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Research Article

High Velocity Oxy-Fuel Spray Formed Bulk Aluminium Matrix Composites-Development and Characterization

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Abstract

In the present study, bulk Aluminium matrix composites (AMCs) reinforced by alumina (Al_2O_3) particulates (Al/Al_2O_3) are developed by High Velocity Oxy-fuel (HVOF) thermal spray technique. The HVOF spray technique has been used by many researchers to deposit composite coatings, but there is less available literature discussing the development of bulk metal matrix composites by this technique. The composite powders, as a feed stock of HVOF spraying, was prepared by ball milling of pure Al powders with 15 wt.%, 20 wt.% and 25 wt.% Al_2O_3 particles. The feedstock was deposited on to a die steel metallic substrate. The deposited composite material was then separated from the die steel. Microstructures of the composite powders and the newly developed composite materials were analyzed by Optical Microscopy (OM), Scanning Electron Microscopy (SEM)/Energy Dispersive Spectroscopy (SEM/EDS) and EDS elemental mapping techniques. X-ray Diffraction (XRD) analysis was done to reveal the phases present in the as-sprayed specimens. Porosity and hardness value of the composites was also measured. The study showed the possibility of preparing bulk aluminum metal matrix composites of larger size by HVOF spray technique.

Keywords*: Metal matrix composites, pure Al powder, Al_2O_3 particulates, thermal spraying, HVOF.*

1. Introduction

Metal matrix composites (MMCs) reinforced with ceramic particulates offer significant advantages over pure metals and alloys. MMCs tailor the best properties of the two components, such as ductility and toughness of the matrix and high modulus and strength of the reinforcement [Kevorkijan et al, (1999); Kelkar et al, (2001); Evans et al, (2006)]. The outstanding properties of these materials enable them to be used for numerous applications such as automotive, aerospace and military industries [Valiev et al,(2000); He,(2008); Crainic,(2002); Rawal,(2001)]. MMCs can be divided into three categories: particle reinforced MMCs, short fiber reinforced MMCs and continuous fiber reinforced MMCs. Of these three categories, the fabrication cost of particulate reinforced MMCs is quite low [Kevorkijan et al, (1999)], which makes them attractive and commercially viable to consider for industrial applications.

Processing routes for MMCs are categorized into two types: solid state processing and liquid state processing [Kevorkijan et al, (1999)]. Solid state processing is typically a powder metallurgy based process, in which the matrix powder and reinforcement particles are mixed together and compacted to form a bulk shape. Successful investigations on solid state processing include powder metallurgy [Thostenson et al, (2005); He et al, (2000); Kang et al,(2004)], high-energy milling [Ma et al, (1996)] and severe plastic deformation [Suryanarayana et al; Sherif et al, (1998); He et al, (1998); Valiev et al, (1996)]. In case of powder metallurgy, it has size limitation resulting from the preparation of molds and incurs high costs [Ko et al, (2009)]. Spray techniques allow fabricating coatings and bulk materials with compositions and structures that are not reachable with other processing techniques [Sing et al, (2000); Sahin et al, (2003); Hashim et al, (2002); Gui et al, (2004); Steenkiste et al, (2004)]. These methods consist of melting material feedstock to accelerate and propel heated particles toward a substrate where rapid solidification and thickness build-up occur. The thermal spray processes have been considered to deposit the coatings for protection against the high-temperature induced degradations. To produce bulk MMCs these techniques are still under investigation and have also become the subject of many research projects. The High Velocity Oxy-fuel (HVOF) spray technique has been used by many researchers to deposit composite coatings. However, scarce data is available discussing the development of bulk metal matrix composites by this technique.

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HVOF spraying is a relatively new and rapidly developing thermal spray technology for depositing surface coatings to combat high-temperature corrosion. The HVOF coatings have relatively low porosity, high hardness, high abrasive resistance, good wear resistance with a strong ability to resist high-temperature corrosion [Guilemany et al, (2006)]. The HVOF process is based on a combination of thermal and kinetic energy transfer, i.e., the melting and acceleration of powder particles, to deposit the desired coating. The HVOF unit uses an oxygen-fuel mixture consisting of propylene, propane, or hydrogen and even kerosene depending on user's coating requirements to produce the highest quality coatings. Fuel gases are mixed in a proprietary siphon system in the front portion of the HVOF gun. Powder particles of the desired coating material are fed axially into a hot gas stream, then into a spray gun, where they are melted and propelled to the surface of work piece to be coated [Tan et al, (1999)]. The thoroughly mixed gases are ejected from the nozzle and ignited externally to the gun. Ignited gases form a circular flame configuration which surrounds the powdered material as it flow through the gun. Combustion temperature is between 2760°C to 3315°C depending on fuel. The circular flame shapes the powder stream to provide uniform heating, melting, and acceleration (Fig.1 [Knorris]).

