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RESEARCH ARTICLE - MATERIAL SCIENCE (MISCELLANEOUS)

Residual Stresses Characterization of Hard Ceramic Coating (SiC-5wt%Al_2O_3) Using X-Ray Diffraction Technique

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Abstract

The aim of this study is to measure residual stresses, specifically in wear protection coating, using the sin2ψ based on the X-ray diffraction technique. The combination of silicon carbide substrate and Aluminum oxide (SiC-5wt.% Al2O3) is used as the wear protection coating, and AlNi alloy represents the bond coat, which is formed by the flame spraying method on the mild steel substrate. This, in turn, has enabled the reduction of the thermal expansion constant between the substrate and the composite ceramic layer. The diffraction angle, 2θ, is measured experimentally, and the lattice spacing is calculated using Bragg's Law using the measured diffraction angle and the known X-ray wavelength. Interestingly, stress calculations for the samples demonstrate a linear relationship of a slope proportionate to stress, similar to homogeneous isotropic samples in bi-axial stress. Thus, the relationship between dspacing and sin2ψ is demonstrated as a straight line with a slope proportional to stress. However, the oscillatory trend showed the existence of inhomogeneous stress distribution. To resolve this challenge, X-ray elastic parameters are deployed instead of Poisson ratio (ν) and Young's modulus (E) values. The value of the residual stresses for these coatings calculated is compressive residual stresses of (-594.029 MPa).

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1. Introduction

High-quality thermal spray coating can be noticed in several applications that incorporate adorable resistance to high temperature, cracking, deboning, and spallation, which reflect primitive coating durability. Thermal spray coating is specifically a useful process that can be used to apply a wide range of materials to a wide variety of substrates. This made a positive contribution in a variety of industries, such as power generation, oil and gas, and medical sectors [1]. However, it should be noted that each industry has its own specific constraints and requirements for thermal spray coatings. For instance, the power generation industry necessitates coatings that can endure high temperatures and corrosion conditions [2]. Also, wear, corrosion, and high temperatures are the main challenges of utilizing thermal spray coatings in the oil and gas industry [3]. The medical industry requires coatings that are biocompatible and wear-resistant [4].

Interestingly, residual stresses play a vital role in coating strength. Specifically, residual stresses are internal stresses that occur in a material even in the deficiency of external forces. For instance, tensile residual stresses normally aid in surging the vulnerability to cracking and deboning. Residual stresses can be caused by a diversity of parameters, such as thermal expansion mismatch, phase transformations, and plastic deformation. Residual stresses can meaningfully impact the mechanical properties of a material, such as its strength, toughness, and fatigue life [5, 6].

Multiple colleagues were focused on the assessment of residual stresses in coatings [7-9]. Basically, cooling of a thermal spray coating normally forms tensile or compressive residual stresses. This is belonging to the discrepancy of thermal expansion constants of the substrate and coating [10]. The formed residual stresses are affected by several factors, including the substrate temperature through spray deposition, coating thickness, roughness, and porosity [11]. An increase in coating thickness and deposition temperature would positively increase the residual stresses, as experimentally confirmed by Ghafouri-Azar et al. [12]. The most reliable method of thermal barrier coatings (TBC) is the protection of high-temperature gas turbine engines. Such coating is widely demanded due to higher temperature engines being developed [13-15]. This thermal coating is carried out by various mechanisms, such as high-flame temperature methods of plasma spray, flame spray, and arc spray [16, 17]. Solidifying and instantly a few microseconds cooling represent partial and full striking of molten particles onto the substrate's surface. Once the particles are hardened and adhered to the substrate's surface, the contraction of the splats can be limited. This is carried out by

Nomenclature & Symbols			
XRD	X-Ray Diffraction	E	Young's Modulus
ν	Poisson Ratio	TBC	Thermal Barrier Coatings
AISI	American Iron and Steel Institute	SEM	Scanning Electron Microscope

substrate material or by considering solidifying coating material, which causes inherent deposition or quenching tensile stresses. A great theoretical residual stress of up to 1 GPa is caused due to massively high-temperature variance. On the contrary, many relaxation approaches, including the splats' sliding, micro-cracks, plastic distortions, and creeping of material, lead to much lower than 100 MPa of residual stress [18]. In this aspect, X-ray diffraction was noticeably deployed for various types of materials to determine macro and micro residual stresses in a thin film layer. Interestingly, the neutrons' penetration power permits through-thickness stress profiling without the need for any material elimination.

