

On the nature of phases in Al_2O_3 and Al_2O_3 –SiC thermal spray coatings

M. Uma Devi*

Laboratory for Surface Engineering, University of Siegen, D-57068, Siegen, Germany

Received 11 June 2003; received in revised form 7 July 2003; accepted 4 September 2003

Abstract

Nature of alumina phases in thermal sprayed Al_2O_3 and nanocomposite Al_2O_3 –SiC coatings is investigated. The coatings were prepared using high velocity oxy fuel spraying (HVOF) and air plasma spraying (APS) techniques on mild steel substrates either with Ni5Al or Ni20Cr bond layer. Coating top layer thickness was 250 μm and 1 mm. The amount of SiC was varied from 2 to 8 mass%. Flat and roller geometries were used for substrates. While coatings on flat specimens were used for low temperature heat treatments (100 and 700 $^\circ\text{C}$); peels (free forms) of roller specimens were used for high temperature (≥ 1000 $^\circ\text{C}$) heat treatments. X-ray diffraction technique was used for phase characterisation. The coatings showed the presence of varied amounts of well-known alumina phases and the relatively new U phase. It was observed that depending upon the thermal energy of the spray process even smaller particles <20 μm , gave rise to (contrary to the earlier hypothesis) unmelted α -seeds (nuclei) and α -alumina nucleated in preference to γ -alumina. Presence of SiC (mostly inside alumina grains) in alumina nanocomposite coatings increased the amount of α -phase formation in the coatings. The transformation temperatures, of alumina polymorphs, of the thermally sprayed alumina coatings, are found to be in accordance with the dehydration of boehmite. The discrepancies (higher transformation temperatures) in the earlier literature on alumina sprayed coatings were due to the presence of impurities in the coating. SiC increased the transformation of metastable alumina polymorphs to stable α -phase by 200 $^\circ\text{C}$. The alumina phases in HVOF Al_2O_3 –SiC coatings undergo $\gamma \rightarrow \delta \rightarrow \theta \rightarrow \alpha$ phase transformations on heating (1000–1400 $^\circ\text{C}$) as observed during dehydration of boehmite. However, the transformation temperatures of metastable alumina phases in SiC containing nanocomposite coatings were 100–200 $^\circ\text{C}$ higher than the pure alumina coatings.

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Keywords: B. Nanocomposites; B. X-ray methods; Thermal spray coatings

1. Introduction

Alumina exhibits polymorphism. In addition to the stable rhombohedral α -alumina, the known metastable alumina polymorphs are γ (cubic spinal), δ (either tetragonal or orthorhombic), θ (monoclinic), η (cubic spinal), κ (orthorhombic), χ (cubic), β (hexagonal) and θ' (monoclinic) [1–3]. Additionally, a new grain boundary phase called U-phase was reported in thermally sprayed alumina-based coatings [4]. These polymorphs can be obtained by dehydration of different alumina hydroxides, rapid quenching from the melt, vapour deposition, thermal spraying and crystallisation of amorphous alumina. The crystalline forms of alumina are constructed of stacked, close packed layers of oxygen ions

with aluminium ions and the vacancies distributed on the tetrahedral and octahedral sites within the oxygen ions lattice. The polymorphism results from the possibilities for (i) different oxygen layer stacking sequences, (ii) variations in the distribution of aluminium ions on tetrahedral and octahedral sites and (iii) ordering of aluminium ions and vacancies on these sites.

Process variables in terms of coating techniques, operating parameters, feed characteristics, substrate and post-coating treatment make the entire gamut of alumina phase formation very complex. Flame sprayed alumina coatings were reported to contain predominantly metastable γ - Al_2O_3 in addition to the equilibrium α - Al_2O_3 phase [5–7]. Plasma as-sprayed alumina coatings from α - Al_2O_3 feed stock additionally showed presence of δ - Al_2O_3 [7,8]. Sokolova et al. [9] used α - Al_2O_3 powder of 25–50 μm size (60%) as feed stock and reported, that depending upon plasma spraying

* Tel.: +49-271-740-4646; fax: +49-271-740-2442.

E-mail address: mu.devi@rediffmail.com (M. Uma Devi).

conditions either γ -Al₂O₃ or δ - and α -Al₂O₃ are formed in the coating. Measurement of particle temperatures in spray systems has shown that with an increase in particle size the fraction of partially melted particles increases rapidly with associated increase in α -Al₂O₃ phase [10]. Formation of α -Al₂O₃ from unmelted seeds and γ -Al₂O₃ from molten alumina is indicated during plasma spraying [7,9–12].

Attempts have also been made to alter the phase constitution of plasma-based alumina coatings through addition of another oxide [7,11,13] or non-oxide constituent, such as SiC [8]. Studies by Kozlova [7] have shown that addition of oxides such as ZrO₂ and MgO increases the α -Al₂O₃ content in the coatings, and other oxides, such as Co₂O₃, Fe₂O₃, Cr₂O₃, NiO, CaO, SiO₂ and TiO₂, behaves in quite opposite way. She explained these results in terms of effect of these oxides on melt temperature and corresponding change in the amount of unmelted α -Al₂O₃ seeds.

