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ARTICLE

Effect of Atomization Gases on the Elastic Modulus of Thermal-sprayed NiCr Coatings

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Abstract: Accurate evaluation of the elastic modulus of thermal-sprayed coatings is very important to ensure the safety and durability of industrial components, but it seems difficult via a conventional technique. Although the indentation technique is available to estimate the local modulus of coatings, it does not reflect the effects of porosity or cracks. Impulse excitation technology (IET) is a simple and accurate method to evaluate the elastic modulus of bulk materials. It was used to determine the modulus of thermal-sprayed coatings via relative method in this work. An analytical relationship among the moduli of the coating, the substrate and the coating/substrate system was derived. Thus, the modulus of coatings was obtained via the measured modulus of the samples before and after coating. Experiments on Q235 steels coated with NiCr coatings, were carried out to investigate the effect of atomization gases on the elastic modulus of NiCr coatings. The results indicate that the elastic modulus of NiCr coating prepared by air atomization (NiCr-Air) is higher than that of NiCr coating prepared by N_2 gas atomization (NiCr-N_2). The reasons for this are the formation of Ni(Cr_2O_4) reinforced NiCr alloy laminated composite, and the laminated composite structure has a positive influence on the elastic modulus.

Key words: elastic modulus; impulse excitation technology; relative method; NiCr coatings; atomization gases

Thermal-sprayed coatings have been used in many different industrial fields to provide excellent performance on the resistance of erosion, oxidation, corrosion, wear and heat[1-5]. Some properties of the coating/substrate system, such as the adhesion strength, residual stress, and contact stress field, cracking and spalling of the coatings are highly dependent on the value of elastic modulus[6-8]. Accurate evaluation of the elastic modulus of thermal-sprayed coatings is in great need to ensure the safety and durability of modern machinery and to optimize the design of the coating/substrate system.

The coating materials need to be heated in a molten or superheated state in the thermal spray process, and these liquid particles are accelerated to the substrate surface by an atomization gas[9]. The atomization gas property is crucial to the in-flight oxidation of spraying materials, and many authors have reported the effect of the oxidation reaction on the adhesive strength, the corrosion and wear resistance of the coatings[10,11]. However, there is little information available in literature about the effect of atomization gases on the elastic modulus of the thermal-sprayed coatings to the author's knowledge. And the mechanical properties of thermal-sprayed coatings are the essential prerequisites of their function realization, so it is very important to evaluate their elastic modulus accurately. In the previous researches, the elastic modulus of thermal-sprayed coatings was measured by indentation[8,12-14]. But the indentation method could only obtain the local properties that are often different from the practical properties for the nonhomogeneous materials. The thermal-sprayed coatings often contain the pores and secondary phases, so the indentation results are not equal to the practical modulus of the coatings. Therefore it is necessary to develop feasible approaches to estimate the elastic modulus of the thermal-sprayed coatings fast and accurately.

The impulse excitation technology (IET) is one of simple, fast and accurate methods to evaluate the modulus of bulk materials, without any expensive experimental devices [15,16].

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The IET has found world-wide acceptance both in industry and research [17], and it could be applied at different circumstances such as high temperature, low temperature and controlled humidity, in which other methods seem not so convenient. Obviously, it will be significant if the elastic modulus of coatings could be determined by IET, but this technique is unavailable to evaluate the coatings' modulus directly. Because the coatings are usually thin and it is very difficult to pull off the coatings from the substrates. Nevertheless, IET can be used to determine the elastic modulus of coated samples and substrates easily. So the relative method [18] can be considered as a solution to gain the coatings' modulus which cannot be measured directly.

This paper extends the IET to evaluate the elastic modulus of thermal-sprayed coatings combined with a relative method, and the method was named as the relative IET. Analytical relationship among the modulus of coating, substrate, and coating/substrate system was presented, so that the modulus of the coating can be obtained simply from the properties of the coated and uncoated samples. Additionally, the three-point bending test combined with the relative method [18, 19] is used to verify the correctness of the relative IET method. In this work, Q235 steel coated with NiCr coating which is used on structural alloys in energy conversion and utilization systems to protect surface from oxidation and erosion [20,21], deposited by high velocity electric arc spraying (HVAS), was used. And the results indicate that: i) the comparison of the measured elastic modulus obtained by relative IET and three-point bending test showed that the relative IET was valid and reliable to evaluate the modulus of coatings; ii) the elastic modulus of NiCr coating prepared by air atomization is higher than that of NiCr coating prepared by N_2 gas atomization.

