

Behavior of residual stress in plasma sprayed ZrO_2 coatings with different contents of LaPO_4

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In this study, ZrO_2 - LaPO_4 composite coatings were deposited by a plasma spraying technique. For nondestructive evaluation and easy practicability, the X-ray diffraction method was chosen to determine the residual stress in plasma sprayed ZrO_2 - LaPO_4 composite coatings. The relationship between residual stress and content of LaPO_4 was investigated.

Key words: Plasma spray, X-ray diffraction, Residual stress.

Introduction

Lanthanum phosphate (LaPO_4) has been used to produce composite ceramic materials so as to get excellent mechanical and thermal properties. Many researchers have investigated LaPO_4 containing composite ceramics which were produced by a sintering process [1]. In this study, we tried to deposit ZrO_2 - LaPO_4 composite coatings by a plasma spray technique and study the behavior of the residual stress in ZrO_2 coatings with different contents of LaPO_4 .

Experimental Procedure

Because of their low mass and inability to be carried in a moving gas stream and deposited on a substrate, ZrO_2 nanoparticles can not be used directly to deposit ZrO_2 coating. Before spraying, ZrO_2 nanoparticles also have been reconstituted into micro-sized powders by a spray-drying process. The SEM morphology of the spray-dried nanostructured zirconia powder is shown in Fig. 1. It was found that they are spherical or nearly-spherical. The ZrO_2 powder was mixed with LaPO_4 powder, the contents of LaPO_4 powder were 0%, 10%, 20%, 30%, 40% by volume.

ZrO_2 - LaPO_4 composite coatings were deposited under optimal spray conditions (See Table 1). AISI 304 stainless steel plates with the dimensions of $50 \times 20 \times 2 \text{ mm}^3$ were used as the substrates. Before the plasma spraying, all the stainless steel plates were degreased ultrasonically in acetone and grit-blasted with 220 grit alumina. Plasma spraying was carried out using an A-2000 atmospheric plasma spraying equipment (Sulzer Metco AG, Switzerland). H_2 and Ar were used as the plasma gas.

X-ray beams can be scattered by atoms in a material. The elastic strain in the crystal lattice can be inferred from the variation of lattice space, a change in the lattice space will result in a peak shift. Thus, the strain determined from peak shift measurements is representative of a macroscopic elastic strain [2]. In this study, the X-ray source was a Cr tube, Ni and Fe were used as filters, in order to get a $\text{CrK}\alpha$ beam ($2.2897\text{\AA}$) to give as a large peak shift $\Delta\theta$ as possible. X-ray diffraction from these coatings, boosted the tilt angle ψ from $0-45^\circ$ in 5° steps. A position sensitive proportional counter (PSPC, Rigaku Co., Ltd., Japan) was used to record and analyse the X-ray diffraction beams. The recorded peaks were stored in computer for analysis.

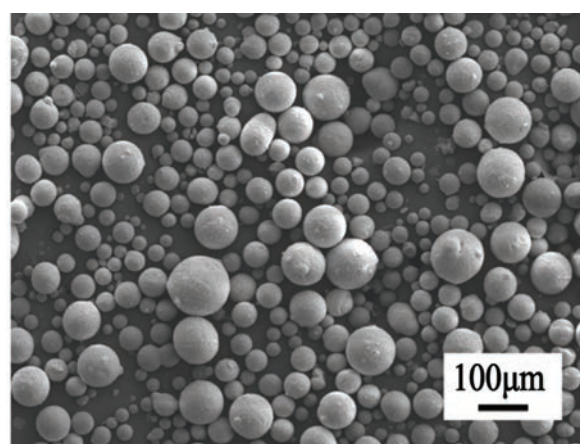


Fig. 1. SEM micrograph of spray-dried ZrO_2 powder.

