

Investigation of Residual Stresses in ZrO₂-20%MgO Thermally Sprayed Coating Using X-Ray Diffraction Technique

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Abstract

The aim of this work is to investigate the residual stresses and microstructural characteristics of advanced composite ceramic coatings (ZrO₂-20%MgO) which were produced by thermal spraying coating (flame spraying) onto the low carbon steel substrate (AISI 1015) steel. The bond coat used in this work was NiCrAlY alloy between metallic substrate and advanced composite ceramic coatings which was implemented by flame spraying technique. The residual stresses were evaluated by X-ray diffraction techniques using $\sin^2\psi$ method. Microstructural examination studied by using scanning electron microscopy (SEM) and (EDS). The thickness of bond coating was (260 μm) and for composite ceramic coating was (520 μm). The results indicated that the residual stresses were compressive residual stresses. The magnitudes of the stresses in the as-sprayed condition are low and its value was (-19.041 MPa).

Keywords: X-ray; residual stresses ZrO₂-20%MgO; flame spray technique.

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1-INTRODUCTION

Residual stresses can be defined as those stresses that remain in a material or structure after manufacturing and processing in the absence of external forces or thermal gradients. Causes of residual stresses may be manufacturing process such as , welding, machining, forming and heat treatments through thermal gradient. Residual stresses are known to play an important role in coating durability; for example, tensile residual stresses typically increase the susceptibility to cracking and debonding [1]. Residual stresses develop during cooling of a thermal spray coating due to the mismatch of thermal expansion coefficients of the coating and substrate. Depending on the relative magnitudes of the thermal expansion coefficients of the coating and substrate [2-3] residual stress can be either tensile or compressive. Parameters that strongly affect the magnitude of residual stresses are coating and substrate temperature during spray deposition and properties of the coating such as thickness, roughness and porosity. Experiments have shown that residual stresses increase with coating thickness and deposition temperature [4]

Generally, the residual stresses of thermally sprayed coatings are induced by different mechanisms and sources

[5-7]. In a thermal spray process with a high flame temperature, such like flame spray, plasma spray, or arc spray, fully and partially molten particles striking onto the surface of the substrate, are flattened, solidified, and cooled down in a very short period of time (few microseconds)[8,9]. After their solidification and adhesion onto the surface of the substrate, the contraction of the splats can be hindered by substrate material or the underlying solidified coating material which results in tensile stresses which are called intrinsic, deposition, or quenching stresses. Due to an extremely high temperature difference, a high theoretical residual stress in the order of up to 1 GPa can be induced. However, due to the many relaxation mechanisms, such as the sliding of the splats, micro cracks, plastic deformations, and material creep, the experimentally measured values are much lower (<100 MPa)[10]. X-ray diffraction has used as a complementary technique; it can determine stress only in a thin surface layer [11]. Many researchers have determined the residual stresses of thermal spray coatings employing different techniques.

Santana et al., 2008 [12] Studied the residual stresses of coating by using of XRD and hole drilling methods. High-velocity oxygen-fuel (HVOF) spraying has shown to be one the best methods for depositing WC-Co powders on plan Carbon steel substrate. It has been found that the mean residual Von Mises stress was higher in the thinner coating than in the thicker one, of approximately 180 and 107 MPa, respectively. Chen *et al.*, 2015 [13] investigated the residual stresses in a NiCoCrAlY bond coat for thermal barrier coating on a Ni-based super alloy substrate by high velocity oxygen fuel spraying (HVOF) using X-ray diffraction. The stresses were found to be tensile of magnitude closed to 60 MPa. Zoei, Sadeghi and Salehi, 2016[14] studied the residual stresses of WC-10Co-4Cr coated by high velocity oxy fuel (HVOF) on the steel plates type AISI 1010. And evaluated them using X-Ray diffraction (XRD) technique. The results were found to be compressive residual stresses of (210.3±75.1) MPa.

Qu *et al.*, 2018 [15] Investigated the high temperature residual stress in a typical Thermal barrier coatings (TBC), nanostructured 8 wt% yttria partially stabilized zirconia (YSZ) coating by in-situ high temperature indentation method. The residual stress was measured to be - 131.3, - 55.5 and - 45.5 MPa, correspondingly. Moreover, the

residual stresses decrease with increasing environmental temperature.