1 Principle

For IET, the elastic modulus of a rectangular bar can be determined by [16, 22-25],

$$E = 0.9465\left(\frac{mf_{\mathrm{f}}^2}{b}\right)\left(\frac{L^3}{t^3}\right)T_1 \tag{1}$$

where E is the elastic modulus of the specimen in Pa, m is the mass of the specimen in g, f_{f} is the fundamental flexural resonant frequency in Hz, b, L and t are the width, length and thickness of the specimen in mm, respectively, and T_1 is the correction factor for fundamental flexural mode to account for finite thickness of bar, Poisson's ratio, ν, etc.

$$T_1 = 1 + 6.585(1 + 0.0752v + 0.8109v^2)\left(\frac{t}{L}\right)^2 - 0.868\left(\frac{t}{L}\right)^4 - \left[\frac{8.340(1 + 0.2023v + 2.173v^2)\left(\frac{t}{L}\right)^4}{1.000 + 6.338(1 + 0.1408v + 1.536v^2)\left(\frac{t}{L}\right)^2}\right] \tag{2}$$

A value of v=0.3 was used in estimating T_1 for plastic materials, such as metals, to obtain a simplified expression.

$$T_1 \approx 1 + 7.2141\left(\frac{t}{L}\right)^2 \tag{3}$$

Substituting Eq.(3) into Eq.(1), the elastic modulus measured by IET is expressed as:

$$E = 0.9465\left(\frac{mf_{\mathrm{f}}^2}{b}\right)\left(\frac{L^3}{t^3}\right)\left[1 + 7.2141\left(\frac{t}{L}\right)^2\right] \tag{4}$$

So the modulus of substrates and coating-substrate systems could be obtained easily by Eq.(4), and the elastic modulus parallels the length of the prismatic tested specimens.

But the elastic modulus of coatings cannot be measured by IET directly, so the relative method[18, 26] is considered to solve this issue. The relative method is an indirect method and its key issue is to establish the theoretical relationship between the known and unknown parameters. In this work, the elastic modulus of substrate was defined to be E_{s}, the equivalent elastic modulus of the coated sample be E_{q}, and the elastic modulus of the coating be E_{c}. IET was used to determine the E_{q} and E_{s} in this work because of its good repetition, high accuracy and simple operation. Then the theoretical formula between E_{c}, E_{q} and E_{s}, $E_{\mathrm{c}}=f(E_{\mathrm{q}},E_{\mathrm{s}})$, should be established. And the E_{c} could be calculated by plugging E_{q} and E_{s} into $E_{\mathrm{c}}=f(E_{\mathrm{q}},E_{\mathrm{s}})$.

The single coated specimen is schematically shown in Fig.1. The thickness of the coating and substrate were h and H, respectively, and a was the distance from the neutral axis to the coating-substrate interface. In this work, perfect interfacial-bonding was assumed for the stress translation between the coating and substrate.

When the single coated beam specimen was subjected to a pure bending, the bending stress in the cross section is shown in the Fig.2, where, σ_{s} is the bending stress of substrate, and σ_{c} is the bending stress of coating.

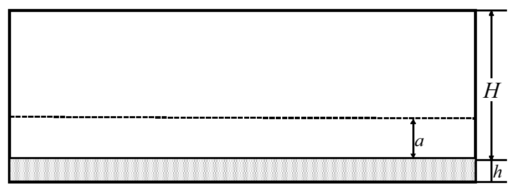

Fig.1 Schematic illustration of cross-section of single coated beam specimen

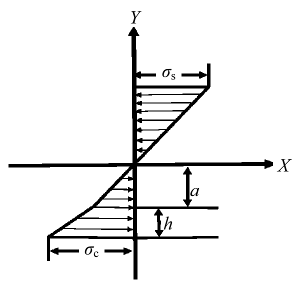

Fig.2 Distribution of the bending stress along the thickness

The σ_s and σ_c can be calculated by mechanics of materials [27], and they have the form

$$\sigma_s = E_s \frac{y}{R} \quad (5)$$

$$\sigma_c = E_c \frac{y}{R} \quad (6)$$

Where R is defined to be the radius of curvature of the neutral axis. From equilibrium of the axial force, the following equation has to be satisfied:

$$\int_{-a}^{H-a} \sigma_s b\mathrm{d}y + \int_{-a-h}^{-a} \sigma_c b\mathrm{d}y = 0 \quad (7)$$

