Evaluation of the residual stresses in 95wt% Al2O3-5wt% SiC wear protection coating using X-Ray diffraction technique

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Abstract. This paper aims to measuring the residual stresses practically in wear protection coatings using the sin2ψ method according to X-ray diffraction technique. The wear protection coatings used in this study was composite coating 95wt% Al2O3-5wt% SiC, while bond coat was AlNi alloy produced by using flame spraying technique on the mild steel substrate. The diffraction angle, 2θ, is measured experimentally and then the lattice spacing is calculated from the diffraction angle, and the known X-ray wavelength using Bragg's Law. Once the d spacing values are known, they can be plotted versus sin2ψ, (ψ is the tilt angle). In this paper, stress measurement of the samples that exhibit a linear behaviour as in the case of a homogenous isotropic sample in a biaxial stress state is included. The plot of d spacing versus sin2ψ is a straight line which slope is proportional to stress. On the other hand, the second set of samples showed oscillatory d spacing versus sin2ψ behaviour. The oscillatory behaviour indicates the presence of inhomogeneous stress distribution. In this case the X-ray elastic constants must be used instead of Young's modulus (E) and Poisson ratio (ν) values. These constants can be obtained from the literature for a given material and reflection combination. The value of the residual stresses for the present coating calculated was compressive stresses (-325.6758 MPa).

1. Introduction
Growing use of thermal spray coatings, specially for high-temperature environmental resistance, needs assurance in coating durability, i.e. debonding, resistance to cracking, and spallation, both during appliance and in service. Residual stresses are acknowledged to play important roles in coating durability; i.e., tensile residual stresses commonly increase the susceptibility to cracking and debonding. Various studies have been concerned to the measurement of residual stresses in coatings [1-4]. Residual stresses advance during cooling of a thermal spray coating because of the dissimilarity of thermal expansion coefficients of the coating and substrate. Depending on the relative consequences of the thermal expansion coefficients of the coating and substrate [5-7] residual stress can be either tensile or compressive Parameters that heavily affect the value of residual stresses are coating and substrate temperature during spray deposition and properties of the coating such as thickness, roughness and porosity. Experiments have demonstrated that residual stresses increase with coating thickness and deposition temperature [8]. Thermal barrier coatings (TBC) are the finest way to preserve components of gas turbine engines and the demand for this coatings is becoming more vital as higher temperature engines are being advanced [9-14]. Normally, the residual stresses of thermally sprayed coatings are made by diverse mechanisms and sources [15-17]. In a thermal spray procedure
with a high flame temperature, such like plasma spray, flame spray, or arc spray, entirely and partially molten particles striking onto the surface of the substrate, are become flat, solidified, and cooled down in a very small period of time (few microseconds). After their solidification and adhesion onto the surface of the substrate, the shrinkage of the splats can be stalled by substrate material or the underlying solidified coating material, which outcomes in tensile stresses which are called intrinsic, deposition, or quenching stress. Due to a very high temperature difference, a high theoretical residual stress in the order of up to 1 GPa can be made. However, Because of the many relaxation mechanisms, such as micro cracks, the sliding of the splats, plastic deformations, and material creep, the experimentally measured values are much lower (<100 MPa) [18]. X-ray diffraction was used as a corresponding technique; it can measure stress only in a thin surface layer, whereas the penetrating power of neutrons allows through-thickness stress profiling without any material exclusion [19]. This work aims to evaluation of the residual stresses in 95 wt% Al2O3-5wt% SiC wear protection coating using X-ray diffraction technique (XRD).

2. Materials and method

2.1. Materials and parameters of the spraying processes

The coatings were applied by thermal spraying method (flame spraying) in air on the plain-carbon steel (AISI 1050), cylindrical substrate that’s dimensions were 15 mm in diameter and 10 mm in height. The flame spraying system is designed and implemented in the welding laboratory of Mechanical Engineering Department, College of Engineering, University of Diyala, Iraq using spray gun the heat flame is produced by the burning of oxygen and acetylene, where the molten powder is carried out in the gas mixture and is attached to the surface to be coated by the high temperature of the torch which can raise to 3000 °C. It is required to control the pressure of the gases to obtain the flame equal to the speed of the powder rush. The oxygen pressure should be adjusted according to the spray gun used no more than 4 bar and the acetylene pressure not more than 0.7 bar before spraying process. Two layers coatings were used in this work are the bond coat from (AlNi) alloy to reduce of thermal expansion coefficient between substrate and composite ceramic coating as a top coating layer. Conditions of deposition process are listed in Table1.

| Table1. Operating parameters during coating deposition process |
|---------------------------------------------------------------|
| **Operating Parameters** | **Values** |
| Oxygen pressure | 4 bar |
| Acetylene pressure | 0.7 bar |
| Distance | 20 cm |
| Powder feed rate | 7 cm³/min |
| Particle size | Mish (100-300) |
| Temperature substrate | (350 – 400) °C |