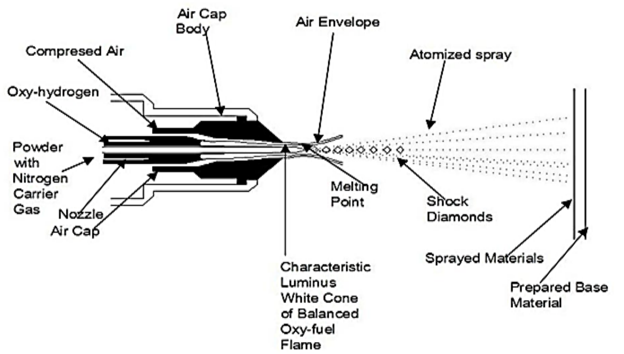


Figure 1- The High Velocity Oxy-Fuel (HVOF) Process [Knorris]

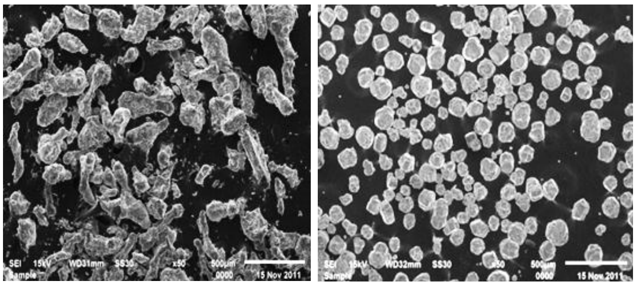
Aluminum matrix composites (AMCs) have attracted considerable attention because of their light weight, high strength, high stiffness, and wear resistance [Schmidt et al, (2011); Alizadeh et al, (2010); Rezayat et al, (2012); Liu et al, (2013)]. Aluminium is the most popularly used matrix material. Its low density, capability to be strengthened by precipitation, good corrosion resistance and high thermal and electrical conductivity are some of the features which make it quite attractive. AMCs have been widely studied and are now used in sporting goods, electronic packaging, armours and automotive industries. They offer a large variety of mechanical properties depending on the chemical composition of the Al-matrix. They are usually reinforced by Al_2O_3, SiC, C but SiO_2, B, BN, B_4C, AlN may also be considered [Singh et al, (2012)]. A series of AMCs reinforced with Alumina particulates have been developed by Duralcan USA, Division Alcan Aluminium corporation, San Diego, California [Higgins et al, (1986)]. The choice of alumina as the reinforcement in aluminium composite is primarily meant to use the composite in missile guidance system replacing certain beryllium components because structural performance is better without special handling in fabrication demanded by latter's toxicity [Wei et al, (1992)].

It has been learnt from the literature that Al matrix composites are the material candidate for structural applications where weight saving is of primary concern. The objective of the present research is to explore the possibility of developing bulk AMCs by HVOF spray technique. In the current investigation, bulk Al-Al_2O_3 composites with 15 wt.%, 20 wt.% and 25 wt.% Al_2O_3 powder as the reinforcement material are produced. The in-depth characterization of the prepared composites was studied by Optical microscopy (OM), X-ray Diffraction (XRD) analysis, Scanning Electron Microscopy (SEM)/ Energy Dispersive Spectroscopy (SEM/EDS) and EDS elemental mappings. Porosity and hardness value of the composites are also reported.

2. Experimental Procedure

2.1 Starting Material

Pure aluminium with 99.9% purity and 75 µm particle size was selected as the matrix material. Al_2O_3 particulates with 99.7% purity and 45 µm particle size were used as reinforcement material. Both the powders used in this study were commercially available. The SEM morphology of pure Al powder and Al_2O_3 particulates is shown in Fig. 2. The Al powder particles were found to be longitudinal and irregular in shape and size [Fig.2 (a)].The Al_2O_3 particulates were found to have spherical morphology. All the particles were regular in shape [Fig. 2(b)]. The Al matrix powder was blended with 15 wt. %, 20 wt. % and 25 wt. % Al_2O_3 particles.

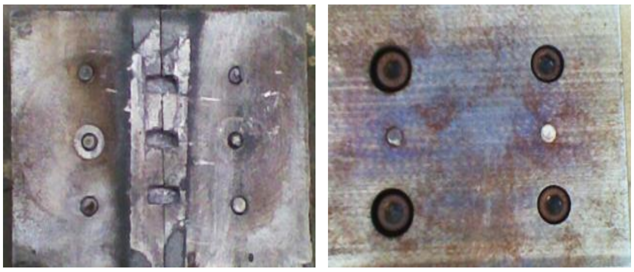


(a) (b)