Table 1. Deposition parameters of ZrO_2 - LaPO_4 composite coatings

Deposition parameters	
Spray distance (mm)	Spray power (kW)
120	43.4

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Peak Top, FWHM (full wave at half maximum) and Gravity methods were used to calculate the residual stress, both of these use only a very small part of the diffraction peak to determine a characteristic value of this

peak. Consequently, every part of the peak was maintained in the displacement [3]. In order to overcome statistical counting fluctuations, local polynomial smoothing was carried out before, around the angular area under

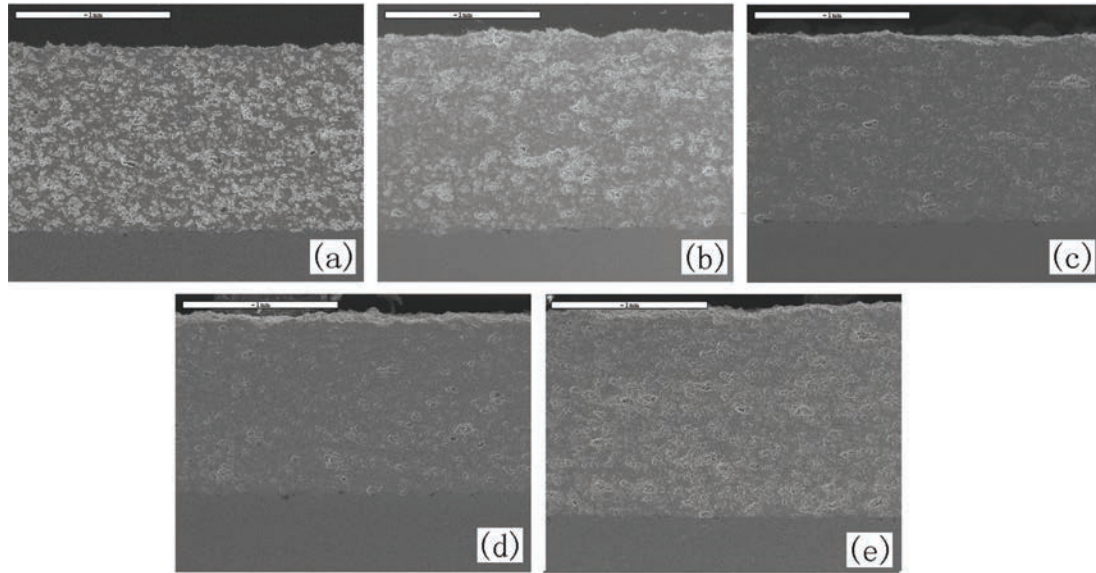


Fig. 2. SEM micrographs of ZrO_2 and $\text{ZrO}_2\text{-LaPO}_4$ coatings: (a) ZrO_2 coating, (b) $\text{ZrO}_2\text{-10%LaPO}_4$ coating, (c) $\text{ZrO}_2\text{-20%LaPO}_4$ coating, (d) $\text{ZrO}_2\text{-30%LaPO}_4$ coating, (e) $\text{ZrO}_2\text{-40%LaPO}_4$ coating.

Table 2. Residual stress (Peak Top) of $\text{ZrO}_2\text{-LaPO}_4$ coatings

Sample		Residual stress (MPa) +: tensile, -: compressive					
		1	2	3	4	5	6
ZrO_2	Avg.	-5.55	-23.05	2.2	-0.66	-1.01	-9.23
	SD	24.34	6.52	10.71	8.62	14.07	14.85
$\text{ZrO}_2\text{-10%LaPO}_4$	Avg.	39.18	3.62	4.79	10.39	-20.12	9.82
	SD	13.82	11.19	14.69	9.47	15.64	22.69
$\text{ZrO}_2\text{-20%LaPO}_4$	Avg.	-122.25	-161.22	-82.09	-60.92	-131.89	-129.52
	SD	21.23	27.38	12.12	21.07	16.31	13.41
$\text{ZrO}_2\text{-30%LaPO}_4$	Avg.	146.16	54.76	96.38	-61.42	-61.18	-88.13
	SD	151.85	111.64	110.08	152.53	107.84	207.47
$\text{ZrO}_2\text{-40%LaPO}_4$	Avg.	279.7	-49.86	4.54	115.74	10.72	-112.36
	SD	289.68	87.37	261.23	132.47	141.68	168.04

Table 3. Residual stress (FWHM) of $\text{ZrO}_2\text{-LaPO}_4$ coatings

Sample		Residual stress (MPa) +: tensile, -: compressive					
		1	2	3	4	5	6
ZrO_2	Avg.	9.09	-19.74	-2.87	-2.49	2.69	1.21
	SD	9.76	8.90	8.08	6.98	10.36	6.34
$\text{ZrO}_2\text{-10%LaPO}_4$	Avg.	-1.84	-8.84	-1.97	-4.16	-5.78	0.89
	SD	8.9	8.06	9.8	9.89	12.54	14.23
$\text{ZrO}_2\text{-20%LaPO}_4$	Avg.	-53.3	-66.31	-62.91	-37.14	-56.98	-27.09
	SD	18.58	14.29	16.4	16.1	15.83	21.41
$\text{ZrO}_2\text{-30%LaPO}_4$	Avg.	-65.17	1.56	-50.56	-104.57	-17.36	-83.94
	SD	41.83	62.74	52.58	71.36	61.37	56.96
$\text{ZrO}_2\text{-40%LaPO}_4$	Avg.	6.69	-54.89	-25.6	-1.36	-88.26	-120.66
	SD	58.2	74.7	99.85	55.45	65.64	81.53