Complex behaviour of alumina-based materials has limited their use especially at elevated temperatures. The processes occurring at temperatures above 1000 °C include complex phase transformations accompanied by change in alumina density as well as sintering [5]. Kozlova [7] studied the phase transformations and their kinetics in thermally sprayed alumina coatings between 950 and 1250 °C. It has been reported that metastable alumina phases undergo $\gamma \rightarrow \delta \rightarrow \theta \rightarrow \alpha$ transformations similar to the one observed during heating boehmite (AlOOH). The $\gamma \rightarrow \delta \rightarrow \theta$ -Al₂O₃ transformations are reported to involve only change in cationic positions, whereas $\theta \rightarrow \alpha$ -Al₂O₃ is a complete structural transformation. Extensive work has been done in last years on the subject of understanding and controlling of the phase transformation temperatures by addition of other oxides [7,11,13]. Even better, it was found possible to control phase composition of the as-sprayed deposits by variations of feed stock chemistry [11,13]. Nanocomposite plasma coatings containing alumina and other oxide, such as TiO₂, have recently been investigated [11]. Presence of TiO₂ is reported to suppress θ -phase formation [11,13]. Addition of Cr₂O₃ increases alpha phase formation temperature and also increases amount of alpha phase present in as-sprayed deposits. Larger amounts of Cr₂O₃ stabilize alpha phase in the as-sprayed deposits avoiding phase transformations entirely [13]. The information on alumina-based nanocomposite coatings containing non-oxide such as SiC is scarce.

In this paper results are presented on the nature of alumina phases formed in Al₂O₃ and Al₂O₃-SiC nanocomposite coatings that are prepared on mild steel substrate using HVOF and APS techniques. There is greater emphasis in the paper on the Al₂O₃-SiC nanocomposite coatings prepared using HVOF. The effect of different bond coats, Ni5Al and Ni20Cr, is investigated. The $\gamma \rightarrow \delta \rightarrow \theta \rightarrow \alpha$ transformations in the Al₂O₃ and Al₂O₃-SiC nanocomposite coatings are examined, in particular to understand the role of SiC on the transformation temperatures.

2. Experimental

The feed stock powders, containing 2/4/8 mass% (i.e., 2.5, 5 and 10 vol.%) of α -SiC (rest α -Al₂O₃) were made by high-energy attrition milling (mechanical alloying) and agglomeration. Agglomeration was carried out by spray drying process, which transformed the angular as-milled powders into spherical agglomerates. Typical particle size distribution of Al₂O₃-4SiC feed stock had following size characteristic: $d_{0.1} = 2.2 \mu\text{m}$, $d_{0.5} = 9.1 \mu\text{m}$, $d_{0.9} = 19.3 \mu\text{m}$.

All the coatings were prepared on mild steel substrates using HVOF and APS techniques. Flat (3.7 cm × 7.5 cm) and roller (4 cm diameter) geometries were used for the substrates. Grit blasting, for the substrate surface preparation, was carried out with 600–800 # (DIN) alumina grit at 5 bar pressure to get a surface roughness of 32–50 μm (R_z). In the preparation of all the investigated coatings, feed stocks were injected internally, with no tilt relative to the torch axis. The coating temperature, during spraying, was limited by blowing compressed air. The spray parameters are given in Table 1. Ni5Al or Ni20Cr (50 μm thick) was used for the bond layer. Pure alumina coatings prepared by HVOF (feed stock: $d_{0.5} = 5 \mu\text{m}$, $d_{0.9} = 22 \mu\text{m}$) and APS (classified, –32 μm powder) were used as reference. Four series of coatings of each composition were prepared with different thickness of top layer and bond coats. The characteristics of the coated specimens used are summarised in Table 2. The specimens are designated as A-*n* and S_{*m*}-*n*. ‘A’ and ‘S’ represents Al₂O₃ and Al₂O₃-SiC coatings, respectively. The subscript *m* signifies the mass% SiC in Al₂O₃-SiC coatings. ‘*n*’ indicate type of coating technique (*n* = 1–3: HVOF coating, *n* = 4: APS coatings) and bond coat (*n* = 2, 3: Ni20Cr, and *n* = 1, 4: Ni5Al bond coat): A-0 is APS alumina coatings without any bond coat.

Seifert PTS X-ray diffractometer (XRD) is used for diffraction measurements using Cu K α radiation at 50 kV and 39 mA with Ni filter and without monochromator. XRD measurements were carried out, on the coatings along with the substrate and peeled free-forms. Coatings with substrate were examined without any heat treatment and after heat treatment at 100 and 700 °C for different length of time, ranging from 0.5 to 5.5 hour (h), and air-cooled. Peeled free-forms, deposits with and without bond coat, were examined by XRD without heat treatment and after high

Table 1
Spraying parameters used during HVOF and APS process

	HVOF	APS
Gun	Top gun	Sulzer Metco 9MB (30 kW power level)
Fuel/plasma gas	Ethylene	Ar + H ₂
Spray distance (mm)	180 mm (Al ₂ O ₃ -SiC coatings) 200 mm (Al ₂ O ₃ coatings)	70 mm

Table 2

Characteristics of the specimens (all top/bond coating compositions are expressed in mass%)