Substituting Eq. (5) and (6) into Eq. (7), the equilibrium equation of axial force can be expressed as the following equation:

$$\int_{-a}^{H-a} E_s \frac{y}{R} b\mathrm{d}y + \int_{-a-h}^{-a} E_c \frac{y}{R} b\mathrm{d}y = 0 \quad (8)$$

Where b is the width of the beam sample, and a can be obtained from Eq. (8).

$$a = \frac{E_s H^2 - E_c h^2}{2(E_s H + E_c h)} \quad (9)$$

The elastic modulus of coating-substrate composite system, E_q, can be determined by IET, so the bending moment of the section can be calculated by the pure bending theory.

$$M = \frac{EI}{R} = \frac{E_q b(H+h)^3}{12R} \quad (10)$$

Where I is the inertia moment of the section for the single coating specimen, and the bending moment can be divided into two parts:

$$M = \int_{-a}^{H-a} yE_s \frac{y}{R} b\mathrm{d}y + \int_{-a-h}^{-a} yE_c \frac{y}{R} b\mathrm{d}y = 0 \quad (11)$$

Combining Eq. (9), (10) and (11), a simplified relation is derived by

$$AE_c^2 + BE_c + C = 0 \quad (12)$$

Where $A = h^4$, $B = 4E_sHh^3 + 4E_sH^3h + 6E_sH^2h^2 - E_q(H+h)^3h$, $C = E_s^2H^4 - E_qE_sH(H+h)^3$. And the E_q can be obtained by Eq. (4), E_s can be determined by materials handbook or evaluated by Eq. (4). So the E_c can be determined by the following equation.

$$E_c = \frac{\sqrt{B^2 - 4AC} - B}{2A} \quad (13)$$

Obviously, the value of E_c can be determined uniquely by the sample size and both the elastic moduli of substrate and the coated sample. Also the three-point bending tests by relative method [18, 19] were used to prove the correctness and reliability of the testing method by IET.

The sizes including the length, width, thickness of coating and substrate, and the mass of the specimen should be measured before the tests. The experiments for determining the elastic modulus of the coatings by IET were performed in the following steps: 1) measure the modulus of the substrate by IET or by checking the materials handbook, 2) measure the modulus of the coated specimen by IET, and 3) calculate the values of E_c by Eq. (13).

2 Materials and Experimental Process

Commercially available Ni-20%Cr wire was used as feedstock alloys in the study. The HVAS system (TLAS-400C, Shaanxi Tongli Electric Co., Ltd) was employed to obtain the NiCr coatings on the Q235 steel substrate. The spray parameters for NiCr coatings were shown as follows: 30~32 V of arc voltage, 180~220 A of arc current, 0.7 MPa of gas pressure, 200 mm of the stand-off distance, and air and nitrogen gas was selected as the atomization gases. The NiCr coating samples produced by air atomization were defined as NiCr-Air, while the NiCr coating samples produced by N_2 gas atomization were defined as NiCr-N_2. In addition the substrate was grit-blasted before the preparation of the coatings, and the parameters of grit blasting were displayed as follows: 250 μm of brown fused alumina, 0.4~0.6 MPa of blasting pressure, 45° of inclination angle, and 160~180 mm of stand-off distance. After the grit blasting, it was dipped into acetone and cleaned with ultrasonic washer for 5 min to remove the surface impurities.

Before the IET testing, the coated samples and Q235 steel substrates should be cut into the size of 55 mm×4 mm×3.5 mm. The mass of the samples were measured by an electronic balance with the precision of 0.0001 g. Then the fundamental resonant frequency of the coated samples and Q235 steel substrates could be measured by the impulse excitation testing system (RFDA-MF, IMCE), and the elastic modulus of coated samples and substrates can be calculated by Eq. (4). Then, the thickness of coating was measured with the digital microscope (VHX-600, KEYENCE). Finally, the elastic modulus of coatings can be calculated by substituting the geometrical size, and the measured E_q and E_s into Eq. (13).

Furthermore, the three-point bending test combined with the relative method was used to verify the correctness and reliability of the relative IET. The three-point bending test was performed via universal testing machine (MTS Criterion C45, MTS Systems Corporation). The loading rate was controlled by the cross beam displacement and set as 0.2 mm/min. The support span was 40 mm and the upper limits of the load were 300 N. At least five samples should be tested to obtain an average testing data.