Table 4. Residual stress (Gravity) of ZrO_2 - LaPO_4 coatings

Sample		Residual stress (MPa) +: tensile, -: compressive					
		1	2	3	4	5	6
ZrO_2	Avg.	26.98	9.82	-2.93	3.89	17.79	14.78
	SD	14.7	22.07	12.89	16.69	13.15	9.53
ZrO_2 -10% LaPO_4	Avg.	-5.91	6.44	-18.45	-3.94	-4.85	15.89
	SD	8.98	11.19	14.69	9.47	15.64	22.69
ZrO_2 -20% LaPO_4	Avg.	-53.55	-82.16	-66.49	-34.58	-58.86	-49.06
	SD	23.46	21.91	16.47	17.2	15.87	35.41
ZrO_2 -30% LaPO_4	Avg.	-51.49	-48.88	1.79	-107.58	0.45	-177.79
	SD	36.63	53.84	74.94	78.28	66.27	94.29
ZrO_2 -40% LaPO_4	Avg.	-20.3	-45.91	-13.06	17.13	-97.98	-88.54
	SD	41.07	58.27	52.28	57.93	56.16	54.35

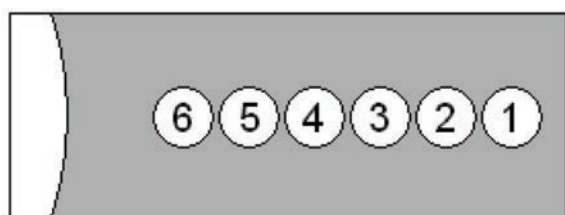
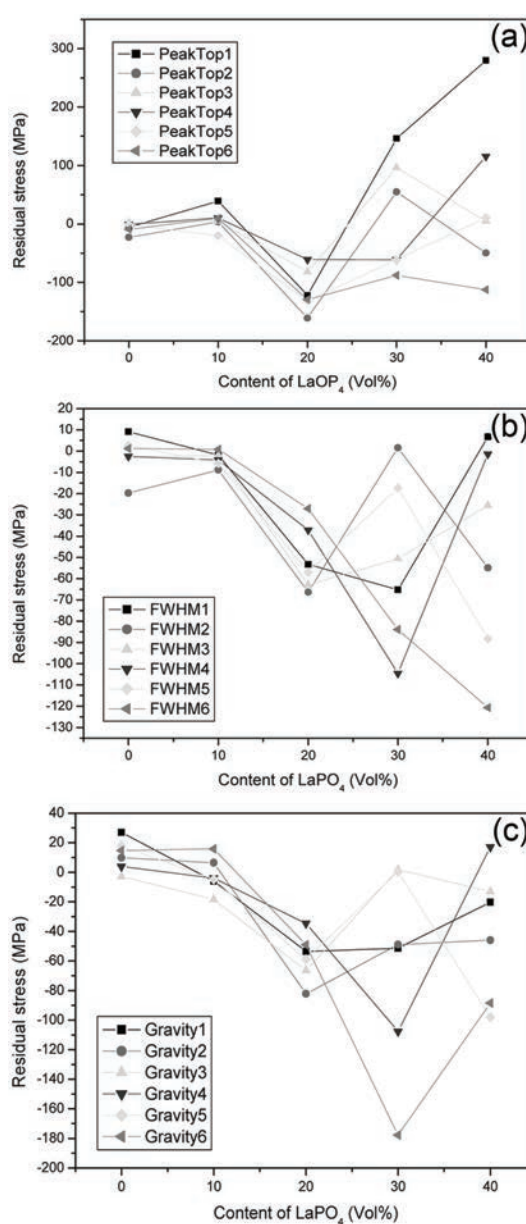
consideration, and the continuous background was removed in order to keep only the net intensities. However, it should be noted that the same portion of the peak can only be processed if the limits selected for smoothing follow the displacement of the peak to be analyzed, which is obviously a problem since this is exactly what we are looking for.