Sample code	Sprayed material	Spray process	Top coat thickness	Bond coat (50 μm)
A-1	α -Alumina	HVOF	250 μm	Ni5Al
A-2	α -Alumina	HVOF	250 μm	Ni20Cr
A-4	α -Alumina	APS	250 μm	Ni5Al
A-0	α -Alumina	APS	250 μm	–
S ₂ -1	Agglomerate of (α -Al ₂ O ₃ + 2 mass% SiC)	HVOF	250 μm	Ni5Al
S ₂ -2	Agglomerate of (α -Al ₂ O ₃ + 2 mass% SiC)	HVOF	250 μm	Ni20Cr
S ₂ -3	Agglomerate of (α -Al ₂ O ₃ + 2 mass% SiC)	HVOF	1 mm	Ni20Cr
S ₂ -4	Agglomerate of (α -Al ₂ O ₃ + 2 mass% SiC)	APS	250 μm	Ni5Al
S ₄ -1	Agglomerate of (α -Al ₂ O ₃ + 4 mass% SiC)	HVOF	250 μm	Ni5Al
S ₄ -2	Agglomerate of (α -Al ₂ O ₃ + 4 mass% SiC)	HVOF	250 μm	Ni20Cr
S ₄ -3	Agglomerate of (α -Al ₂ O ₃ + 4 mass% SiC)	HVOF	1 mm	Ni20Cr
S ₄ -4	Agglomerate of (α -Al ₂ O ₃ + 4 mass% SiC)	APS	250 μm	Ni5Al
S ₈ -1	Agglomerate of (α -Al ₂ O ₃ + 8 mass% SiC)	HVOF	250 μm	Ni5Al
S ₈ -2	Agglomerate of (α -Al ₂ O ₃ + 8 mass% SiC)	HVOF	250 μm	Ni20Cr
S ₈ -3	Agglomerate of (α -Al ₂ O ₃ + 8 mass% SiC)	HVOF	1 mm	Ni20Cr
S ₈ -4	Agglomerate of (α -Al ₂ O ₃ + 8 mass% SiC)	APS	250 μm	Ni5Al

temperature (1000–1500 °C) heat treatment and cooling to room temperature in air unless specified otherwise.

3. Results and discussion

3.1. Phase constitution of the alumina-based coatings

3.1.1. Effect of SiC on the formation of alumina phases

X-ray diffractograms of HVOF sprayed Al₂O₃ (A-2) and Al₂O₃–SiC (S₂-2; S₄-2; S₈-2) coatings with Ni20Cr bond

coat are shown in Fig. 1. The phase constitution results of X-ray studies on all the investigated coatings are summarised in Table 3. All the coatings have shown the presence of α - and γ -phases. In addition, peaks corresponding to hexagonal SiC were observed in S₄-2 and S₈-2. SiC diffraction peaks were not detected in S₂-2 possibly due to its small amount below the detection limit of powder XRD technique. It is observed that the amount of α -phase is \sim 20% less in alumina (A-2) coating as compared to Al₂O₃–SiC (S-2 series) coatings. Thus, it appears that the presence of SiC, which was found to be present mostly as intra-granular phase (by

