Residual Stress Measurement of Plasma-Sprayed Coatings*

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Residual stresses of plasma sprayed coatings, NiCr alloy, CoNiCrAlY alloy, Al_2O_3 ceramics and ZrO_2 -8% Y_2O_3 ceramics, were measured in this study. SACHS method with a strain gauge was used to measure the residual stress. Type 304 stainless steel and mild steel were selected for the substrates. The results obtained were summarized as follows: 1) Tensile residual stresses were generated in plasma sprayed coatings. 2) The values of residual stress in ceramics coatings were relatively small and the values took lower than 40 MPa. 3) Tensile residual stress values of metallic alloy coatings on mild steel substrates were higher than those on type 304 stainless steel substrates. 4) Residual stresses in Low Pressure Plasma Sprayed (LPPS)-coatings were larger than those in Atmospheric Plasma Sprayed (APS)-coatings. 5) Pre-heating of a substrate will effectively reduce tensile residual stress in a coating.

Key Words: Residual Stress, Plasma-Sprayed Coating, Thermal Stress, Stress Relaxation

1. Introduction

As the improvement in the turbine inlet temperature directly leads to the improvement in efficiency of a power plant, thermal barrier coating (TBC) on the elements for high temperature service, such as a combustor, has become an indispensable technology. Generally, the thermal barrier coating is fabricated by plasma spraying. The half-molten particle by plasma energy flew at high speed and impacted to a substrate

or a layer laminated already. The particle was cooled, solidified and laminated. The coating was formed by deposition of the particles. Heat-cycle fatigue strengths and high temperature fatigue strengths of thermal barrier coated materials were studied by many researchers. However, there are very few measurement results⁽¹⁾⁻⁽⁶⁾ of the residual stress which has large influence on such fatigue strengths.

It has been said that the X-ray stress measurement method which is a nondestructive measurement method is effective to measure the residual stress of a thin layer like a sprayed coating. Almost all the studies of thermal barrier coatings were carried out by means of the X-ray method. The X-ray method which measure a stress of a surface layer (the penetration depth is several 10 μm) is useful for measuring a microscopic residual stress. By the way, authors⁽⁷⁾ and Socie et al.⁽⁸⁾ has found that the relative deformation between particles is larger than the deformation of a particle because the sprayed coatings have many pores and defects. So the relative deformation is known to govern the stress-strain behavior of a

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sprayed coating.

For this reason, understanding of not only the microscopic residual stress by X-ray measurement but the macroscopic average residual stress is important. Moreover, a ZrO_2 -8% Y_2O_3 coating which is a typical TBC is hard to measure the stress by X-ray method. The reason is that the coating has few isolated peaks of X-ray diffraction⁽⁹⁾, because the material is a tetragonal system similar to a cubic system. In this study, the macroscopic average residual stress of the whole sprayed coating was mechanically measured by the SACHS method⁽¹⁰⁾ which was the stress relaxation method. The arising mechanism of residual stress was discussed with the measured residual stresses. As this study was a part of the high temperature fatigue strength researches^{(11),(12)} of thermal barrier coated material, residual stresses were measured in Al_2O_3 and ZrO_2 -8% Y_2O_3 used for ceramics coatings, and in NiCr and CoNiCrAlY used for bond coatings.

2. Test Procedures

2.1 Materials and specimens

Specimens for measuring residual stresses of Al_2O_3 and ZrO_2 -8% Y_2O_3 ceramic coatings and NiCr and CoNiCrAlY metallic coatings were manufactured. The manufacturing procedure of the specimen is as follows: First, a coating was sprayed on the surface of a columnar substrate as shown in Fig. 1. In order to investigate the influence of a coefficient of linear expansion, α , on a residual stress, two kinds of materials, austenite stainless steel (SUS304) and mild steel (SS400), were selected for the substrates. All the coatings were deposited by atmospheric plasma spraying (APS) on the substrates after blast (WA # 24) treatments. CoNiCrAlY coatings were also deposited by low pressure plasma spraying (LPPS). The spraying powders used in this study are shown in Table 1. The spraying conditions are shown in Table