The phases and crystalline structures of the NiCr coatings were investigated using an X-ray diffractometer (D8 ADVANCE, Brucker Corporation). The polished cross-sections of the NiCr coating samples were investigated using a scanning electron microscope (S-4800, Hitachi Limited) employing secondary and backscattered electron radiation. And the local elemental analyses were performed by an energy dispersive spectroscopy (EDS).

3 Results and Discussion

3.1 Validity of the relative IET

Q235 steel beam samples coated with NiCr coatings (350~450 μm in thickness) were used in this work. Fig.3 shows the cross-section of the NiCr-Air coating samples which were produced by air atomizing. As shown in Fig.3, the interface between coating and substrate is clear and tightly-bonded.

The specific mechanical resonance frequencies of the Q235 steel bar were tested by IET, and the elastic modulus of the substrates, ~218.96 GPa, was calculated by Eq. (4). Five NiCr-Air samples were used to determine the modulus of the coatings, and E_c could be calculated by Eq.(13) (as shown in Table 1). Also the five NiCr-Air coating samples could be used in the three-point bending tests, because the impulse excitation tests are nondestructive. And the elastic modulus of the Q235 steel, ~196.53 GPa, was got using the three-point bending tests. The three-point bending measurements of the elastic modulus of the five NiCr-Air coating samples are also shown in Table 1.

The measured elastic moduli of the NiCr-Air coatings are 86.73 GPa and 88.09 GPa via the relative IET and three-point bending test, respectively. The measurement results show few differences between these two methods. It reveals that the new method of relative IET is valid and reliable to determine the elastic modulus of coatings. Moreover, the new method using IET is nondestructive, and the testing equipment is very simple. It demonstrates that the new method is very suitable for the elastic modulus measurements of coatings because of its convenience and efficiency.

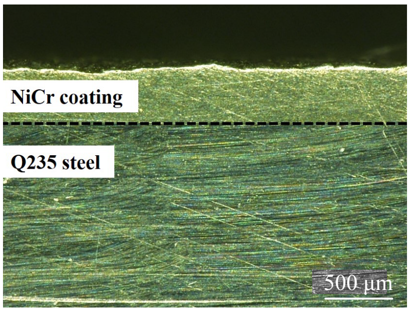

Fig.3 Cross-section of the NiCr-Air coating sample

Table 1 Test results of modulus of NiCr-Air coatings (GPa)

Relative IET			Three-point bending test		
E_s	E_q	E_c	E_s	E_q	E_c
218.96	173.66	83.10	196.53	161.35	89.84
218.96	174.81	84.89	196.53	160.31	85.77
218.96	176.95	91.09	196.53	162.99	93.58
218.96	176.97	93.32	196.53	160.26	87.70
218.96	173.04	81.25	196.53	159.16	83.58
		86.73+4.67[#1]			88.09+3.44[#2]

[#1]: mean of E_c for relative IET; [#2]: mean of E_c for three-point bending

3.2 Influence of atomization gases on the elastic modulus of NiCr coatings

The relative IET was used to determine the elastic modulus of NiCr-Air and NiCr-N_2 coatings, and the measured elastic modulus of the NiCr-Air and NiCr-N_2 coatings are 86.73± 4.67 GPa and 77.02 ± 5.56 GPa, respectively. It demonstrates that the modulus of NiCr-Air is greater than that of NiCr-N_2.

To explore the mechanism of the difference between the modulus of NiCr-Air and NiCr-N_2 coatings, the microstructure and phase composition were investigated by SEM and XRD. The interface between the NiCr coatings and Q235 steel was observed by SEM, with the results of linear scanning of elements displayed in Fig.4. It indicates that the oxygen content of NiCr-Air coating is obviously higher than that of the Q235 steel substrate, while the oxygen content of NiCr-N_2 coating is close to that of substrate. The main reason for this is the in-flight oxidation reaction of the NiCr coating when the air is selected as the atomization gas.

Fig.5 shows the differences between the XRD patterns of the NiCr-Air and NiCr-N_2 coatings. The XRD patterns of the NiCr coatings show that the $Ni(Cr_2O_4)$ phase is formed in the preparation of NiCr-Air coatings, but its content is very low with the presentation of low characteristic diffraction peaks. The oxygen in the air would react with the NiCr to form $Ni(Cr_2O_4)$ phase during the thermal spraying process, so that the NiCr-Air coatings contain some oxygen (as shown in Fig. 4). And the oxygen is present in NiCr-Air coatings in the form of the oxide of NiCr alloy-$Ni(Cr_2O_4)$. But the reaction time is short, so the level of oxidation is low.