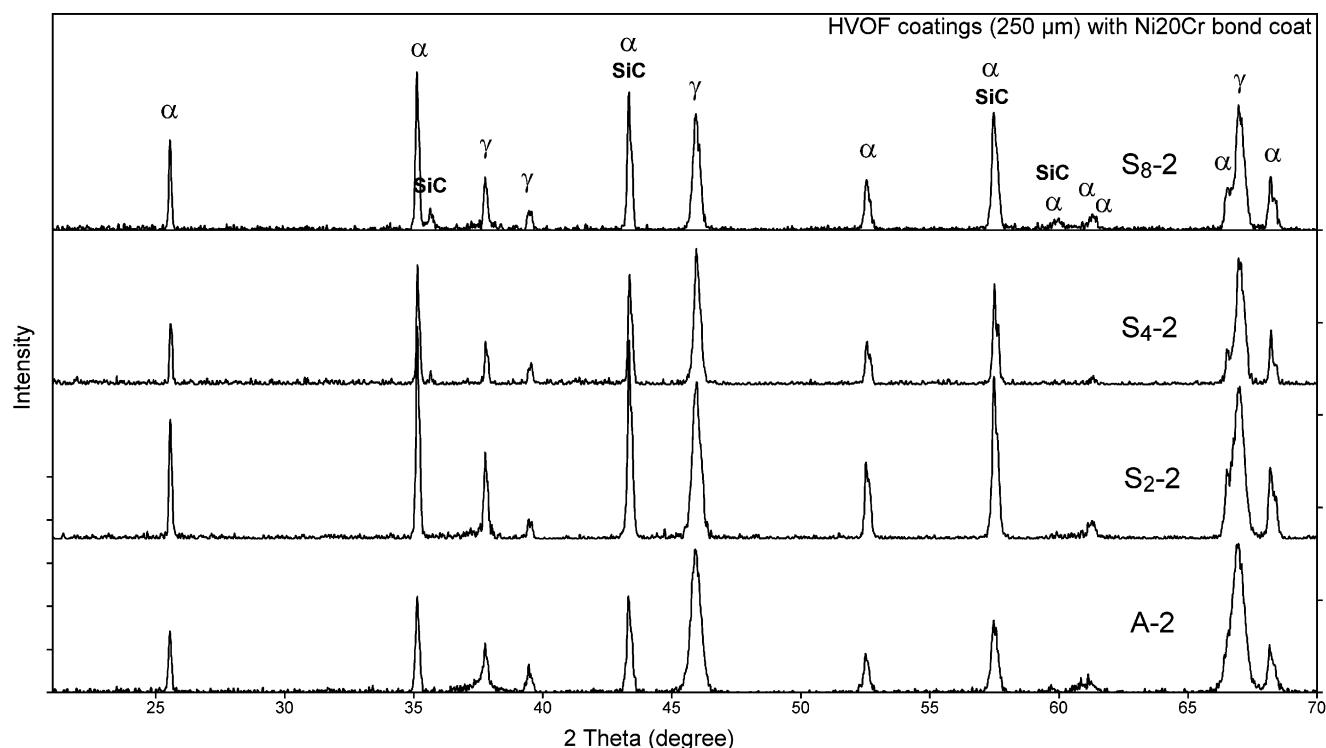


Fig. 1. XRD patterns of HVOF sprayed alumina-based coatings with Ni20Cr bond-coat (A-2; S₂-2; S₄-2; S₈-2 coatings).

Table 3

Phase analysis Al_2O_3 and Al_2O_3 -SiC as-sprayed coatings prepared using HVOF and APS process

Coating	Spraying technique	Bond layer	
		Ni20Cr	Ni5Al
Al_2O_3	APS	$\gamma + \alpha + \delta^a$	$\gamma + \alpha + \text{U} + \delta$
Al_2O_3	HVOF	$\gamma + \alpha$	$\gamma + \alpha + \text{U}$
Al_2O_3 -2% SiC	APS	—	$\gamma + \alpha + \text{U}$
Al_2O_3 -2% SiC	HVOF	$\alpha + \gamma$	$\alpha + \gamma + \text{U}$
Al_2O_3 -4% SiC	APS	—	$\gamma + \alpha + \text{U} + \text{SiC}$
Al_2O_3 -4% SiC	HVOF	$\alpha + \gamma + \text{SiC}$	$\alpha + \gamma + \text{U} + \text{SiC}$
Al_2O_3 -8% SiC	APS	—	$\gamma + \alpha + \text{U} + \text{SiC}$
Al_2O_3 -8% SiC	HVOF	$\alpha + \gamma + \text{SiC}$	$\alpha + \gamma + \text{U} + \text{SiC}$

^a Result for Al_2O_3 coating without any bond coating (sample A-0).

SEM), favors the formation of increased amount of α -phase in the HVOF coatings. Kozlova [7] has studied the effect of large number of second phase oxides (<2 mass%) on the amount of α - Al_2O_3 formed in plasma sprayed alumina coatings. It was found that addition of second phase oxides, such as MgO and ZrO_2 , which has higher melting temperature than alumina, give rise to a higher melt temperature and increased the α - Al_2O_3 content in the sprayed coating. On the other hand, oxides like Co_2O_3 , Fe_2O_3 , Cr_2O_3 , NiO, CaO, SiO_2 and TiO_2 , which decreased the melt temperatures, gave rise to lower amount α - Al_2O_3 than the pure alumina coatings. Since the melting point of SiC (2700 °C) is over 650 °C higher than alumina, it is likely that it raises the amount of unmelted seeds resulting in the formation of greater amount α -phase in the composite coatings.

3.1.2. Alumina phases in HVOF and APS coatings

Typical XRD patterns of Al_2O_3 -SiC composite coatings, prepared by HVOF and APS techniques are shown in Fig. 2. The coatings prepared by both the techniques have shown the presence of α , γ and U phases with varied amount of α - and γ -phases. HVOF sprayed coatings showed α - Al_2O_3 as the dominant phase. On the contrary, all the APS nanocomposite ceramic coatings have shown higher amount of γ -phase and only ~10–15% of α - Al_2O_3 phase (Figs. 2 and 3). Similar results were obtained for Al_2O_3 coatings. Additionally, presence of δ -phase is observed in Al_2O_3 coatings prepared by APS.

It has been suggested that α - Al_2O_3 is formed in deposits by nucleation from unmelted seeds and that γ - Al_2O_3 is the product of the solidification of droplets, which do not contain these nuclei [12]. In plasma spraying, higher power and finer particles are reported to yield more melting and consequently more γ -phase. On the contrary, lower power and coarser particles favor more unmelted seeds and greater amount of α -phase. Flame temperatures of APS technique are much higher than HVOF spray technique and hence availability of partially melted (may be >99% melted with a fine unmelted core) α - Al_2O_3 seeds is much less in APS technique. The particle size remaining constant, greater amount of α -phase in HVOF Al_2O_3 -SiC coatings may be attributed to more unmelted alumina seeds due to lower flame temperatures as compared to APS.