Fig. 1 Shape and dimensions of the specimen

2. Thicknesses of all the coatings were about 300 μm .

Longitudinal-sections of coatings were observed using a scanning electron microscope (SEM). Longitudinal-sections of metallic coatings (CoNiCrAlY, NiCr) and ceramic coatings (ZrO_2 -8% Y_2O_3 , Al_2O_3) are shown in Figs. 2 and 3, respectively. In addition, the coatings are laminated in the lateral direction of the figures, and the longitudinal directions of specimens are the vertical direction of the figures. CoNiCrAlY (APS) and NiCr coatings deposited by APS have the lamination structures with oxides which look black in the figures. On the other hand, oxides aren't recognized and defects are few in the CoNiCrAlY (LPPS) coating deposited by LPPS. Next, an Al_2O_3 coating is fine with relatively few defects, while a ZrO_2 -8% Y_2O_3 coating has long defects parallel to the longitudinal direction.

2.2 Measurement method of residual stress

Measurement of a residual stress was carried out by the SACHS method⁽¹⁰⁾. The stress was calculated from the surface strain relieved when a substrate was removed. In order to measure the strain relieved, a 2-axis foil strain gauge was applied to the center of a coating shown in Fig. 1. The strains in the longitudinal direction, z , and the circumferential direction, θ , were measured using the strain gauge. The gauge length was selected as 1 mm. The relaxation procedure of a stress is as follows: In the case of a mild steel substrate, a hole with a diameter of 6 mm in the substrate was made by a lathe. Subsequently, the mild steel cylinder with the remaining thickness of 2 mm

Table 1 Plasma-spraying powders

	Al_2O_3	ZrO_2 -8% Y_2O_3	NiCr	CoNiCrAlY
Chemical composition (mass%)	98% Al_2O_3	ZrO_2 , 8% Y_2O_3	Ni, 20%Cr	Co, 32%Ni, 21%Cr, 8%Al, 0.5%Y
Powder size (μm)	15~45	45~75	45~106	5~37

Table 2 Plasma-spraying conditions

	APS Metal alloy coating	APS Ceramics coating	LPPS Metal alloy coating
Pressure (MPa)	0.1	0.1	0.013
Arc current (A)	500	850	500
Arc voltage (V)	60	35	60
Arc	Ar (MPa)	0.69	0.41
	He (MPa)	—	0.62
	H ₂ (MPa)	0.41	—
Spraying distance (mm)	150	100	360

Fig. 2 Microscopic observations in the longitudinal-section of metallic coatings

(a) $\text{ZrO}_2\text{-}8\%\text{Y}_2\text{O}_3$

(b) Al_2O_3

Fig. 3 Microscopic observations in the longitudinal-section of ceramics coatings

Fig. 4 Relation between relieved strain and inner diameter of a substrate

was dissolve out by chemical-polishing solution ($\text{HNO}_3:\text{H}_2\text{O}=1:1$). In the case of a SUS304 substrate, all the substrate was removed using a lathe and grinder, because a foil strain gage was spoiled by hydrochloric acid which could dissolve out the stainless steel. Residual stresses, σ_θ and σ_z , are given by the following equation^{(1),(10)}, where ε_θ and ε_z represent a longitudinal strain and circumferential strain, respectively.

$$\begin{aligned}\sigma_z &= -E \frac{\varepsilon_z + \nu \varepsilon_\theta}{1 - \nu^2} \\ \sigma_\theta &= -E \frac{\varepsilon_\theta + \nu \varepsilon_z}{1 - \nu^2}\end{aligned}\quad (1)$$

An example of the relationship between the relieved strain and a diameter of the removed substrate is shown in Fig. 4. It is found that the strain is unchanged after the substrate was lathed by a diameter of 6 mm. Subsequently, the strain is gradually relieved with removal of a substrate.