SiC substrate and 95wt.% Al_2O_3 has been applied in several applications as a coating alloy characterized by low friction and high resistance. Protective coatings against corrosion in steel, aerospace moving constituents, and metal working tools represent some of these industrial applications [19].

Mahmoud et al. [20] used sin2ψ based on the X-ray diffraction (XRD) approach to investigate the residual stresses essentially found in wear protection coatings. Specifically, the coating of wear was conducted via 95% Al_2O_3 and 5wt.% SiC substrate besides using AlNi alloy as bond coat that being generated via flame spraying method. This in turn has confirmed compressive residual stresses of (-325.67 MPa) for the recent coating. It is noteworthy to mention that (-325.67 MPa) of compressive residual stress might be enhanced via the employment of a novel mixture of Al_2O_3 and SiC substrate.

The development of new thermal spray coatings is an ongoing process where several researchers are persistently working to enhance the performance of thermal spray coatings by advancing new materials and processes. Up to the authors' knowledge, the assessment of the residual stresses of SiC substrate and 5wt.% Al_2O_3 (this is quite the reverse composition of the one presented by Mahmoud et al. [20] wear protection coating was not achieved yet. In this regard, the selection of SiC substrate can be attributed to the high hardness and wear resistance of SiC substrate besides its good thermal stability (it endures high temperatures without losing its mechanical features), making it ideal for use in protective coatings [21]. Due to its good adhesion property to metal substrates, SiC can be a competitive choice for coatings that are applied to metal components [22]. On top of this, the addition of 5wt.% Al_2O_3 would also improve the wear resistance of the coating. Here, it should be noted that most previous studies endorsed the feasibility of using 5wt.% Al_2O_3. More recently, Sekaran et al. [23] ascertained that the composite containing SiC and 5wt.% Al_2O_3 has the most superior mechanical properties of hardness, impact, and tensile strength. Indeed, in the case of hard ceramic coatings, such as SiC substrate and 5wt.% Al_2O_3, residual stresses can be predominantly imperative. These coatings are frequently utilised to protect substrates from wear and corrosion. However, if the residual stresses in the coating are too high, they can lead to premature failure of the coating. Therefore, this study intends to resolve this challenge via X-ray diffraction methodology to measure the residual stresses.

The following factors could account for the variance in the residual stresses:

- Differing coefficients of thermal expansion for the substrate, bond coat, and top coat, respectively;
- Breakdown of the coating system;
- The substrate's specific heat capacity;
- The thermal spraying process's kinetic impact of the particle on the substrate, which is brought on by heat transfer from the coating layer to the substrates. All of these factors are vital for the cooling and solidification of the coating layer once the coating process is complete. Surface morphology and coating process factors may also be involved.

2. Materials and Parameters of the Spraying Processes

The thermal spraying procedure, known as flame spraying, is used for coatings in the air on plain-carbon steel type AISI 1050. The carbon steel is a cylindrical substrate with a diameter of 18 mm and a height of 10 mm. The heat flame is generated via a spray gun in a flame-spraying unit as a result of the burning of acetylene in the presence of oxygen. Specifically, the melted powder is achieved in the gas mixture and committed to the coated surface using a high-temperature torch of around 3000 °C. To gain and maintain a flame equivalent to the powder's rush speed, it is necessary to manage the pressure of the gases. Therefore, the oxygen pressure must be altered based on the spray gun. This work has been characterized by two top bond coating layers of AlNi alloy. The main intention was to reduce the thermal expansion constant between the substrate and composite ceramic layer. the operating settings are provided in Table 1, while Fig. 1 shows the system of flame spray using the oxygen and acetylene gases mixture to complete the heating and spraying process.

In this regard, it should be noted that the operating parameters of Table 1 were carefully selected based on accumulative experience to maintain the desired thermal coating properties and performance of the composite SiC and 5wt.% Al_2O_3. For instance, the oxygen pressure of 4 bar was selected to ensure the melting of the SiC and Al_2O_3 powder particles while controlling the temperature of the flame. However, any further increase in the oxygen pressure would lead to the oxidation of the powder particles. Furthermore, the acetylene pressure was controlled to be 0.7 bar in a way to control the size and shape of the flame, while providing uniform coverage of the substrate. However, any further increase in the acetylene pressure would cause a carburizing flame. The distance between the flame spraying torch and the substrate was also selected to guarantee a higher heat input without damaging the substrate. This, in turn, would ensure that the powder particles bond to the substrate.