Figure 2 - SEM morphology of (a) Al powder (b) Al_2O_3 powder

2.2 Sample Preparation

Selection of substrate material as die for fabricating composites has been done after exhaustive literature survey. Die steel with composition of 1% C, 1% Mn, 5% Cr, 0.3% Ni, 1% Mo, and 0.3% V and remaining Fe was selected for fabrication of bulk material (Fig. 3). This die steel is used in punching operations where it is required to separate the two materials having different mechanical properties. The dimensions of the die steel were taken as 100 mm × 77 mm ×5 mm. Three rectangular equally spaced slots of 10 mm × 5 mm × 5 mm were cut about the transverse axis [Fig. 3 (a)]. The die was cut into two halves along the center line and is supported by a plate of dimensions 100 mm×77 mm×12 mm with 4 bolts and two aligning keys. This was prepared for the easy removal of the composite material.



(a) (b)

Figure 3- Die Steel Substrate (a) front view, (b) rear view

2.3 Powders Preparation

Three types of powder compositions namely Al-15%(Al_2O_3), Al-20%(Al_2O_3) and Al-25%(Al_2O_3) were selected for HVOF spraying on the given die steel. Homogeneous dispersion (mechanical alloying) of matrix and dispersed phase was achieved by using ball milling apparatus for 6 hours. The matrix Al phase was blended with Al_2O_3 particulates phase by weight. The mixing was performed at Metallizing Equipment Company Pvt. Ltd., (MECPL) Jodhpur. Ball milling conditions are shown in Table 1.

Table 1 - Ball Milling Conditions

Mill jar	Stainless steel
Milling media	Stainless steel
Atmosphere	Air
Weight ratio of media to powder	10:01
Mill times	6 hrs

2.4 Development and Characterization of AMCs

The composites were then developed by HVOF thermal spraying at MECPL, Jodhpur (India) with their commercial HVOF (HIPOJET-2700) apparatus operating with oxygen and liquid petroleum gas (LPG) as input gases and nitrogen as carrier gas. The spraying parameters adopted for the HVOF spray process are given in Table 2. Composite thickness was kept in a range of 4±2 µm. After successfully obtaining the required thickness of the composite, the die was cooled with pressurized nitrogen gas and the difference in coefficient of thermal expansion resulted in easy separation of bulk material from substrate. The ingots of 10 mm× 5 mm×5 mm were obtained by the HVOF spraying.

The microstructural characterization of the metallographically polished samples of Al-Al_2O_3 composites was carried out using inverted optical microscope. The prepared samples were hot mounted in BAINMOUNT-H (Hydraulic Mounting Press, Chennai Metco Pvt. Ltd., Chennai, India) with transoptic powder. The samples were polished using 180 grit silicon carbide paper followed by 220, 320, 400, 600 and 1000 grades of emery paper. Final polishing was carried out with 1- µm diamond paste. The polished Al-Al_2O_3 samples were etched with Keller's Reagent (2.5 ml Nitric acid (HNO_3), 1.5 ml Hydrochloric acid (HCl), 1ml Hydrofluoric acid (HF) and 95ml water) before optical observation. Porosity measurements were made by the image analysis software Envision 3.0.

X-ray diffraction (XRD) analysis of the composites developed was carried out using a Bruker AXS D-8 Advance Diffractometer (Germany) with CuKα radiation and nickel filter at 20 mA under a voltage of 35 kV. The specimens were scanned with a scanning speed of 1Kcps in 2θ range of $20^o$to 110^o and the intensities were recorded at a chart speed of 1cm/min with 2^o/min as Goniometer speed. The elements present in the composite composition are incorporated into the software. The diffractometer interfaced with Bruker DIFFRAC plus X-Ray diffraction software directly provides the phases present on the respective peaks. The SEM/EDS analysis of the samples was carried out by JEOL scanning electron microscope (SEM), JEOL JSM 6610LV, having resolution of 3 nm and magnification 300,000X equipped with energy dispersive spectroscopy (EDS) software to characterize the surface morphology and composition of the developed composite material. The SEM micrographs along with EDS spectrum were taken with an electron beam energy of 20 keV.

A microhardness tester (model-WILSON) with digital display was used to measure the microhardness of the prepared specimens. An indenting load of 300 gf was applied on the metal matrix avoiding ceramic particles. Three measurements were taken for each case to ensure the repeatability. Indentation load applied during the microhardness testing did not produce any sign of cracking to be measured and analyzed.