The objective of this work has to further explore the use of X-ray diffraction to determine the residual stresses in composite ceramic coating (ZrO_2 -20%MgO) sprayed on low carbon steel substrates, where this technique has high accuracy and it is nondestructive technique therefore used in the present study. And study the microstructure effect on the nature of residual stresses

2-EXPERIMENTAL PROCEDURES

AISI 1015 plain-carbon steel cylindrical substrates that's dimensions have 15 mm in diameter and 10 mm in height . The chemical composition is analyzed in Ministry of Industry and Minerals ,state company for Inspection and Engineering Rehabilitation Baghdad shown in table(1) .The surface roughness of the substrates was increased by using sand blast under pressure 8.5 bar and 40cm of distance .The surface has then cleaned and degreased using acetone An average surface roughness of $5.48\mu m$.A commercial NiCrAlY

was used as feedstock powder for the bond coat, whereas for the top coat ($ZrO_2 - 20\%MgO$)powders were selected. the powder mixture has ball milled for 2 hr with using speed of (300 r.p.m) to get homogenous particles

distribution. mechanical properties of material as shown in table 2.

The powders were sprayed by flame spraying system onto the substrates using an oxygen-acetylene gun. Show in the figure(1) The heat flame was 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 could raise to $3000\text{ }^\circ\text{C}$. 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. The conditions of deposition process were listed in Table (3).

Table 1 Chemical Composition of plain-carbon steel (AISI 1015)substrate

C%	Si%	Mn%	P%	S%	Cr%	Mo%	Ni%	Al%	v%	cu%	Fe%
0.138	0.19	0.538	0.0129	0.0156	0.0803	0.0154	0.0963	0.0069	0.0015	0.318	Balance

Table 2 Mechanical properties of material

material	Elastic modulus	Posion ratio
AISI(1015)	190 - 210 GPa	0.27 - 0.30
ZrO ₂	100 GPa	0.22
MgO	270 GPa	0.35

Table 3 Operating parameters during thermal spraying coating technique

Operating Parameters	Values
Oxygen pressure (bar)	4
Acetylene pressure(bar)	0.7
Distance(cm)	20
Powder feed rate(cm ³ /min)	7
Particle size(Mm)	(100-300)
Temperature substrate (C°)	(300 - 450)

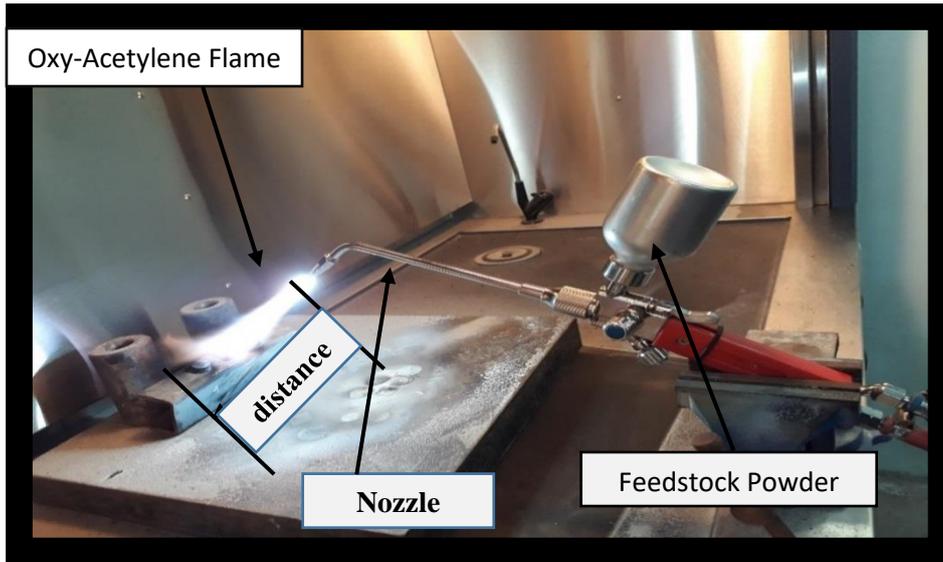


Figure 1: Flame spraying system

3-RESIDUAL STRESS ANALYSIS

XRD-based residual stress measurements were made using standard dspacing vs. $\sin^2\psi$ techniques using Shimadzu X-Ray Diffractometer type XRD-6000 and $\text{CrK}\alpha$ radiation as shown in figure(2). The $\sin^2\psi$ method [16, 17] 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 ψ -angles between 0° and 45° . To calculate the residual stresses the linear regression of the plot (d spacing) versus $\sin^2\psi$ and the X- ray elastic constants. The coating and substrate physical properties (elastic modulus, Poisson's ratio,) thickness of the top coating, might be calculated. To calculate elastic

modulus and Poisson's ratio for composite coatings using the following equations [18] .

$$(1/EC)=(wt_1\%/E_1)+(wt_2\%/E_2) \tag{1}$$

$$v=\sum v * wt\% \tag{2}$$

The deposition temperature used in the present work during coating process for the topcoat and bond coat was 850°C .