3. Result and Discussions

3.1. X-ray diffraction (XRD) phase analysis results

The X-ray diffraction is a non-destructive method that can be used to measure residual stresses in materials. By measuring the lattice spacing of a material, it is possible to determine the amount of strain that exists in the material. This information can then be used to measure the residual stresses.

The phase composition of the wear protection coatings depends on the powder, velocity of the particles in-flight, solidification rates, cooling rate and the temperature of the deposited particles. The phase structures of the ceramic coating layers are conducted using X-Ray diffraction technique (XRD), where the X-Ray results show αAl_2O_3, α-SiC as the projecting phase in pure SiC substrate, respectively, as represented in Fig. 2.

Table 1. Operating parameters during the coating deposition process

Operating Parameters	**Values**
Oxygen pressure	4 bar
Acetylene pressure	0.7 bar
Distance	20 cm
Powder feed rate	$7\ cm^3/min$
Particle size	Mish (100-300)
Temperature substrate	$(350-400)^{\circ}C$

Fig. 1. The system of flam spray

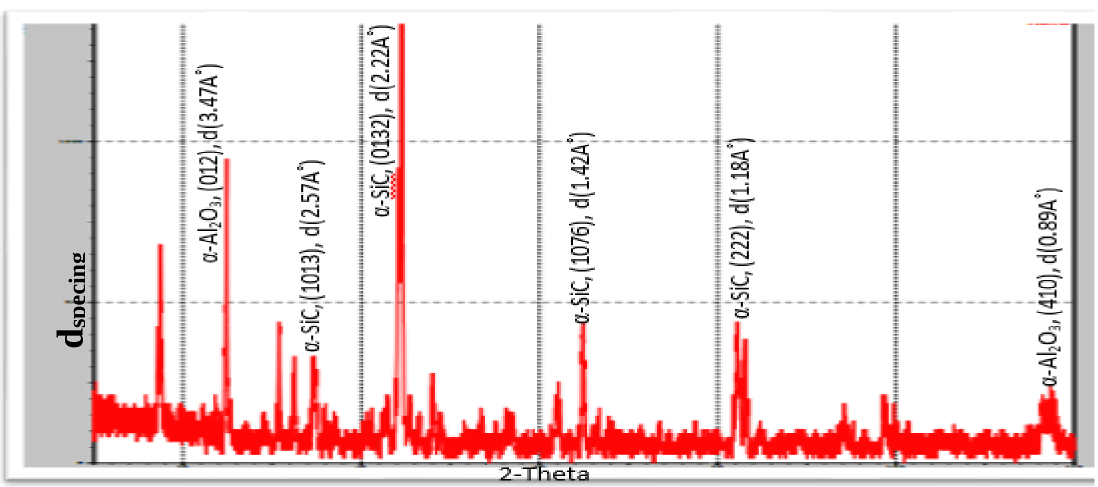

Fig. 2. X-ray diffraction chart for SiC-5wt.% Al_2O_3 coating layer

3.2. Scanning electron microscope

The importance of using a Scanning Electron Microscope (SEM) for various groups is to allocate the surface characteristics. This includes the surface morphology, size, porosity type, cracks, and micro-cracks of the coating layers, as depicted in Fig. 3. Specifically, Fig. 3 depicts the SEM morphology of the flame-sprayed SiC-5wt.% Al_2O_3 coating layer. The coating contains a matrix of SiC substrate and a network of Al_2O_3 particles. Angular particles of the SiC substrate can be seen, while the Al_2O_3 particles are spherical. The coating contains a number of open and closed pores. The open pores are interconnected and permit the flow of fluids, in comparison to the isolated closed pores, which do not allow for the flow of fluids. The coating also comprises a number of voids, which are empty spaces that are not filled with any material.

Fig. 3. SEM morphology of the flame-sprayed SiC-5wt.% Al2O3 coating layer

3.3. X-ray stress evaluation

The standard technique of lattice plane distance ($d_{spacing}$) against $\sin^2\psi$ was normally used to determine X-ray diffraction XRD-based residual stress values [20, 24]. In this regard, sin2 ψ denotes the residual stresses. XRD is originally investigated using Shimadzu X-Ray Diffractometer type XRD-6000 and CrKα radiation. Moreover, the peak shift of the relevant reflection, which signifies the variation of a lattice plane distance ($d_{spacing}$) of a phase was calculated for 0° and 45° of tilt ψ-angles. A linear regression method of the plot ($d_{spacing}$) against $\sin^2\psi$ is used to measure the residual stresses and estimate the X-ray elastic coefficients. Table 2 lists the physical characteristics of both the coating and the substrate. Furthermore, the coating technique employed in the current study's deposition temperature was 850 °C, the same as modelling for the top coating, bond coat, and substrate. Table 3 presents the gained values from the XRD chart.