As compared to APS alumina coating, greater amount of α - Al_2O_3 in alumina coating prepared by HVOF is interesting. The particle size of HVOF feed stock ($d_{0.5} = 5 \mu\text{m}$,

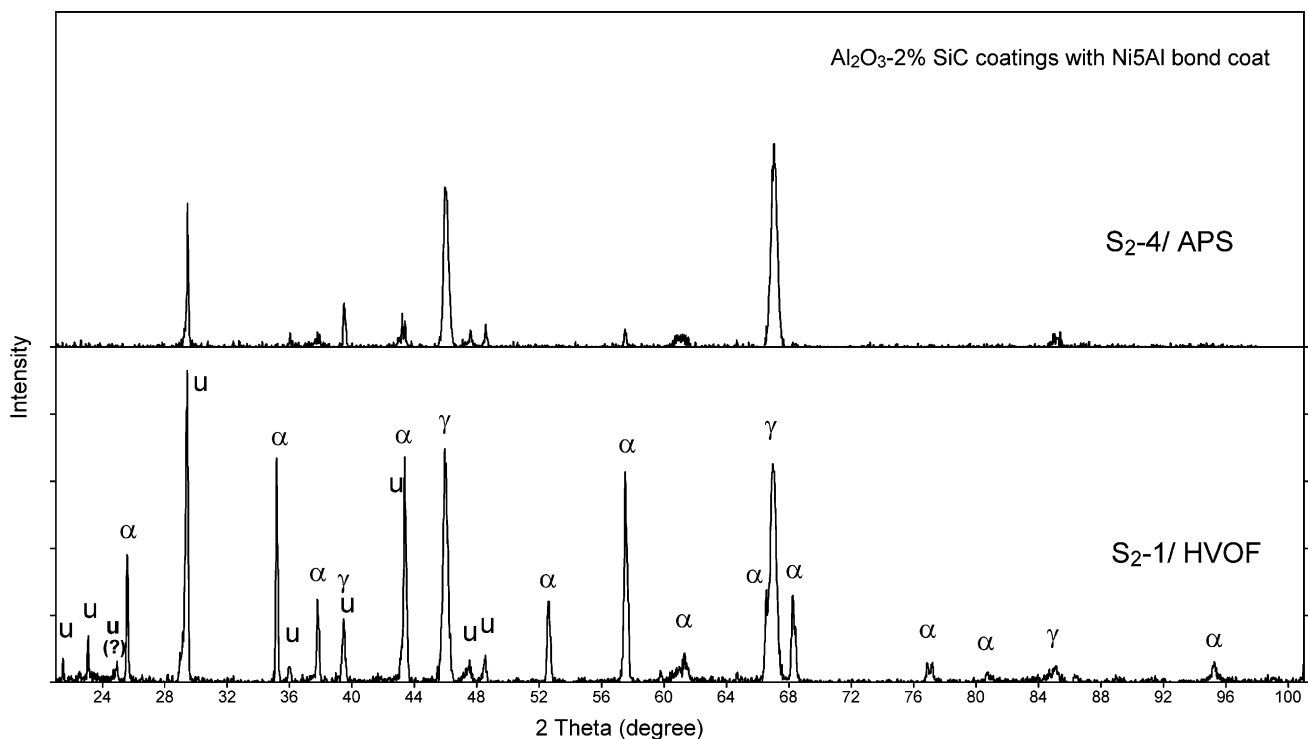


Fig. 2. XRD patterns of HVOF and APS Al_2O_3 -2% SiC nanocomposite coatings with Ni5Al bond-coat.