From Shimadzu X-Ray Diffractometer XRD-6000 chart, will be getting on the following values are shown in the table 4.

Table 4. The relationship between 2θ & ψ for $(\text{ZrO}_2 + 20\%\text{MgO})$ coating

2θ degree	Ψ degree
156.231	0
156.271	15
156.271	30
156.328	45

By Brag Law ($n\lambda = 2d \sin\theta$) may be calculated (d), where $n=1$, $\lambda= 2.28970 \text{ \AA}$ and θ (0, 15, 30, 45) degree. From the figure 3 may be calculated the linear slop of the plot dspacing versus $\sin^2\psi$. The stress can then be obtained from the following equation:

$$\sigma = \left(\frac{E}{1+\nu}\right) * 1/d_0 (\partial d / (\partial [\sin]^2 d)) \tag{3}$$

From figure1 the slop $\partial d / (\partial [\sin]^2 d) = -0.00025$

$$d^\circ = 2.02712 \text{ \AA}$$

from Eq. (3), the value of the Residual stresses is:

$$\sigma = -19.0414\text{Mpa}$$

Table 5.shows symbols of used in equation(1,2,3)

σ	<i>Residual stresses</i>
E	<i>young modulus</i>
ν	<i>poison ratio</i>
E_c	<i>elastic modulus for composite</i>



Figure 2: The X-Ray diffraction instrumentation.

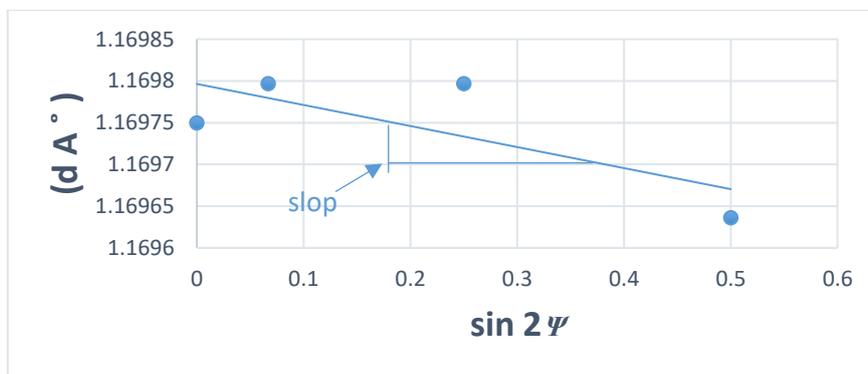


Figure 3: The relationship between (sin2ψ) and d-spacing (A °) for ZrO₂ +20%MgO) coating.

4-REULTS AND DISCUSSION

4-1 Microstructure

Figure (4) shows the surface of (ZrO₂-20%MgO) coating .The surface showed typical microstructure of flame sprayed coating with open pores, lamellae boundaries and splats. A large number of fine particles have observed on the surface of ceramic

coating because of some the coating particles that did not deform appreciably on impact. Sites can be distinguished where continuous, very fine porosity will be prevalent (formed by splat splashing causing the surface roughness and imperfect bonding between adjacent lamella).The lower percentage of the porosity give enhanced mechanical properties ,and good quality of coating .

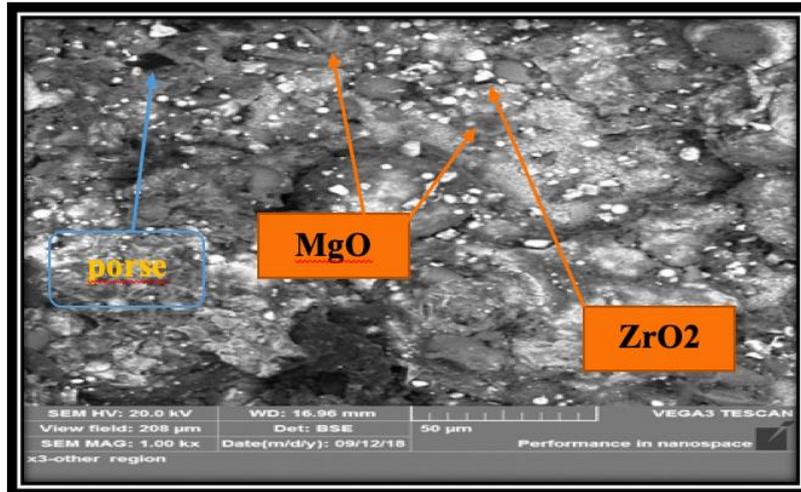


Fig. 4. Cross-sectional SEM images of(ZrO_2 -20%MgO)