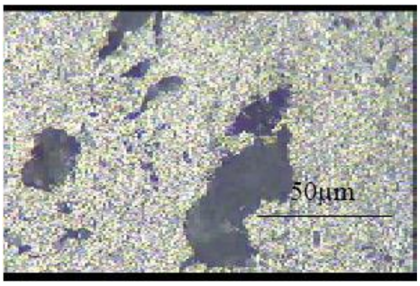
Table 2: Spray parameters as employed during HVOF spraying

Oxygen flow rate (SLPM)	240-250
Fuel (LPG) flow rate (SLPM)	50-55
N_2 flow rate (SLPM)	15-20
Oxygen pressure (kg/cm^2)	9
Fuel pressure (kg/cm^2)	5.6
Powder pressure (kg/cm^2)	3
Spray distance (mm)	250

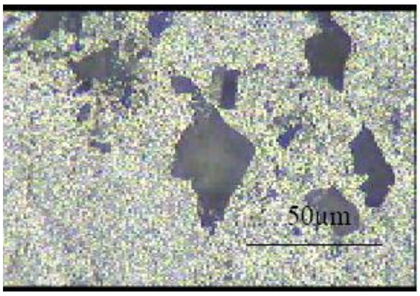
3. Results and Discussion

3.1 Optical Microscopy

The microstructures for the prepared bulk material as seen under an optical microscope are shown in Fig 4. Fig. 4 (a) with 15wt% reinforcement shows very dark Al_2O_3 particulates distributed in Al matrix. The particulates are of irregular shape. In case of 20% Al_2O_3, uniformly embedded alumina particles are observed in the Al matrix [Fig. 4 (b)]. On the other hand in Al-25%Al_2O_3 composite, the clusters of alumina particles at various places are revealed under the microscope [Fig. 4 (c)]. [Bansal et al, (2011)] developed Al-Al_2O_3 composites by stir casting technique. The content of the alumina particles was up to 10 %. The author also observed the uniform distribution of alumina particles in aluminum matrix. Irregular shape of alumina particles was may be due to breakage of particles during ball milling. With the increase in the reinforcement content the area fraction of the dispersed phase increased as revealed by the optical micrographs thus resulting in clustering of alumina particulates at various places in Al matrix [Fig. 4 (c)].



(a)



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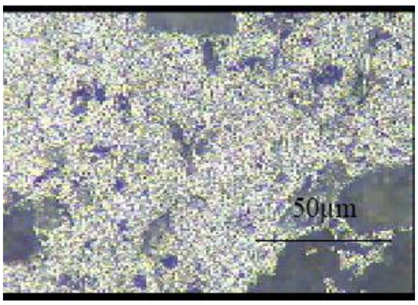
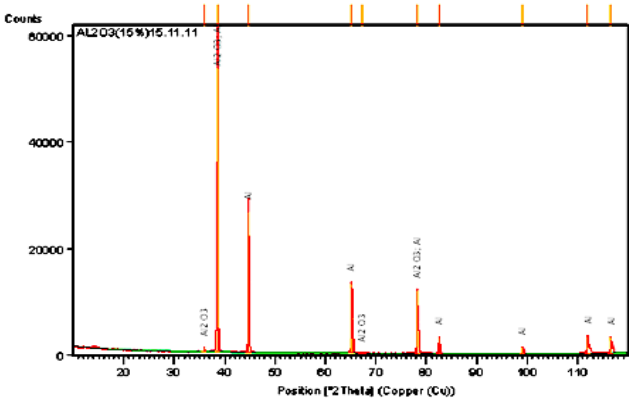


(c)

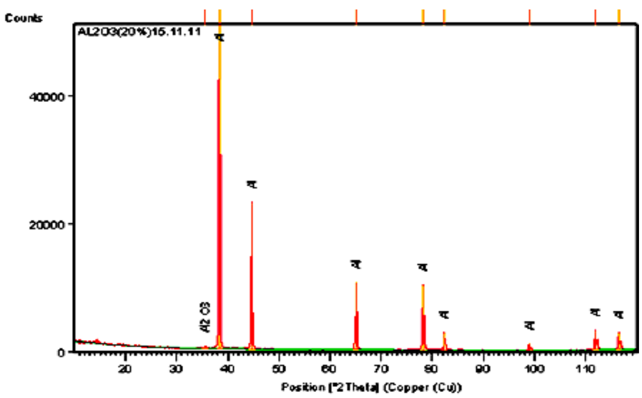
Figure 4- Optical micrographs of HVOF spray formed bulk Al matrix composites (a) Al-15%Al_2O_3 (b) Al-20%Al_2O_3 (c) Al-25%Al_2O_3