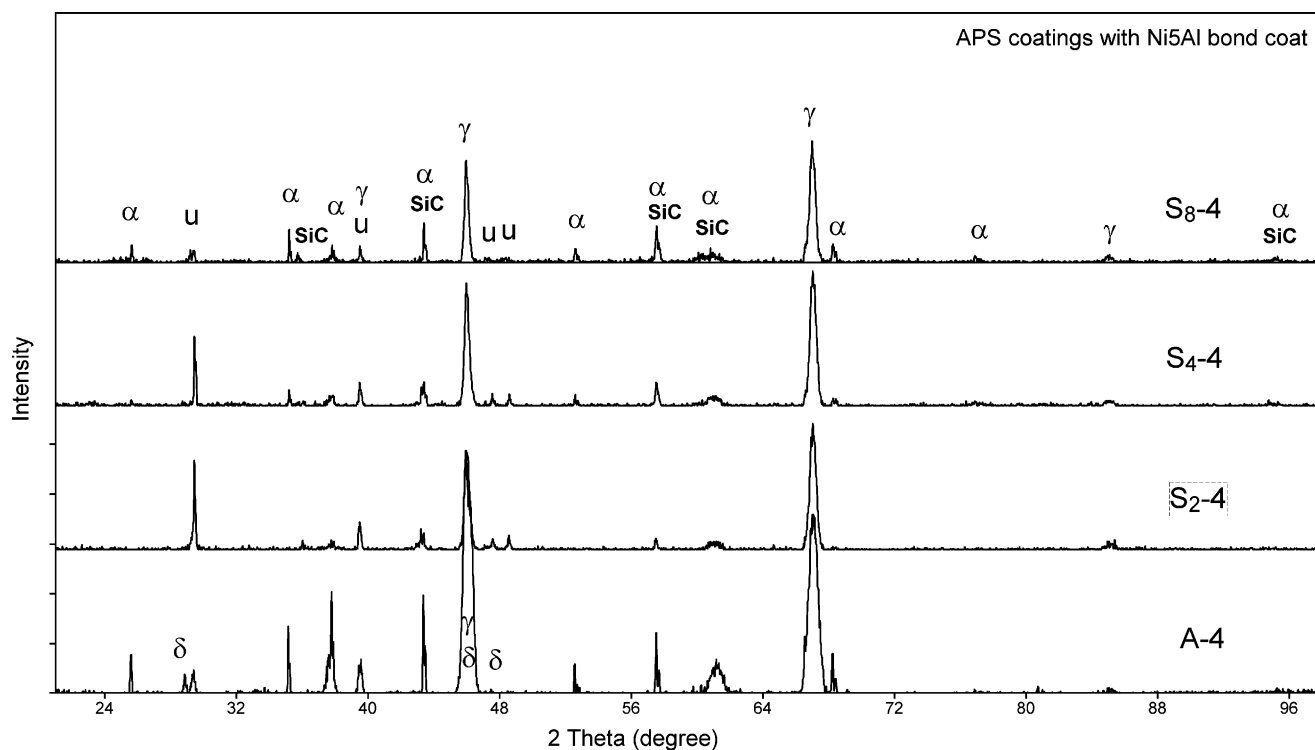


Fig. 3. XRD patterns of APS alumina and Al_2O_3 -SiC nanocomposite coatings with Ni5Al bond-coat.

$d_{0,9} = 22 \mu\text{m}$) was much smaller to the feed stock used for APS (classified $-32 \mu\text{m}$ powder). This suggests that in HVOF even finer particles can yield more unmelted seeds and greater α - Al_2O_3 due to lower, residence time (due to higher gas velocities) and flame temperature (HVOF flame temperature used was $\sim 2900^\circ\text{C}$).

3.1.3. Effect of coating thickness

Thicker, 1 mm coatings have more α -phase (S_2 -3) compared to coatings of $250 \mu\text{m}$ thickness (S_2 -2). Alumina being thermally insulating, an increase in the heat content of successive passes, result in surface temperature higher than about 1200 K, nucleating in higher amounts of α - Al_2O_3 phase on the surface layers, an effect similar to preheating the substrate [7,14].

3.2. $\gamma \rightarrow \delta \rightarrow \theta \rightarrow \alpha$ phase transformation in HVOF coatings

For Al_2O_3 , it has been established that on heating boehmite (AlOOH), the boehmite $\rightarrow \gamma \rightarrow \delta \rightarrow \theta \rightarrow \alpha$ phase transformations take place. The transformation temperatures for boehmite $\rightarrow \gamma$, $\gamma \rightarrow \delta$, $\delta \rightarrow \theta$ and $\theta \rightarrow \alpha$ phase transformations are reported to be about 450, 750, 1000 and 1200°C [11,15]. Kozlova [7] reported that alumina in APS coatings undergoes similar transformations at somewhat higher temperatures. The transformation starts at about 1000°C , and the transformation temperatures are time dependent.

In order to see the nature of this transformation in HVOF Al_2O_3 and Al_2O_3 -SiC nanocomposite coatings, A-2 and S_4 -2 peels were subjected to heat treatment between 700 and 1500°C . Figs. 4 and 5 show the X-ray diffractograms of A-2 and S_4 -2 peels after heat treatment between 1000 and 1500°C . Parts of these figures are shown in Figs. 6 and 7 for better resolution. The overall findings are summarised in Table 4. The U phase was observed in the heat treated peels at all the temperatures.

3.2.1. Alumina coatings

A-2 sample showed the presence of γ -phase at 700°C . At 1000°C , no γ -phase is observed and δ -phase appears in the coating indicating that $\gamma \rightarrow \delta$ transformation is completed between 700 and 1000°C . δ -Phase shows up in the sample heat treated at 1100°C and not at 1200°C . At 1200°C and above, only α and U phases are observed. No change in the intensity of α -phase is observed between 1200 and 1500°C .

These above findings are only in partial agreement with the finding of Kozlova [7]. No θ -phase is observed in any of the heat treated Al_2O_3 coating between 1000 and 1500°C . Furnace cooling of some of the heat treated samples also did not result in the formation of θ -phase. Gansert et al. [13] have reported that θ -phase is not always observed. In our studies shifting of δ - Al_2O_3 peaks with rise in temperature was noticed. The distinct observation was a progressive shift of δ -phase (042) peak by 0.1° (2θ) with a rise in temperature from 1000 to 1100°C in A-2 peels. This may be due to changes in cation positions, with high degree of pos-

