0.00	0.00	0.03	0.00	0.00	0.00	0.00
A31				1	<u>0.66</u>	0.06	0.02	0.44	0.38	0.00	0.00	0.04	0.06	0.00	0.00	0.00	0.00
A32					1	0.10	0.07	0.80	0.70	0.02	0.0	0.08	0.20	0.00	0.00	0.01	0.01
B11						1	<u>0.96</u>	0.12	0.19	0.41	0.24	0.60	0.70	0.10	0.10	0.46	0.55
B12							1	0.08	0.14	0.41	0.23	0.57	0.63	0.08	0.13	0.46	0.56
B21								1	<u>0.88</u>	0.02	0.00	0.20	0.24	0.00	0.00	0.00	0.00
B22									1	0.12	0.05	0.30	0.32	0.00	0.00	0.02	0.02
B31										1	<u>0.72</u>	0.16	0.20	0.44	0.58	0.82	0.66
B32											1	0.07	0.10	0.70	0.92	0.50	0.36
C11												1	<u>0.98</u>	0.02	0.02	0.14	0.18
C12													1	0.03	0.04	0.20	0.25
C21														1	<u>0.75</u>	0.21	0.13
C22															1	0.32	0.20
C31																1	<u>0.82</u>

means if it is not further performed the statistical test.

Table 4 and Table 5 show that the microhardness data sets of all the samples can be described using the Normal distribution at the confidence level more than 95%, which can also be verified by Fig.1. In Fig.1, the cumulative densities of probability for microhardness data sets of all the samples are plotted on the Normal probability paper and show approximate patterns of linearity [10]. Thus the microhardness means of the different samples can be compared employing the student's t-test. Combined Table 2 and Table 3, it can be seen that the nearing means are statistically equal although the data sets appear rather scatter, and the relative variations change for the different samples. According to the above results, it can further be concluded that the statistical analysis can discriminate influence of gun translation speed and coating thickness on microhardness of the Cr_3C_2-NiCr coatings from random vibration.

Figure 2 shows the means of microhardness for different coating thickness and gun translation speeds. In Fig.2, the vertical bars represent the confidence intervals of the microhardness at 90 confidence level. As shown in Fig.2, the microhardness of the Cr_3C_2-NiCr coatings appears different trends with thickness if the gun translation speed is different. This result demonstrates that the microhardness of the Cr_3C_2-NiCr coatings are related to spraying parameters

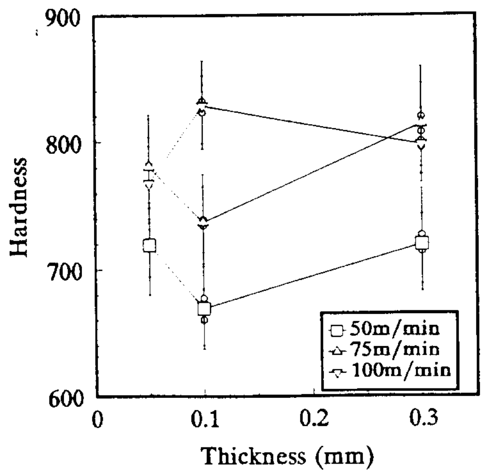

Fig. 2 The means of microhardness for different coating thickness and gun translation speeds.