3.2 XRD Analysis

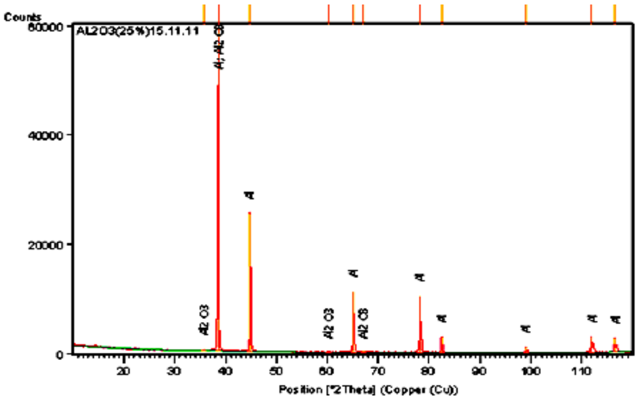
The XRD diffractograms for the surfaces of the bulk material prepared by HVOF spray process are shown in Fig. 5 on reduced scales. The AMCs developed with 15%Al_2O_3, and 25%Al_2O_3 indicate the presence of Al as a very strong intensity phase and Al_2O_3 as a medium intensity phase. The composite with 20%Al_2O_3 revealed the presence of Al as a very strong intensity phase and weak alumina phase [Fig. 5 (b)]. [Spencer et al, (2009)] studied the behaviour of Al-Al_2O_3 composite coating by cold spray process on Mg substrate. The authors also observed that the XRD compositions showed lesser volume fraction of Al_2O_3 in the as-sprayed coating than that in the source powder mixture. The authors found that the relative deposition efficiency of the Al_2O_3 powder is lower than that of the Al powder. It showed that as more Al_2O_3 is added to the source powder mixture, the relative deposition efficiency of Al_2O_3 decreases. [Yin et al, (2008)] also revealed that most of α-Al_2O_3 particles in the starting powders converted into γ-Al_2O_3 after spraying. In the present study, the XRD results are well supported by the subsequent SEM/EDS (surface as well as cross-sectional) and by EDS elemental mapping analysis in the respective cases. Strong presence of Al in the composite surface attributes that instant oxidation of the aluminium phase has not taken place during the deposition with HVOF spray technique. Whereas [Yin et al, (2008)] on studying the Al-Al_2O_3 composite coating with plasma spray process hardly found the presence of aluminum in the coating surface.



(a)



(b)



(c)

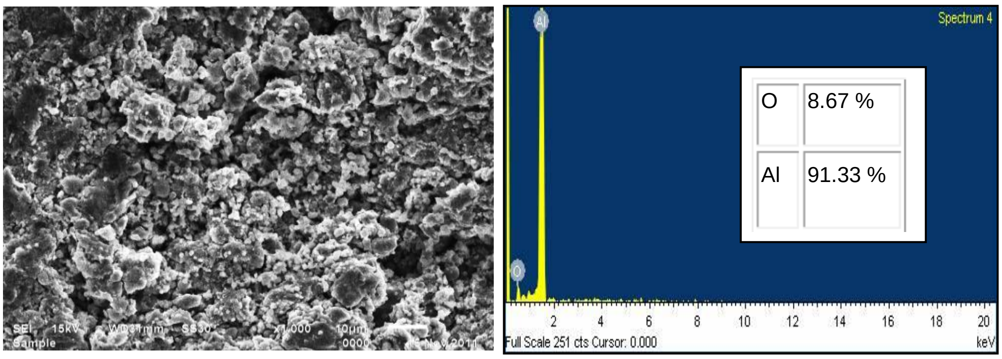
Figure 5- XRD diffractograms of HVOF spray formed bulk Al matrix composites (a) Al-15%Al_2O_3 (b) Al-20%Al_2O_3 (c) Al-25%Al_2O_3

3.3 SEM/EDS Analysis

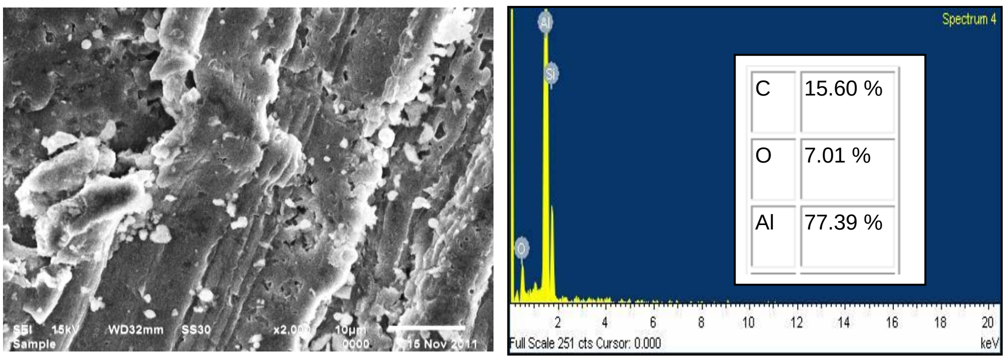
The SEM/EDS morphology of the Al-15%Al_2O_3 bulk composite prepared by HVOF thermal spray process is shown in [Fig. 6 (a)]. The bulk material is found to have splat-like morphology. The splats are interconnected with each other, however superficial voids are also present at some places. The morphology is a typical characteristic feature of thermal spray coatings. The dense microstructure seems to be formed. The EDS analysis shows the presence of mainly Al (91.33%) in the composite. Some amounts of O were found to present indicating the presence of aluminum oxide. [Yin et al, (2008)] carried out microstructural investigation of as-sprayed Al-Al_2O_3 composite coating by plasma spray process. The authors also observed dense coating with layered structure and less large volumetric pores. Moreover, the composite coating showed distinct lamellar structure and developed splat interface. However, in the current investigation, the HVOF spray formed composite is free from any defects.