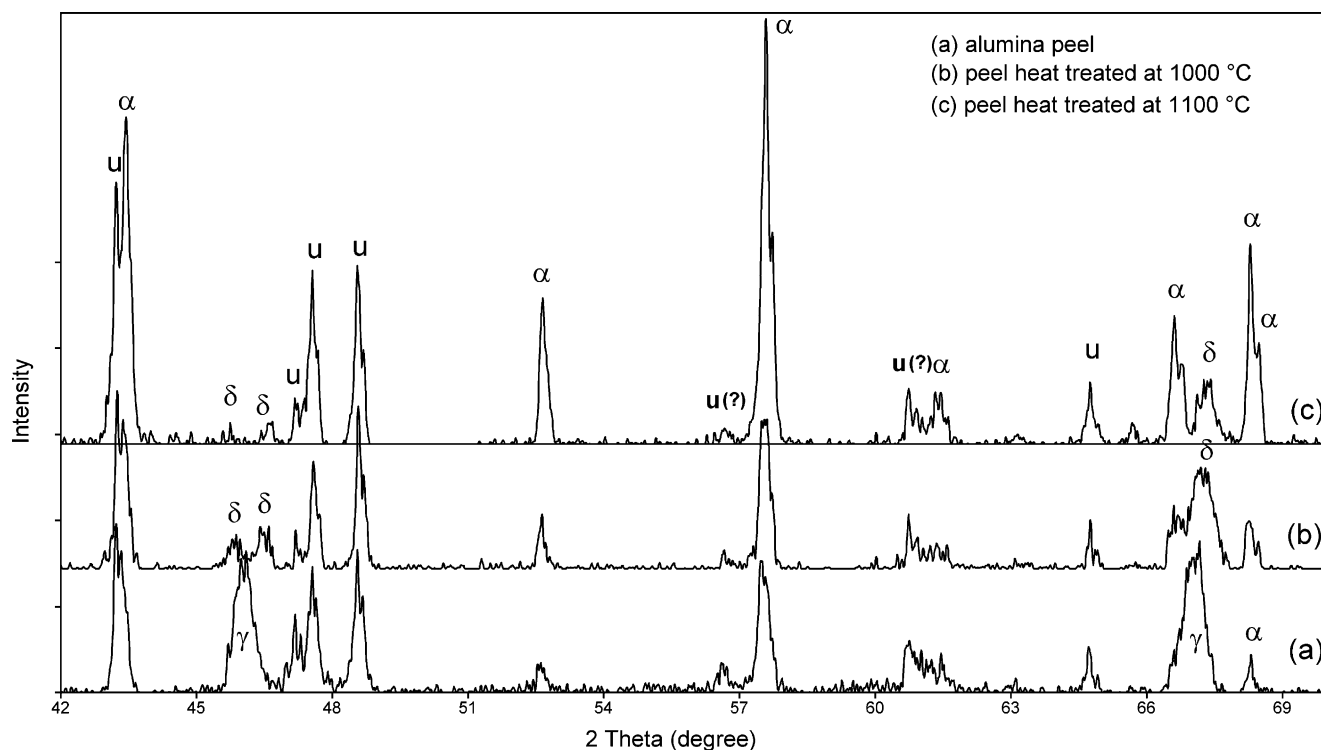


Fig. 6. XRD patterns of alumina (HVOF) coatings; as-peeled, 1000 and 1100 °C heat treated.

isted. The reason for not observing θ - Al_2O_3 in the present study may be one or more of the following: (a) stability of θ -phase in narrow temperature region between 1100 and 1200 °C, (b) prolonged heating required for the forma-

tion, (c) preparation of the coating by a different technique, and (d) possible error in the indexing of θ -phase by Kozlova, since δ -phase indexing of Repelin [16] include all the θ lines of Kozlova. More detailed studies are required to

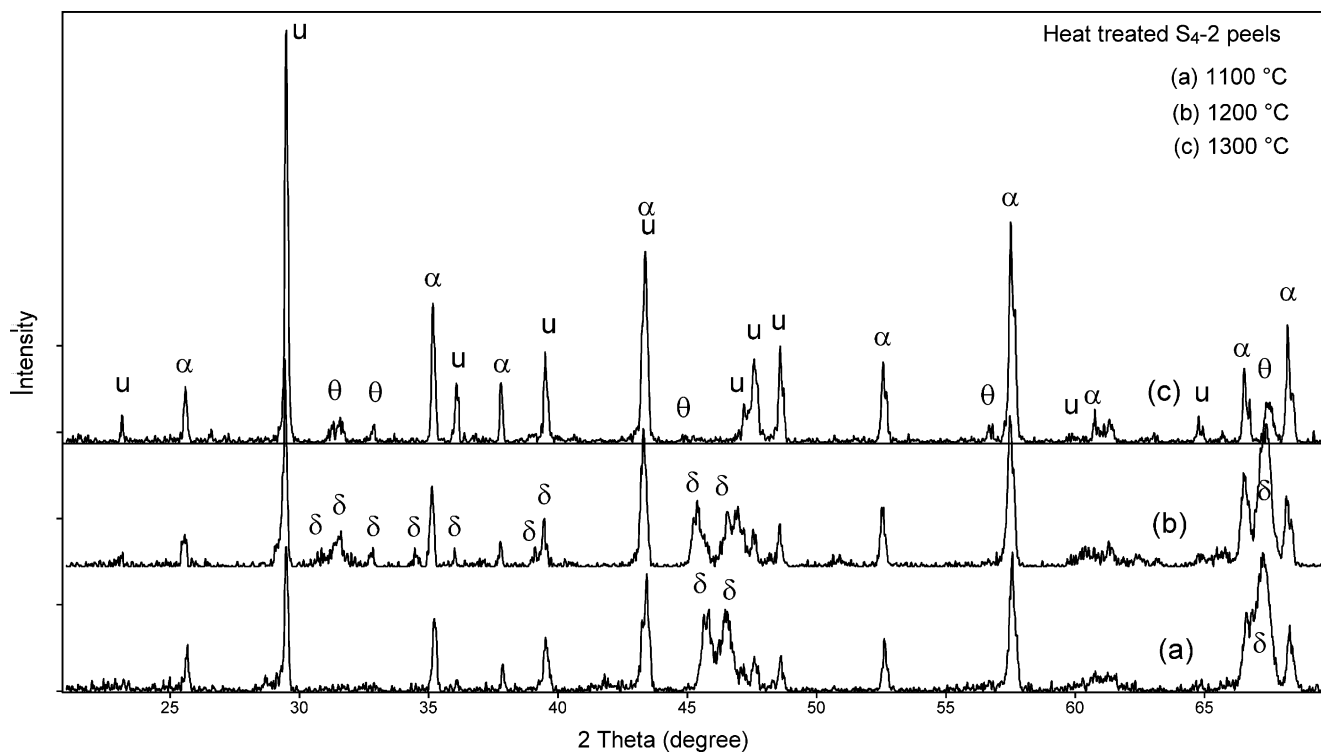


Fig. 7. XRD patterns of Al_2O_3 -4% SiC nanocomposite (HVOF) coatings; peels heat treated at 1100–1300 °C.