The inspection was photographed in backscattered electron signal mode to provide the information on the sample composition (as shown in Fig.6), and the elemental analysis of the region of interest was performed by EDS. Fig.6 reveals the lamellar structure in the NiCr-Air coating containing some pores. Examination of the microstructure of coatings reveals the presence of three different types of zones: 1) The first zone represented by a-1 and b-1 area is dark and these areas contain primarily pores and cracks. 2) The second zone represented by a-2 area is of gray colour and the elemental analysis shows that atomic percent of O is about 36.25%, and the rest are Ni and Cr. That means the gray zone mainly possesses the oxides of NiCr alloy. 3) The third zone represented by a-3 and b-3 area is white and the EDS testing result reveals Ni and Cr are the major elements, with the atomic percent of O only about 3.82%. Combined with the XRD patterns of the coatings, the gray zone consists of a certain amount of $Ni(Cr_2O_4)$, and the layered composite is formed by the stacking of $Ni(Cr_2O_4)$ layer and NiCr alloy layer.

It has been reported that the elastic modulus of thermal sprayed coatings were dependent on the microstructural conditions [28]. When the air was selected as the atomization gas, the liquefied NiCr alloy would react with the oxygen from air to form $Ni(Cr_2O_4)$ which is a kind of hard and brittle phases. The