Fig. 6 (b) shows the SEM morphology of Al-20%Al_2O_3 bulk composite. The morphology shows the dispersion of alumina particles in Al matrix. The presence of distinct layers of the deposits can be visualized. Figure shows the homogenous distribution of particles on the layered surface. Clustering of alumina particles in some regions is also visible. The EDS analysis indicates the presence of mainly Al along with significant amount of C in the matrix. Small amounts of O are also present. [Safai and Herman et al, (1977)] suggested that during spraying, in the case of Al/Al_2O_3 composite, good ductility and thermal conductivity of metallic Al contributed to minimize localized solidification of impacted droplets and even delay its initiation until after the spreading is nearly completed. [Sampath et al, (1999)] opined that the improved spreading and deformation of impacted droplets will result in developed deposition of coating and enhanced interlamellar bonding.

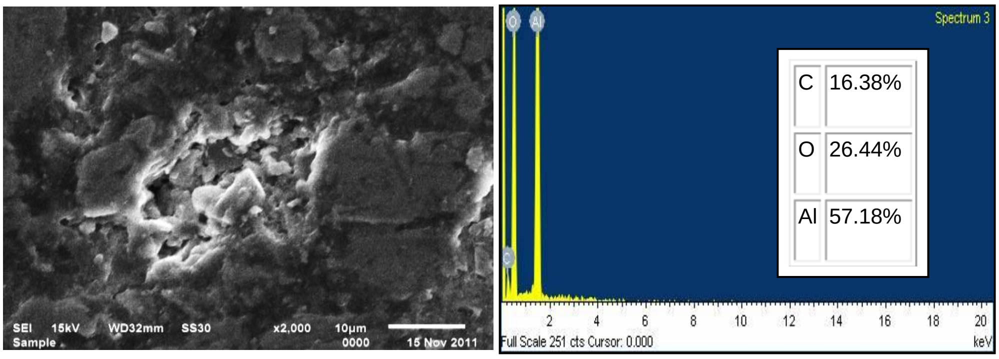
The microstructure of Al-25%Al_2O_3 composite formed [Fig. 6 (c)] showed a fully molten layer of alumina particles dispersed in the Al matrix. Agglomeration of the particles in one region is clearly visible in this case. This may be due to presence of air and moisture entrapped during the development of the composite formed by HVOF thermal spray process resulting in the superficial voids and pores. Higher amounts of O (26.44%) were detected along with Al (57.18%) and C (16.38%) from the EDS analysis.



(a)



(b)



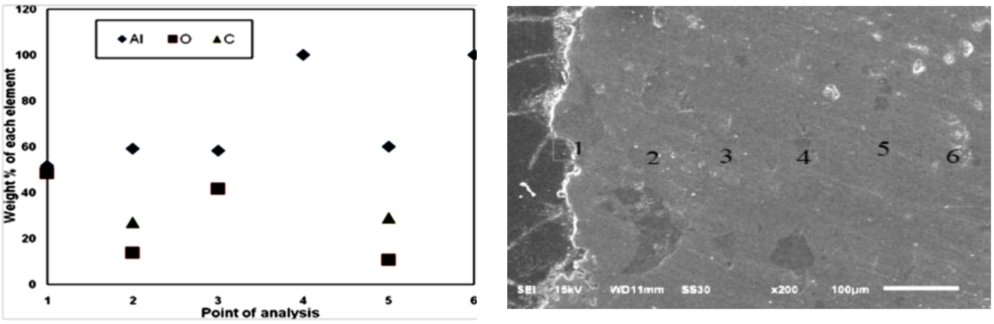
(c)

Figure 6- SEM/EDS morphology of HVOF spray formed bulk Al matrix composites (a) Al-15%Al_2O_3 (b) Al-20%Al_2O_3 (c) Al-25%Al_2O_3