Table 2. The physical properties of substrate and coating [25-27]

Physical properties	Substrate	Bond coating	Top coating
Yong's modulus (Gpa)	200	105	301
Poisson ratio	0.33	0.315	0.144

Table 3. 2Θ and ψ to SiC-5wt.% Al_2O_3

2Θ	$(Sin^{\varphi})^2$
0	0
157.891	0.066987
158.585	0.25
158.401	0.5

At n = 1, λ = 2.28970 Aº and Θ (0, 15, 30, 45) degree, the value of d can be calculated using the Brag Law (2d sinθ = nλ). The linear slope of the plot $d_{spacing}$ against $\sin^2\psi$ can be estimated from Fig. 4.

Eq. 1 used to calculate the stress

$$\sigma = \frac{E}{(1+V)} \frac{1}{do} \frac{\partial d}{\partial sin^2\psi} \quad (1)$$

σ is the elastic module, V is the poussin ratio, d is the $d_{spacing}$. Fig. 3 shows the slop ∂d/ (∂sin2ψ)) = -0.00876. Therefore, $d_{spacing}$ values from XRD and CrKα radiation are extracted from Eq. 1. Occasionally, Eq. 1 identifies that SiC-5wt.% Al_2O_3 topcoat was exposed to maximum compressive residual stresses of (-594.029 MPa). In this regard, Mahmoud et al. [20] concluded that the compressive residual stresses of the contrast composite of 5wt.% SiC substrate and 95% Al_2O_3 is (-325.67 MPa). This difference in the compressive residual stresses between these two composites can be attributed to the difference in the thermal expansion coefficient between the associated compounds of the coating and substrate (SiC and Al_2O_3). SiC and Al_2O_3 have thermal expansion coefficients of 4.0×10^{-6}/K and 8.5×10^{-6}/K, respectively. This means that SiC expands less than Al_2O_3 when heated. Referring to the composite used in this research of 95wt.% SiC substrate and 5% Al_2O_3, it is fair to expect that the higher volume fraction of SiC in the coating can generate a higher difference in thermal expansion between the SiC and Al_2O_3 particles. In turn, this would lead to a greater degree of compression and, therefore, a higher compressive residual stress compared to the low fraction of SiC in the composite of 5wt.% SiC substrate and 95% Al_2O_3 of Mahmoud et al. [20].

These residual stresses were assessed in the surface layer of coatings adherent to the substrates. Furthermore, a 500 µm thickness of layers was noted as compatible with X-ray penetration. More importantly, several sites of each coating were tested in the current study besides determining the residual stresses along two perpendicular directions corresponding to azimuth angles of 0 and 90º. For each sample examined, the results identified the characteristics of a plane-equi-axial and compressive stress state, with fixed values at locations far from borders or irregularities. The upcoming results of this study will confirm these characteristics.

The top surface residual stresses can be determined using the SiC-5wt.% Al_2O_3 coating standard that is applied via flame spraying. The three most important factors in this regard are the substrate's composition (AISI 1050), thickness (10 mm), and bond coat (200 m) of NiAl. As a result of widespread micro-cracking caused by spraying, the level of residual strains remained constant. The stress release is significantly impacted by the coating imperfections, porosities, and micro-cracks, and only quenching stresses are left in the final deposit [18].

Interestingly, the above research presents the deployment of the X-ray diffraction method to identify the residual stresses in the ceramic coating of SiC-5wt.% Al_2O_3 with acceptable accuracy. To ascertain the real loads that a component experiences, it is crucial to understand the residual stress conditions. Changes in residual stresses can directly influence a diversity of mechanical properties of a material, including its strength, toughness, fatigue life, corrosion resistance and wear resistance. For instance, high residual stresses can cause a reduction in strength and toughness while increasing fatigue life and corrosion resistance. However, compressive residual stress on a component's surface is often advantageous. It usually increases fatigue strength and fatigue life, slows the spread of cracks, and increases resistance to environmental cracking, such as hydrogen-induced cracking and stress corrosion cracking. Tensile residual stress on the component's surface is often not desired since it reduces fatigue life and fatigue strength, speeds up crack growth, and weakens resistance to environmentally induced cracking.