2.2. Residual stress analysis

2.2.1. X-ray stress evaluation. XRD-based residual stress measurements were made using standard $d_{\text{spacing}}$ vs. $\sin^2 \psi$ techniques using Shimadzu X-Ray Diffractometer type XRD-6000 and CrKα radiation. The $\sin^2 \psi$ method [20, 21] was used to determine the residual stresses in this work, the change of a lattice plane distance (d spacing) of a phase, i.e., the peak shift of the corresponding reflection, was measured for tilt $\psi$-angles between 0° and 45°. To calculate the residual stresses the linear regression of the plot ($d_{\text{spacing}}$ versus $\sin^2 \psi$ and the X-ray elastic constants. The coating and substrate physical properties (elastic modulus, Poisson’s ratio, and coefficient of thermal expansion), thickness of the top coating, bond coat and substrate are shown in the table 2. The deposition temperature used in the present work during coating process and modeling for the top coating, bond coat and substrate was 850°C.
Table 2. The coating and substrate physical properties [22-25].

|                        | Substrate | Bond coating | Top coating |
|------------------------|-----------|--------------|-------------|
| Young’s modulus (Gpa)  | 200       | 105          | 65.78       |
| Poisson ratio          | 0.33      | 0.315        | 0.231       |
| Thickness (mm)         | 10        | 200μm        | 500μm       |
| Thermal expansion coefficient α, μƐ K | 12.6 | 11.9 | 7.825 |

3. Results and discussion

From Shimadzu X-Ray Diffractometer XRD-6000 chart, will be getting on the following values are shown in the Table 3.

Table 3. Showing 2Θ & ψ to 95%Al₂O₃ & 5%SiC

| 2Θ          | ψ     |
|-------------|-------|
| 1.169565983 | 0     |
| 1.168720831 | 0.06698 |
| 1.168310272 | 0.25  |
| 1.166402633 | 0.5   |

By Brag Law (2d sinθ = nλ) may be calculated (d), where n=1, λ= 2.28970 Å° and Θ (0,15, 30, 45) degree. From the figure1 may be calculated the linear slop of the plot d-spacing versus sin²ψ

Figure 1. Relationship between (sin²ψ) and d-spacing (Å).
The stress can then be obtained from the following equation:

$$\sigma = \frac{E}{(1+\nu)} \frac{1}{d_0} \left( \frac{\partial d}{\partial \sin^2 d} \right)$$

(1)

From figure 1 the slope \( \frac{\partial d}{\partial \sin^2 d} \) = -0.0058421, \( d_0 = 0.95856 \).

From Eq. (1), the value of the residual stresses is: \( \sigma = -325.6758 \) MPa.

3.3.1. Quenching stresses. Quenching stresses are of a level that corresponds to the values determined in the top surface of free standing coatings (Fig. 2a). They are always tensile and roughly constant through the deposit thickness. XRD data of quenching stresses, determined on the surface of different free-standing samples, are shown in Table 3.

![Schematic explanation of residual stresses generation in thermal sprayed coatings](image)

**Figure 2.** Schematic explanation of residual stresses generation in thermal sprayed coatings [26].

3.3.2. Cooling stresses. The cooling stresses can be related to the effect of the coating-substrate connection by studying the residual stress relieving in free-standing coatings. For instance, the XRD data for 95wt% Al2O3-5wt% SiC flame spraying coating given in Table 3. The composite coating has a different behavior and the values remain constant, because of their brittleness, which induces mechanisms of strain accommodation inside the deposit. The value of the residual stresses is \( \sigma = -325.6758 \) MPa which was calculated using equation (1). This cooling stresses are due to the mismatch in thermal contraction between coating and substrate. They contribute to a stress gradient in dense coatings with a relatively high stiffness, because they are not affected by the release of quenching stress. In that case cooling component of the residual stress state increases with the coating thickness. The spalling of some thick coatings after spraying and during cooling to room temperature can be explained by the effects of the cooling stresses. So, interface with the substrate as a consequence of thermal mismatch strains. In fact, the difference of the stresses determined in the surface layer of these samples is opposite in sign to the cooling stresses calculated for the coating at the interface with the substrate. They are representative of the maximum bending stress at the surface side of the coatings while they are connected to the substrates. Cooling stresses, as responsible for a stress gradient in the coating thickness, explain the spalling of thick of materials that can be affected by their action. Industrial practice shows that such a phenomenon occurs, not during the spraying process, but after some time has elapsed, during the cooling to room temperature. This is explained by the bending moment due to the stress gradient, which would reach considerable magnitude in the thickest coatings. The residual stresses within the flame sprayed composite ceramic top coating are not distributed uniformly through the thickness, but change gradually, with the maximum in plane compressive stress at the interface, and diminish away from the interface, presenting very small value of residual stress on the free surface, this is confirmed by the study [27].
4. Conclusions
The X-ray diffraction technique has been demonstrated to be a powerful tool for evaluating and analyzing residual stress distributions in composite wear protection coating systems. In contrast to rather ‘simple’ single film on substrate systems, which are usually analyzed by means of the sin2ψ-method or related techniques, stress evaluation in complex coating systems requires the application of advanced methods that are different for the coating and the substrate.

In the present work may be drawn the following points:

1. The analysis of the in-plane stress was done in the individual layer of 95wt% Al2O3-5wt% SiC flame spraying composite coating.
2. The evaluation of the residual stresses is \( \sigma = -325.6758 \) Mpa which was calculated using the least-square-fitting the corresponding function to the experimental \( \sin2\psi \)-data. This cooling stresses are due to the mismatch in thermal contraction between coating and substrate.
3. The residual stresses within the flame sprayed composite ceramic top coating are not distributed uniformly through the thickness, but change gradually, with the maximum in plane compressive stress at the interface, and diminish away from the interface, presenting very small value of residual stresses on the free surface.

5. References
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