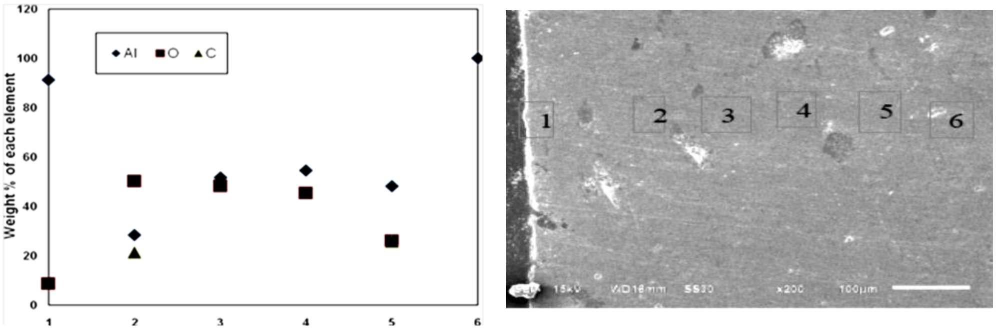
3.4 Cross-Sectional Analysis

The HVOF sprayed composite coating was deposited on the stationary substrate by moving the gun and the required thickness of the coating was obtained by several passes of the gun. The deposited composite material was then separated from the die steel Cross-sectional morphologies along with variation of elemental composition across the cross-section of the bulk composites are depicted in Fig. 7. The Al-15%Al_2O_3 bulk material has shown a denser morphology without the presence of voids and pores. The prepared material is free from cracks [Fig. 7 (a)]. The developed composite mainly consists of Al and O throughout the cross-section, which indicates the embedment of alumina particles in the Al matrix. Some amounts of C are also visible at points 2 and 5 from the EDS cross-sectional analysis.

The Al-20%Al_2O_3 composite is found to have similar morphology with well dispersed alumina particles along the cross-section. The white Al_2O_3 particles with spherical morphology are clearly visible in [Fig. 7 (b)]. The EDS cross-sectional analysis indicates the decrease in the amount of oxygen from point 2 to point 6. The carbon is revealed at points 2 and 5 only. [Spencer et al, (2009)] also observed uniform dispersion of both Al and Al_2O_3 particles in the coatings developed by them. During the experimentation, the authors suggested that under the spray conditions used the two species Al and Al_2O_3 did not separate in the gas flow stream. The cross-sectional EDS morphology of Al-25%Al_2O_3 bulk composite [Fig. 7 (c)] showed intact morphology with the presence of mainly aluminium and oxygen along with some amounts of carbon throughout the cross-section. Higher amounts of oxygen can be seen along the cross-section.



(a)



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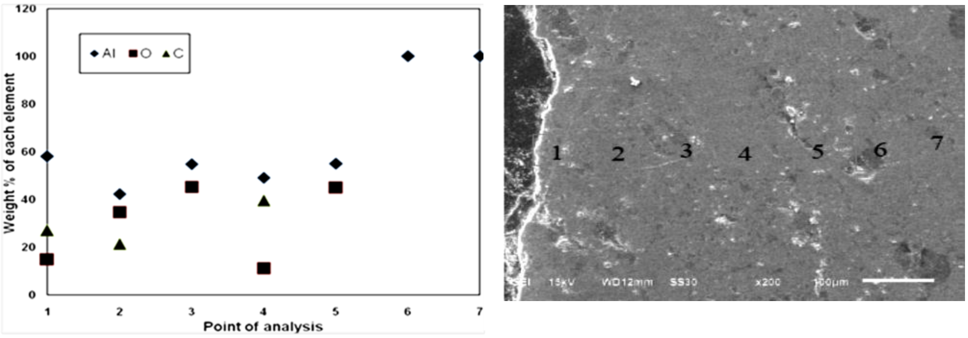

(c)

Figure 7- The EDS Cross-sectional morphology of HVOF spray formed bulk Al matrix composites (a) Al-15%Al_2O_3 (b) Al-20%Al_2O_3 (c) Al-25%Al_2O_3

3.5 EDS Elemental Mapping

Composition image (SE) and EDS elemental mappings of the HVOF spray formed bulk composites are shown through Fig. 8 to Fig. 10. The elemental mappings for all composites developed showed that the basic elements of the powder viz. Al and Al_2O_3 have indicated uniform distribution within the region. Composition image (SE) and X-ray mappings of the Al-15%Al_2O_3 bulk material is shown in Fig. 8. Alumnium has shown its maximum presence in the composite. O is also found co-existing with Alumnium. C is seen distributed in the rarified manner. A typical layered structure is revealed for Al-20%Al_2O_3 composite as is clear from Fig. 9. Al is found to present mainly in the composite. O is present along with Al in rarified manner, C is also detected in the composite material. A similar analysis of the Al-25%Al_2O_3 bulk composite is shown in Fig. 10. Alumnium and Oxygen coexist in the material. At the places where Al is absent, some amount of C is found to be present.