3. Test Results and Discussions

3.1 Measurement of mechanical properties of plasma sprayed coatings

Both the coefficients of linear expansion and Young's moduli of sprayed coatings were measured, because these values were necessary in calculation of residual stresses. In order to measure the coefficient of linear expansion of a sprayed coating, a coating independent of a substrate was manufactured. The manufacturing procedure of the specimen is shown in Fig. 5(a) - (c). First, a mild steel cylinder shown in Fig. 5(a) was prepared. Next, a coating was sprayed on the mild steel as shown in Fig. 5(b) after blast treatment by alumina grid (WA #24). A cylindrical sprayed coating specimen as shown in Fig. 5(c) was obtained by dissolving out only the mild steel cylinder with chemical-polishing solution ($\text{HNO}_3 : \text{H}_2\text{O} = 1 : 1$). The thicknesses of the coatings were $300\text{ }\mu\text{m}$. The sprayed coating was heated, and the surface strain which correspond to be linear expansion strain was measured during a heating test using a laser speckle strain measurement gauge (SSDG)⁽¹¹⁾⁻⁽¹³⁾. Heating was carried out by high frequency induction heating. A ceramic coating was secondary heated by inserting a stainless steel column with a diameter of 10 mm into the specimen. The thermostable black paint (the emissivity was 0.93) was applied to near the strain measurement area, and the temperature was measured using a radiation thermometer. The relationship between temperature and strain of a sprayed coating is shown in Fig. 6. The measurement results of SUS304 and SS400 substrates are also shown in Fig. 6. The linear expansion strains of substrates (SUS304, SS400) are large, and those of ceramic coatings (Al_2O_3 , $\text{ZrO}_2\text{-}8\%\text{Y}_2\text{O}_3$) are small, and those of bond coatings (NiCr, CoNiCrAlY) are the middle values between those of the ceramics coatings and those of the substrates. The coefficients of linear expansion (average value of 573 K - 773 K) of these coatings are shown in Table 3. In addition, these values are comparable to the values of bulk materials. Next, the coating independent of a substrate was manufactured, and Young's modulus was measured. A cylindrical single axis specimen with a thickness of

Fig. 5 Procedure of specimen manufacturing

$300\text{ }\mu\text{m}$ was manufactured, and the Young's modulus was determined by a slope of stress-strain curve. Details, the manufacturing method of a specimen and the measurement method of a stress-strain relation, were related in other work⁽⁷⁾. The measurement results at room temperature are shown in Table 3.

Young's moduli of sprayed coatings were small as compared with the bulk materials. In the case of $\text{ZrO}_2\text{-}8\%\text{Y}_2\text{O}_3$ and Al_2O_3 ceramics coatings, Young's moduli were about 10% of those of bulk materials, in the case of NiCr and CoNiCrAlY (APS) metallic coatings, those were about 25%, in the case of CoNiCrAlY (LPPS) coating, it was about 50%. Thus, Young's modulus was a very small value as compared with a bulk material, though the coefficient of linear expansion was a comparable value. The mechanical properties, such as a Young's modulus, were dependent on the microstructure. Consequently, a Young's modulus of a coating with many pores and defects was small. On the other hand, a coefficient of linear expansion depends on only the distribution form of the potential energy between molecules. Consequently, the coefficient was comparable to a bulk material.

3.2 Measurement results of residual stresses

The residual stresses of sprayed coatings (Al_2O_3 , $\text{ZrO}_2\text{-}8\%\text{Y}_2\text{O}_3$, NiCr, CoNiCrAlY) on SUS304 and the mild steel substrates were measured. The average elastic moduli, E , and the average coefficients of

Table 3 Elastic moduli and linear expansion coefficients

Material		E (GPa)	$\alpha : 573\text{K} \sim 773\text{K} (10^{-6}/\text{K})$
Substrate	Mild steel	200	14.9
	SUS304	200	17.8
Sprayed coating	NiCr	50	14.0
	APS CoNiCrAlY	50	12.4
	LPPS CoNiCrAlY	100	12.1
	Al_2O_3	40	8.5
	$\text{ZrO}_2\text{-}8\%\text{Y}_2\text{O}_3$	20	9.5