Referring to the above discussion, it should be noted that any changes in residual stresses can influence the potential applications of a composite material. For instance, a composite material with high residual stresses is not a practical option for applications where strength and toughness are important. However, this composite material can be useful for applications where fatigue life and corrosion resistance are important. Accordingly, the compressive residual stresses measured in this study for the SiC-5wt.% Al_2O_3 hard ceramic coating are advantageous for many applications to enhance the strength, toughness, and wear resistance of the coating.

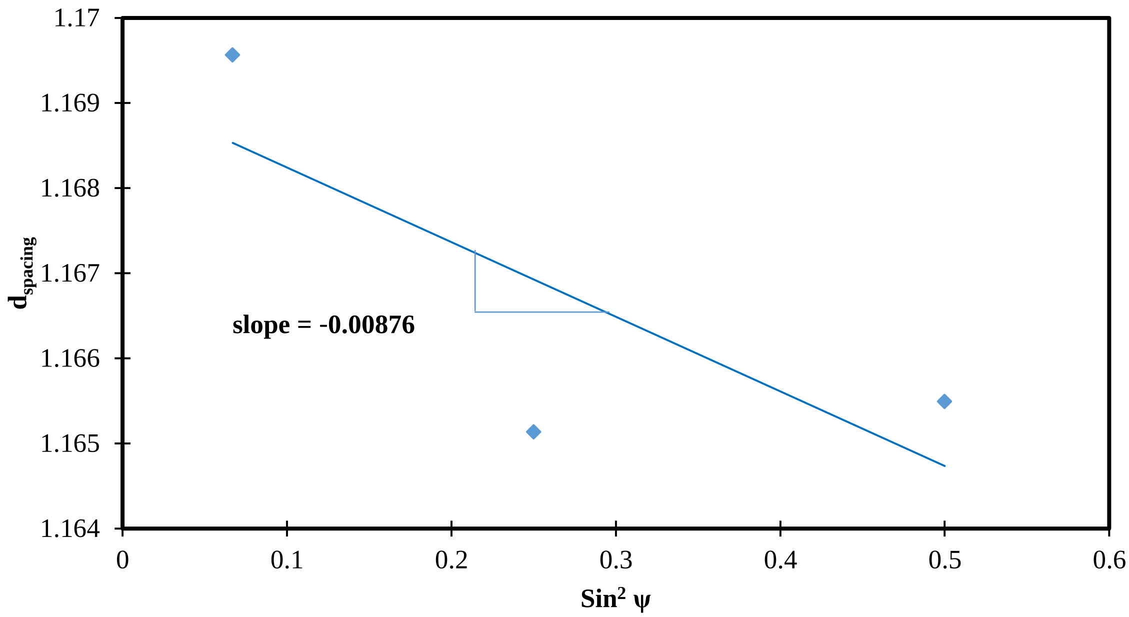

Fig. 4. Relationship between sin^2 ψ and $d_{spacing}$

4. Conclusion

The X-ray diffraction method is proven to be a feasible tool to evaluate and analyses the residual stress distributions in composite wear protection coating units. In this study, the stress assessment of complicated coating systems necessitates advanced approaches to coating and the substrate compared to the coating of single film on substrate systems. Specifically, a recent study has carried out the stress analysis of a single layer of SiC-5wt.% Al_2O_3 flame spraying composite coating. This, in turn, affirmed the maximum value of compressive residual stresses of (-594.029 MPa), which was determined using the least square-fitting method characterized by the related function to the experimental $\sin^2\psi$-data. However, irregular distribution of the residual stresses throughout the thickness was noticed within the composite ceramic top coating. Specifically, the residual stresses have attained its maximum value at the interface and its minimum value out the interface on the free surface.

The findings of this study can provide a better perception of the residual stresses that exist in hard ceramic coatings. This would therefore enable to design coatings that are more resistant to failure. Also, the findings can aid to develop new techniques to measure the residual stresses in hard ceramic coatings while improving the quality control of these coatings. On top of these advantages, the current findings can be utilized to improve new applications of hard ceramic coatings to specifically protect components in harsh environments in a wide set of industries such as aerospace and automotive industries. However, the investigation of the optimum concentration of Al_2O_3 is feasible to be investigated in the upcoming research.

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