4.2. Phase analysis

The high-energy X-ray transmission technique allows for localized determination of the phase composition through the thickness of the top coat layer [19]. Shown in figure 5, the phase composition is analyzed for the as-sprayed and heat-treated coatings. The (ZrO_2 -20%MgO) consisted of tetragonal and monoclinic zirconia phases of the coating layer, in addition cubic the magnesia phase. , that have produced during coating process because there are different

solidification and cooling rate to the composite ceramic coating mixtures during flame spraying process depend on the coating parameters resulted in different phases of coating layers .When increase cooling rate the retained phases increasing ,especially (tetragonal phase) for zirconia consequently, the quantity of compressive residual stresses increased resulted in enhancing in fracture toughness of composite ceramic coating

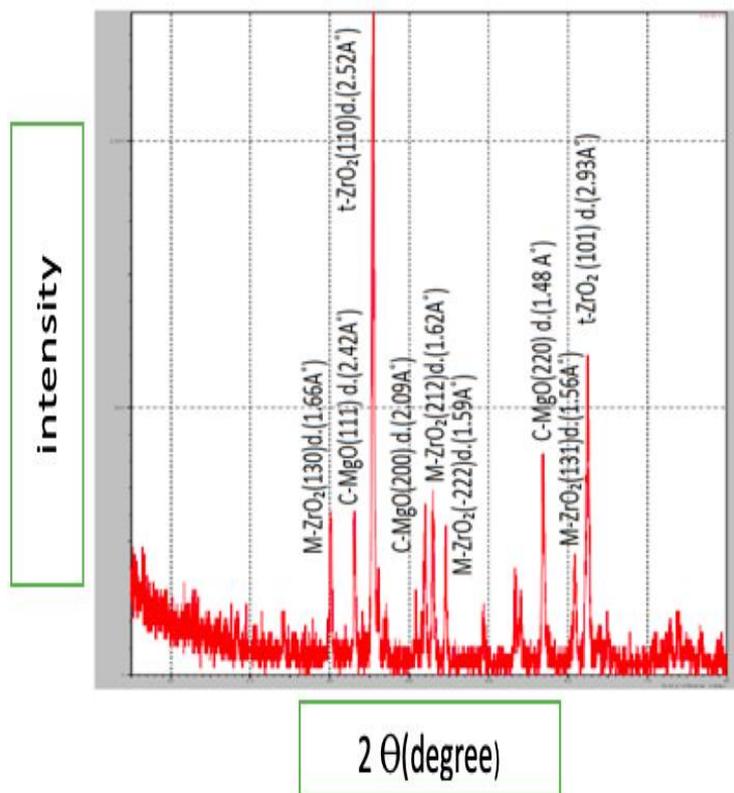


Fig. 5. XRD results of (ZrO_2 -20%MgO) as-sprayed coating

4.3. Residual stress measurement—XRD results

It can be referred from Eq(3) that in topcoat ($ZrO_2 - 20\%MgO$) has subjected to compressive residual stresses (-19.0414 Mpa). This residual stresses have evaluated in the surface layer of coating adherent to the substrate. Thickness of topcoat layer conforming to the X-ray penetration has observed 450 μm . The study has carried out at several sites of each coating and the residual stresses have always determined along two perpendicular directions corresponding to tilt angles of 0° , 15° , 30° , 45° . The results showed for each sample characteristics of a plane- equiaxial and compressive stress state, with constant values at sites far from the borders or

irregularities. The results presented in the remainder of the study confirm this feature. The level of the residual stresses in the topcoat surface has related to characteristics of composite ceramic coating ($ZrO_2 - 20\%MgO$) deposited by flame spraying technique. The parameters considered were the substrate material (AISI 1015), the substrate thickness (10 mm), and a bond coat of NiCrAlY (260 μm). As can be observed, the level of the residual stresses remains constant for all the samples inside the error bars. This behavior can be related to the stress relief by extensive micro-cracking during spraying. The coating flaws, porosities and microcracks have an important effect on the stress release and only quenched stresses remain in the finished deposit.

5-Conclusions

The composite ceramic coating ($ZrO_2 - 20\%MgO$) has been produced successfully by thermal spraying coating (flame spraying technique), the microstructure of this coating showed that the coating layer open pores, lamellae boundaries and splats. The thickness of composite ceramic coating was (520 μm). The phases produced after coatings process were tetragonal and monoclinic structure for zirconia phases in addition to cubic for magnesia phase. The residual stresses evaluated using X – ray diffraction technique, which were the quality of these stresses were compression residual stresses and their value (-19.0414 Mpa). the residual that compressive stresses are useful in advanced ceramic coatings, where, it contribute enhancement of the fracture toughness.

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