Fig. 6 Relation between temperature and thermal expansion strain

linear expansion, α , shown in Table 3 were used for calculation of the residual stresses in this study, though the half-molten particles, in fact, impacted to a substrate and a coating was gradually laminated. The Poisson's ratio, ν , was assumed to be 0.3. The measurement results of residual stresses are shown in Fig. 7(a) and (b). The value of residual stress in the longitudinal direction, σ_z , is approximately similar to that in the circumferential direction, σ_θ , though dispersion of the data is large. This results were the same as the report⁽¹⁾ for NiCr and ZrO_2 -20% Y_2O_3 by the X-ray stress measurement method. Each residual stress of the sprayed coating is tensile stress as shown in Fig. 7. Half-molten sprayed particles impact and adhere to a substrate, and are cooled by the substrate, and form a coating. Since shrinking of the sprayed coating by this cooling is restrained by the substrate, a thermal stress which is a residual stress arises in the sprayed coating. The coefficients of linear expansion of substrates are larger than those of sprayed coatings

as shown in Table 3. This is the factor of arising a compressive residual stress, since the substrate will shrink greatly during the cooling process if the temperature changing range of a substrate is the same as that of a coating. However, in fact, the temperature at which thermal stress begins in the coating is understood to be considerably higher than that of a substrate, since a sprayed coating is cooled down from at a high temperature in a half-molten state. This is the reason that tensile residual stresses have arisen in the sprayed coatings.

Next, the residual stresses on SS400 substrates are compared with those on SUS304 substrates shown in Fig. 7(a) and (b). The tensile residual stress of a sprayed coating on a SUS304 substrate is preceived to be smaller than that on a SS400 substrate, since SUS304 shrinks greatly during a cooling process because of a large coefficient of linear expansion. In the case of metallic coatings (NiCr, CoNiCrAlY), residual stresses on SUS304 substrates are smaller than that on SS400 substrates according to expectation as shown in Fig. 7. On the other hand, in the case of ceramics coating (Al_2O_3 , ZrO_2 -8% Y_2O_3), the values on both SUS304 and SS400 substrates are less than 40 MPa, and the value on a SS400 substrate is oppositely smaller. It was probable that many micro cracks occurred and the stress was relaxed during a cooling and solidification process since the ceramic coating was vulnerable, consequently the large tensile thermal stress was relaxed. The residual stresses of ceramics coatings on SUS304 substrates are about 30–40 MPa. It was considered that larger stresses were applied to ceramics coatings on SS400 substrates than those on SUS304 substrates, consequently cracks occurred and the residual stresses were relaxed. Which was correspond to the fact that tensile strengths of Al_2O_3 and ZrO_2 -8% Y_2O_3 coatings were less than about 30 MPa^{(8),(13)}. Moreover, the similar results that residual stresses of Al_2O_3 and ZrO_2 -20% Y_2O_3 coatings on mild steel and SUS304 substrates was less than 25 MPa was reported^{(1),(5)}.

Next, it was found that a residual stress of a CoNiCrAlY (LPPS) coating is larger than that of a CoNiCrAlY (APS) coating as shown in Fig. 7(a) and (b). Itoh et al.^{(2),(3)} carried out X-ray stress measurements of CoNiCrAlY coatings, and reported that the residual stress of a CoNiCrAlY (LPPS) coating was about 2 times larger than that of a CoNiCrAlY (APS) coating. A Young's modulus of a LPPS coating is about twice that of an APS coating shown in Table 3, details will be described later, which is understood to be the dominant reason of the difference of the residual stress in the two coatings.

Finally, the reported residual stresses measured

(a) SUS304 substrate

(b) SS400 substrate

Fig. 7 Measured residual stresses of plasma sprayed coatings

Fig. 8 Comparison between measured values by the SACHS method and reported values by the X-ray method

by the X-ray method were compared with those measured mechanically in this study. Figure 8 shows the comparison between the values quoted from references^{(1),(4),(5)} and the measurement values in this study. There is a tendency that the values measured mechanically are larger than those measured using the X-ray method, though the comparison can't be strictly performed under different spraying conditions. Noutomi et al.⁽¹⁾ reported that a residual stress of a Ni-50Cr coating measured mechanically was about 1.3 times larger than that using the X-ray method, and Itoh et al.⁽³⁾ reported the similar result for a CoNiCrAlY (APS) coating. The X-ray method was measuring the microscopic lattice strain (X-ray penetration depth was 30 - 50 μm from the surface), while SACHS method was measuring the average strain of whole the coating. The difference in a measurement area was understood to be the dominant reason of the difference of the measured values.