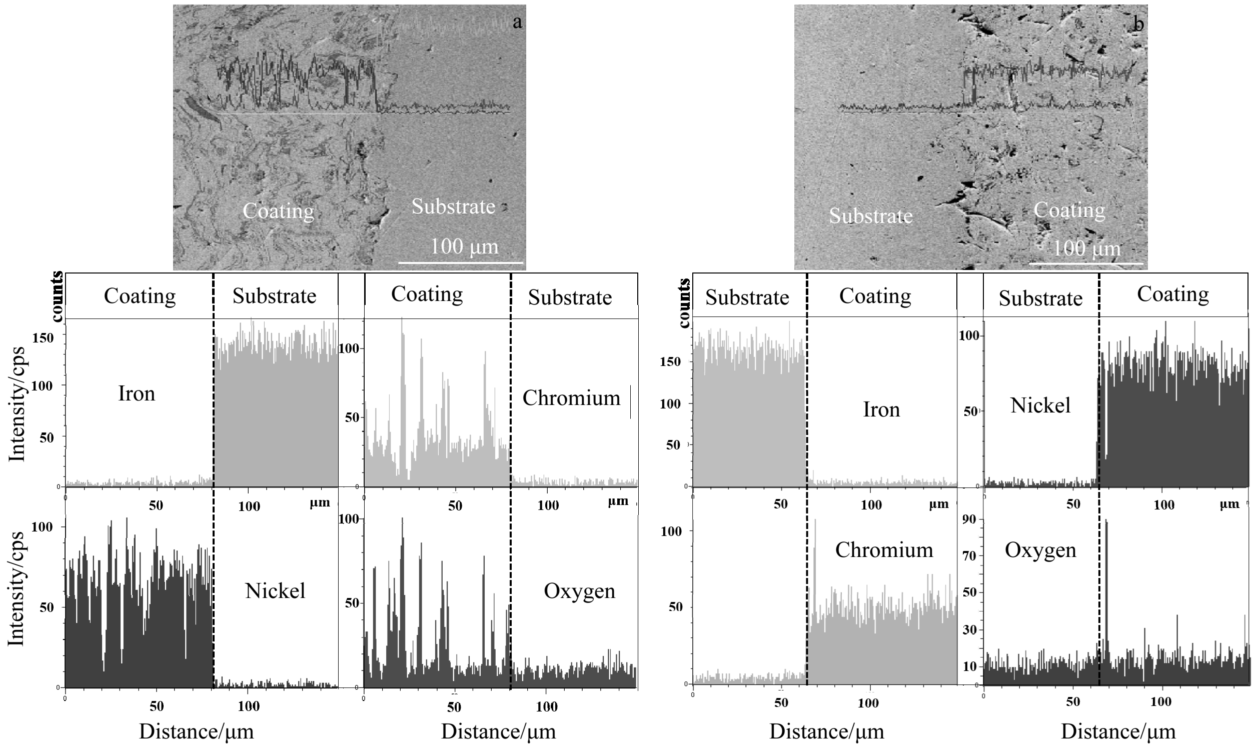

Fig.4 EDS elements linear scanning of the NiCr-Air (a) and NiCr-N_2 (b) coatings

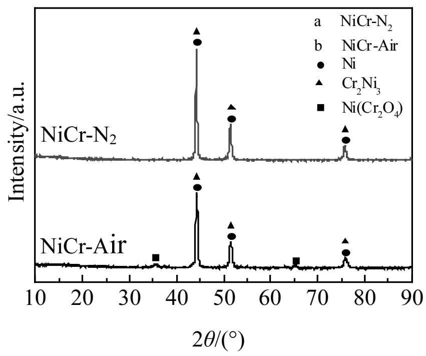

Fig.5 XRD patterns of the NiCr-Air and NiCr-N_2 coatings

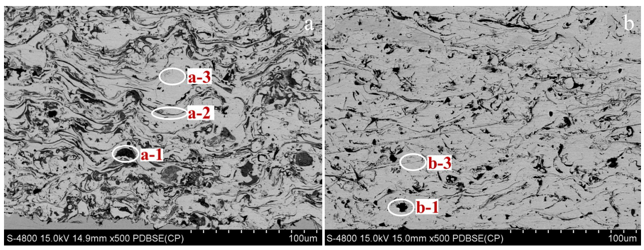

Fig.6 Backscattered electron images of NiCr coatings: (a) NiCr-Air and (b) NiCr-N_2

laminated composite used $Ni(Cr_2O_4)$ as a reinforcing layer material and NiCr alloy as a substrate layer. This would result in the formation of $Ni(Cr_2O_4)$ reinforced NiCr alloy which has the same microstructure as the laminated composites, and this kind of composite structure is conductive to increasing the elastic modulus of NiCr-Air coatings.

The NiCr coatings consist of weak interlamellar boundaries and pores which are generated by the irregular packing of the molten particles. The porosity of NiCr coatings is much higher than that of the bulk material. For this reason, the measured elastic modulus of NiCr coatings (86.73 and 77.02 GPa) are much smaller than that of block Ni-20Cr alloy (218 GPa)[29].

4 Conclusions

1) The elastic modulus of coatings is determined simply by the IET combined with the relative method, as following procedure (3 steps): (i) measure the modulus of the substrate by IET; (ii) measure the elastic modulus of a coated specimen by IET; (iii) calculate the modulus of the coating via the measured values in above two steps based on the theoretical formula derived in this work.

2) By this way, the elastic modulus of coatings could be evaluated under any environments in which the IET is feasible, and the various effects on the modulus can be easily determined due to the convenience of the IET. The elastic modulus of NiCr coatings on Q235 steel substrates is influenced by the atomization gases in the coating process. The measured elastic

modulus of NiCr-Air and NiCr-N_2 coating are 86.73 and 77.02 GPa, respectively, which are much small than that of block Ni-20Cr alloy.

3) The modulus of NiCr-Air is higher than that of NiCr-N_2 because of the formation of $Ni(Cr_2O_4)$ reinforced NiCr alloy laminated composite. The measured elastic modulus is the practical modulus of the coatings with consideration of pores and secondary phase, and can be used to analyze the state of stress and strain of the coatings. This simple testing method provides profitable guidance for the design of substrate/coating systems.

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雾化气体对热喷涂 NiCr 涂层弹性模量的影响

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摘 要：准确评价热喷涂涂层的弹性模量对于确保工业构件的服役安全性与耐久性是十分重要的，但是利用传统技术很难进行评价。尽管纳米压痕技术可以表征涂层局部的弹性模量，但是无法反映出孔隙、裂纹等缺陷对涂层弹性模量的影响。而脉冲激励技术（IET）是一种用于评价块体材料弹性模量的简单、准确方法。结合相对法，利用 IET 可对热喷涂涂层的弹性模量进行评价。首先构建了涂层、基体、涂层/基体复合体系的弹性模量间的解析关系，然后通过测量制备涂层前后试样的弹性模量，即可获得涂层的弹性模量。以 Q235 钢基体/NiCr 涂层为研究对象，研究了雾化气体对 NiCr 涂层弹性模量的影响。结果表明，以空气作为雾化气体制得的 NiCr 涂层（NiCr-Air）的弹性模量高于以 N_2 作为雾化气体制得的 NiCr 涂层（NiCr-N_2）的弹性模量，其原因在于 NiCr-Air 中形成了 $Ni(Cr_2O_4)$氧化物增强 NiCr 合金的层状复合材料，且这种层状复合结构有利于提高 NiCr 涂层的弹性模量。

关键词：弹性模量；脉冲激励技术；相对法；NiCr 涂层；雾化气体

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