Results and Discussion

Figure 2 shows the SEM micrographs of ZrO_2 and ZrO_2 - LaPO_4 coatings. The thickness of all the coatings is about 1.3 mm. The larger pores resulted from a lower degree of melting of the feedstock as evidenced by the unmelted particles in the pores.

Tables 2-4 present the residual stress in the plasma sprayed ZrO_2 coatings with different LaPO_4 contents. The number 1-6 refers to the 6 points at which measurements were made on the surfaces of the plasma sprayed ZrO_2 coatings as illustrated in Fig. 3.

Figure 4 shows the variation of residual stress with the content of LaPO_4 . Generally, in the case of 0% content of LaPO_4 , the residual stress values are near to zero, also, the standard deviations are very low. In the case of 10% content of LaPO_4 , the residual stress values are not very different from the case of pure ZrO_2 coating. In the case of 20% content of LaPO_4 , residual stress values become higher. It can be seen that the residual stress behavior is obvious when the content of LaPO_4 is in the range of 0%-20%. If the content of LaPO_4 increases from 20% to 40%, the behavior of residual stress is not obvious, and the standard deviation becomes larger. Maybe

**Fig. 3.** Determining points on the ZrO_2 - LaPO_4 coatings surface.**Fig. 4.** Comparison of residual stress curves of the plasma sprayed ZrO_2 - LaPO_4 coatings: (a) Peak Top method, (b) FWHM method, (c) Gravity method.

this is due to complex components which cause local inhomogeneity of the coating when the content of LaPO_4 is about 20%.

The Peak Top method was used to determine the position of the maximum net intensity, I_{max} of a peak and required elimination of the background. If we have a beam doublet, it must be unresolved. But the method can easily be extended to the case of a resolved doublet. This method gives the position of the maximum intensity of the peak which does not correspond to the position of the main line, due to the deformation caused by the second line. Smoothing the peak near the maximum intensity by a second degree polynomial can eliminate statistical fluctuations. The position of the maximum intensity gives the required parameter. It would be better to consider only a range in which the intensity is greater than 85% of I_{max} .

For the FWHM method, if some precautions are taken, it can be used with a resolved doublet. The peak sides are smoothed in the area close to 50% of I_{max} . A second or third degree polynomial may be used. This can precisely define two angular values corresponding to 50% of I_{max} .

Both Peak Top and FWHM methods use only a very small part of the diffraction peak to determine a characteristic value of this peak. They are based on the assumption that the peak does not change its shape when it is displaced. Consequently, every part of the peak is maintained in the displacement. In order to overcome statistical counting fluctuations, local polynomial smoothing is carried out before, around the angular area under consideration, and the continuous background is removed in order to only keep net intensities. However, it should be noted that the same portion of the peak can only be processed if the limits selected for smoothing follow the displacement of the peak to be analyzed, which is obviously a problem since this is exactly what we are looking for.

The Gravity method uses all the information contained within the peak, and gives a good statistical reproducibility. The calculation is easily carried out, but is useful in practice only for sharp peaks. This method is very sensitive to

errors in the background determination and in the peak truncation. It is therefore essential to be able to access areas in which the net intensity vanishes, which is not possible when peaks are very wide and position sensitive detectors are used for which the detection window does not exceed 10 to 20 degrees.

As with previous methods, the calculation is made using net intensities, requiring elimination of the continuous background. However, the problem of choosing limits no longer arises since they must be selected within the areas in which intensities are zero. They have two advantages. First, the statistical precision of localization is improved. Second, they determine the value of a parameter which has a physical meaning compatible with the stress determination method theory and which is consequently independent of the shape of the peak.

Conclusions

The residual stress is low in the plasma sprayed ZrO_2 coating. In the plasma sprayed ZrO_2 - LaPO_4 composite coatings, when the content of LaPO_4 increased from 0% to 20%, the residual stress increased. No obvious trend was found when the content of LaPO_4 was in the range of 20%-40%.

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References

1. D.H. Kuo and Waltraud M. Kriven, *Materials Science and Engineering*. A 210 (1996) 123-127.
2. Jian Lu. *Handbook of measurement of residual stresses*, Society for Experimental Mechanics, Inc. (1996).
3. B.D. Cullity and S.R. Stock, *Elements of X-ray Diffraction*, Addison Wesley Series in Metallurgy and Materials, Massachusetts (2001).