Table 4

Thermal stability of alumina phases in Al_2O_3 and Al_2O_3 –SiC coatings prepared by HVOF process on mild steel substrate with bond layer

Specimen detail	Annealing			Phase present based on X-ray analysis									
	Temperature ($^{\circ}\text{C}$)	Time (h)	Cooling	Al_2O_3 coating				Al_2O_3 /SiC (4 mass%) composite coating					
				α	γ	δ	U	α	γ	δ	U	θ	
As-sprayed	No	–	–	α	γ	–	–	α	γ	–	–	–	
Powdered peels ^a	No	–	–	α	γ	–	–	α	γ	–	–	–	
Peeled free-form	No	–	–	α	γ	–	U	α	γ	–	U	–	
	700	0.2	–	α	γ	–	U	α	γ	–	U	–	
	1000	1	Air	α	–	δ	U	α	γ	–	U	–	
	1000	1	Furnace	α	–	δ	U	α	γ	–	U	–	
	1100	1	Furnace	α	–	δ	U	α	–	δ	U	–	
	1200 ^b	1	Air	α	–	–	U	α	–	δ	U	–	
	1200	1	Air	α	–	–	U	α	–	δ	U	–	
	1300	1	Air	α	–	–	U	α	–	–	U	θ	
	1400	1	Air	α	–	–	U	α	–	–	U	–	
	1500	1	Air	α	–	–	U	α	–	–	U	–	

Bond layer, Ni20Cr; coating thickness, 250 μm .^a Powder size, 90 μm .^b The sample is preheated at 1100 $^{\circ}\text{C}$.

solve this problem and these are beyond the scope of this paper.

3.2.2. Al_2O_3 –SiC nanocomposite coatings

In S₄-2 coating, the observed phase transformations are as follows: $\gamma \rightarrow \delta \rightarrow \theta \rightarrow \alpha$ at 1100 $^{\circ}\text{C}$ /1 h; 1300 $^{\circ}\text{C}$ /1.75 h; 1400 $^{\circ}\text{C}$ /1 h, respectively. The progressive shift of δ -phase (042) diffraction peak by 0.1 $^{\circ}$ (2 θ) was observed in S₄-2 coating also but between 1100 and 1200 $^{\circ}\text{C}$. It is interesting to note that θ -phase appears in the Al_2O_3 –SiC nanocomposite coatings although at a temperature which is about 200 $^{\circ}\text{C}$ higher than the temperature reported by Kozlova [7]. Presence of SiC in S₄-2 has raised the $\gamma \rightarrow \delta$ phase transformation temperature by $\sim 100^{\circ}\text{C}$ and $\theta \rightarrow \alpha$ transformation temperature by $\sim 200^{\circ}\text{C}$. All the transformations, $\gamma \rightarrow \delta \rightarrow \theta \rightarrow \alpha$, as reported for boehmite are observed in the nanocomposite Al_2O_3 –SiC coatings prepared by HVOF. Our findings regarding transformation temperatures of alumina polymorphs are in the same line as coarse boehmite [15]. It is found that SiC consistently (in all investigated S-series coatings) increased the transformation of metastable alumina polymorphs to stable α -phase by 200 $^{\circ}\text{C}$. Rise in transformation temperatures have been reported in plasma sprayed coatings containing Cr_2O_3 [13].

4. Conclusions

1. Presence of SiC in Al_2O_3 nanocomposite ceramic coatings increased the amount of α -phase formation in the HVOF coatings.
2. Al_2O_3 and Al_2O_3 –SiC nanocomposite coatings made by HVOF and APS techniques contain α , γ and U phases. Relatively α -phase is more in HVOF sprayed coatings due to the presence of higher amount of

unmelted seeds. Thicker HVOF coatings (1 mm) of Al_2O_3 –SiC have more α -phase compared to coatings of less thickness (ex: 250 μm).

3. The metastable alumina phases in Al_2O_3 –SiC nanocomposite coatings made by HVOF shows $\gamma \rightarrow \delta \rightarrow \theta \rightarrow \alpha$ transformations similar to boehmite. Presence of SiC in Al_2O_3 has raised the $\gamma \rightarrow \delta$ phase transformation temperature by $\sim 100^{\circ}\text{C}$ and $\theta \rightarrow \alpha$ transformation by $\sim 200^{\circ}\text{C}$.

Acknowledgements

The work described in this paper was carried out whilst the author was on leave from Tata Steel, with the Laboratory for Surface Engineering, University of Siegen. Thanks are due to Tata Steel for providing the opportunity and the Laboratory for Surface Engineering (Prof. H. Weiss) for provision of facilities.

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