3.3 Arising mechanism of residual stresses

The arising mechanism of residual stress was examined quantitatively. Spraying particles impacted and adhered in a half-molten state to a substrate, and were cooled by the substrate, as described in the previous section. Shrinking of a sprayed coating caused by the cooling was restrained by the substrate. Consequently, a thermal stress which was a residual stress arose in the sprayed coating. Temperature at which a thermal stress begins to arise is defined as T_c . In order to simplify the understanding of influence factors on a residual stress, following assumptions are used:

(1) Young's moduli and the coefficients of linear expansion of both a sprayed coating and a substrate were constant between high temperature and room

Table 4 Temperature of coating, T_c [K], at $T_s=523$ K

	SUS304	SS400
Al_2O_3	829	-
$\text{ZrO}_2\text{-}8\text{Y}_2\text{O}_3$	874	-
NiCr	676	691
CoNiCrAlY (APS)	706	695
CoNiCrAlY (LPPS)	791	803

temperature, T_0 , though these actually depended on temperature.

(2) A coating could be deposited at once, though the coating was, in fact, gradually formed by deposition of sprayed particles.

(3) In calculation of a thermal stress, the linear model of the coating on a column substrate was considered: a plane strain state (on the cross-section, summation force was zero in the longitudinal direction, furthermore the strain of a coating was same as that of a substrate) was assumed.

Under the above assumptions, a residual stress, σ_R , in the longitudinal direction is equal to that in the circumferential direction at the sufficiently distant region from an edge, and they are given in the following equation^{(1),(4)}.

$$\sigma_R = \frac{E_c E_s A_s}{(E_c A_c + E_s A_s)(1-\nu)} \{ \alpha_c (T_c - T_0) - \alpha_s (T_s - T_0) \} \quad (2)$$

A represents a cross-sectional area, and subscripts C and S represent a coating and a substrate, respectively. T_s represents the temperature of a substrate when the temperature of a sprayed coating becomes T_c .

σ_R which depends on both α and T is understood by Eq. (2) to become both a tensile and a compressive stress. α_s used in this study is larger than α_c as shown in Table 3, which is the factor of arising a compressive residual stress. However, T_c was understood to be quite higher than T_s . Consequently, a tensile residual stress arose in the coating. The temperature of the substrate, T_s , just after thermal spraying was measured, and it was about 523 K. T_c can be calculated from σ_R , E and α under the assumption that $T_s=523$ K. Those values are shown in Table 4. However, the values of the ceramic coatings (Al_2O_3 , $\text{ZrO}_2\text{-}8\%\text{Y}_2\text{O}_3$) on SS400 substrates are excluded, because the stresses of the coatings were relaxed due to many micro cracks maybe occurred during a cooling and solidification process. T_c of the ceramic coatings (Al_2O_3 , $\text{ZrO}_2\text{-}8\%\text{Y}_2\text{O}_3$) in Table 4 deposited with the same spraying parameters are approximately the same, and T_c of APS alloy coatings (NiCr, CoNiCrAlY) are approximately the same, too. Ishida et al.⁽⁴⁾ measured the temperatures of a SUS304 plate substrate and a NiCr coating during a spraying process. They reported that the temperature of a sprayed

Fig. 9 Relation between α_c/α_s and $(T_c - T_0)/(T_s - T_0)$

coating, T_c , was 673 ± 150 K, and the temperature of a substrate, T_s , was 533 K. Comparison couldn't be strictly performed because spraying conditions were different each other, however it seemed that the temperature of the coating calculated from the residual stress in this study was appropriate because the value in Table 4 ($T_c = 676$ K and $T_s = 523$ K when thermal spraying of NiCr was carried out on a SUS304 substrate) was approximately the same as the measurement value mentioned above.