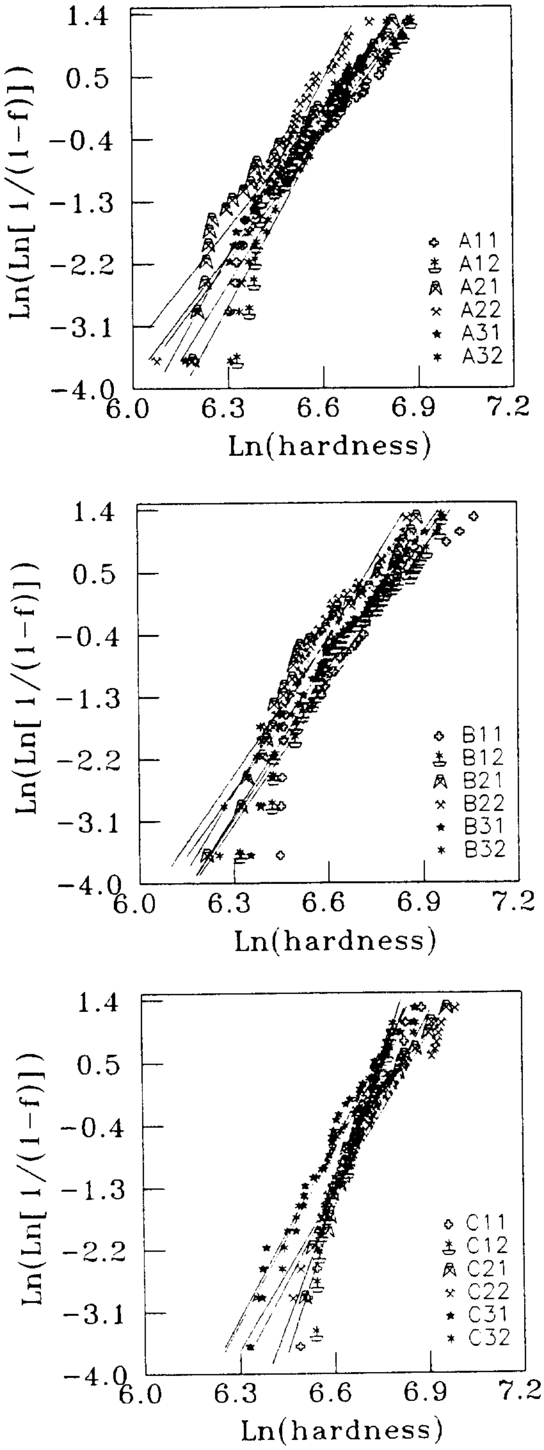

Fig.3 The Weibull plots of microhardness of the different sample.

and coating design.

As the Weibull distribution is often used to describe a wide range of problems including the mechanical properties of ceramic materials and lifetime testing [12,13]. The χ^2-test was also used to evaluate whether the microhardness data sets of the Cr_3C_2-NiCr coatings belong to the Weibull distribution in this study. From Table 4, it can be seen that except for sample A31, B32, C31 and C32, the data sets of the other samples can also be described using the Weibull distribution as shown in Fig.3 if the confidence level 95% is taken. The Weibull modulus m reflects the homogenous degree of data set. The more the m, the more homogeneous the data set is [12]. For this study, the Weibull moduli of the microhardness data sets are obvious more than ceramic deposits normally exhibit values in the range of 4-6, but less than 18 of the hypersonic sprayed Cr_3C_2-NiCr coatings [13]. The less Weibull moduli than that of the hypersonic sprayed Cr_3C_2-NiCr coatings should be attributed to the spraying process. The higher Weibull moduli of the sprayed Cr_3C_2-NiCr coatings may result from that NiCr alloy improve the homogeneousity of the coating as a result that the metal or alloy coating showed the higher Weibull moduli than ceramic coatings [12,13].

Table 4 The Weibull parameters and the results of the χ^2-test to evaluate whether the microhardness data sets belong to the Normal or the Weibull distribution

Sample	m	x_0	χ^2 to evaluate the Normal distribution	χ^2 to evaluate the Weibull distribution
A11	8.6	763	7.508	6.941
A12	6.2	768	9.512	9.048
A21	8.8	696	6.638	7.177
A22	6.2	727	9.036	9.089
A31	8.4	756	16.542	47.303
A32	6.4	777	6.055	5.875
B11	6.4	834	7.542	7.653
B12	7.4	832	10.507	12.413
B21	8.1	777	7.534	8.245
B22	6.9	788	5.633	5.393
B31	7.0	859	5.517	5.374
B32	7.3	872	19.400	46.160
C11	8.5	807	4.954	4.736
C12	6.6	820	4.107	3.427
C21	8.1	879	10.945	11.444
C22	9.1	863	15.298	20.279
C31	10.7	838	18.947	52.467
C32	12.7	828	13.093	28.498

Table 5 χ^2 values of various α confidence intervals at 36-3 degrees of freedom

α%	99.5	99	95	75	25	10	2.5	1
χ^2	15.815	17.074	20.867	27.219	38.058	43.745	50.725	54.776

5. Conclusion

The microhardness data sets of plasma-sprayed Cr_3C_2-NiCr coatings were statistically analyzed by taking forty readings for each sample. It was found that the microhardness data sets of all the samples could be described using the Normal distribution at the confidence level more than 95%. The student's t-test verified that the nearing means of microhardness were statistically equal although the data sets appeared rather scatter, and the relative variations changed for the different samples. Therefore, statistical analysis can discriminate influence of spraying parameters and coating design on microhardness of the Cr_3C_2-NiCr coatings from random vibration. The microhardness of the Cr_3C_2-NiCr coatings showed different trends with thickness if the gun translation speed was different, which demonstrated that the microhardness of the Cr_3C_2-NiCr coatings were related to spraying parameters and coating design. In addition, it was also found that most of the microhardness data sets of the Cr_3C_2-NiCr coatings belonged to the Weibull distribution in this study. The Weibull moduli of the microhardness data sets were in the range of 6.2-12.7.

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