A tensile residual stress may cause cracks and a delamination of a coating. Next, conditions for not arising a tensile residual stress in a coating were discussed. The condition is that the following equation which means that inside of the large parenthesis in Eq. (2) is smaller than zero is able to be satisfied.

$$\frac{\alpha_c}{\alpha_s} \cdot \frac{(T_c - T_0)}{(T_s - T_0)} \leq 1 \quad (3)$$

The relationship between the ratio of the coefficient of linear expansion, α_c/α_s , and the ratio of the temperature range, $(T_c - T_0)/(T_s - T_0)$, is shown in Fig. 9. In the figure, the value acquired in this study shown in Table 4 is also plotted. A tensile residual stress arises in an upper region of the curve in the figure, on the other hand, a compressive residual stress arises in a lower region. It is found that all spraying parameters in this study are the conditions under which a tensile residual stress arises. In order to reduce a tensile residual stress, it is effective to reduce either α_c/α_s or $(T_c - T_0)/(T_s - T_0)$. α_c/α_s is a mechanical property, T_c is the temperature at which thermal stress of a coating arises. These values are difficult to control. Consequently, it is understood that increase of T_s , pre-heating of a substrate, is effective to reduce a tensile residual stress. T_s was calculated from T_c shown in Table 4 under $\sigma_R = 0$. The calculated values are shown in Table 5. In the case of these coatings, it is expected that a residual stress is reduced by pre-heating of a substrate up to about 523 K – 723 K as

Table 5. Pre-heating temperature of substrate, T_s [K], at $\sigma_R = 0$

	SUS304	SS400
Al_2O_3	552	-
$\text{ZrO}_2\text{-}8\text{Y}_2\text{O}_3$	606	-
NiCr	595	668
CoNiCrAlY (APS)	582	628
CoNiCrAlY (LPPS)	633	708

shown in Table 5.

Next, the influence of the coefficient of linear expansion of a substrate was discussed. A tensile residual stress on a SUS304 substrate is understood by Eq. (2) to be smaller than that on a SS400 substrate, namely, a tensile residual stress is small when α_s is large, which means physically that a substrate shrinks greatly. In the case of metallic coatings (NiCr, CoNiCrAlY) with little influence of the stress relaxation by occurrence of a micro crack, it can be confirmed that a tensile residual stress on a SUS304 substrate is smaller than that of a SS400 substrate as related in the previous section.

Next, the influence of linear expansion coefficient in a coating, α_c , was discussed. It is understood by Eq. (2) that a tensile residual stress becomes smaller when α_c is small. If APS metallic coatings (NiCr, CoNiCrAlY) which is deposited with the same spraying parameters and have the same Young's moduli are compared, residual stresses of CoNiCrAlY (APS) coatings with small coefficients of linear expansion on both SUS304 and SS400 substrates are smaller than those of NiCr coatings as shown in Fig. 7(a) and (b).

Finally, the influence of a Young's modulus was discussed. It is understood by Eq. (2) that a tensile residual stress becomes small when E_c is smaller. It is found that the residual stress of CoNiCrAlY (APS) is smaller than that of CoNiCrAlY (LPPS) as shown in Fig. 7(a) and (b). The dominant reason of the difference of residual stresses was understood that the Young's modulus of a CoNiCrAlY (APS) coating was 1/2 of that of a LPPS coating.

4. Conclusions

The residual stresses of metallic sprayed coatings (NiCr, CoNiCrAlY) and ceramics sprayed coatings (Al_2O_3 , $\text{ZrO}_2\text{-}8\text{Y}_2\text{O}_3$) were measured by the SACHS method (the stress relaxation method). The results obtained are summarized as follows:

(1) Each residual stress of a coating was tensile, because the temperature of the coating at which a thermal stress began to arise was higher than that of a substrate.

(2) The residual stresses of ceramics sprayed

coatings (Al_2O_3 , ZrO_2 -8% Y_2O_3) were very small, and less than about 40 MPa.

(3) In the case of the metallic coatings (NiCr, CoNiCrAlY), the tensile residual stresses on mild steel substrates with small coefficients of linear expansion, T_s , were larger than those of stainless steel substrates.

(4) The residual stresses of low pressure plasma sprayed coatings were larger than those of atmospheric plasma sprayed coatings.

(5) Pre-heating of a substrate is effective to decrease a tensile residual stress, and the temperature conditions are proposed.

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