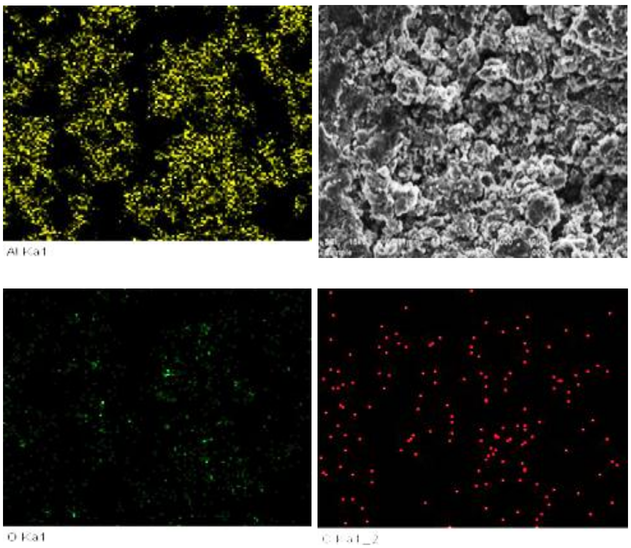

Figure 8 - Composition image (SE) and EDS elemental mappings of the surface of HVOF spray formed Al-15%Al_2O_3 composite material.

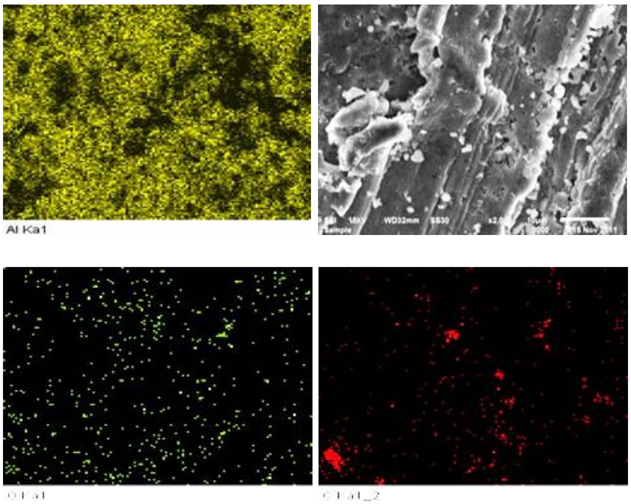

Figure 9 - Composition image (SE) and EDS elemental mappings of the surface of HVOF spray formed Al-20%Al_2O_3 composite material

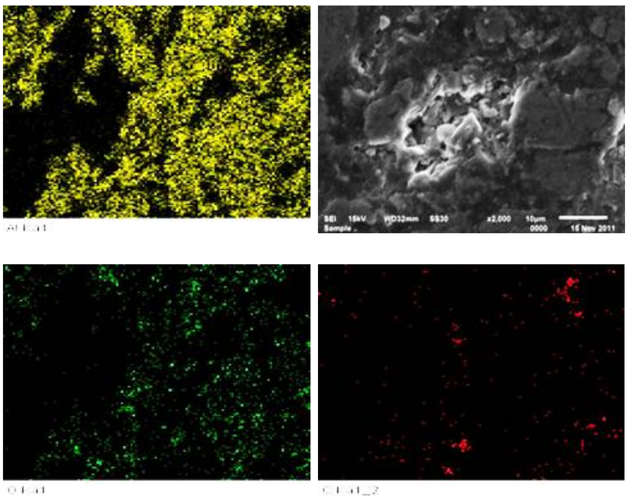

Figure 10 - Composition image (SE) and EDS elemental mappings of the surface of HVOF spray formed Al-25%Al_2O_3 composite material.

3.6 Porosity and Microhardness.

Porosity measurements were made by the image analysis software Envision 3.0 and the porosity values are found to be in a range of 1.58-1.66% for all the reinforcements. Microhardness values were found on the Vickers microhardness tester. The microhardness values of the matrix phase of Al-15% Al_2O_3 bulk material was in the range of 43.9 - 60 Hv with an average value of 51.95 Hv. The microhardness values for the material Al-20% Al_2O_3 lies in the range of 42.1 - 63 Hv, with an average value of 52.5 Hv. For the composite Al-25% Al_2O_3 hardness values were found out to be ranging from 39.9-43.2 Hv with an average value of 41.5 HV. The study showed that on increasing the amount of reinforcement material from 20 to 25%, the microhardness decreases. Among the three compositions, the composite material with Al-20 wt% Al_2O_3 gave the best results. To date, there has been lack of studies regarding bulk Al-Al_2O_3 composites developed by HVOF thermal spray technology.

Conclusions

1. The study showed the possibility of developing AMCs with different proportions of Al_2O_3 as reinforcement material by HVOF spray process.
2. Homogeneous dispersion of the matrix phase (Al) and the dispersed phase (Al_2O_3) was observed in the developed composites
3. The composite materials were totally free from any cracks
4. Thickness of 4±2mm was successfully achieved by HVOF thermal spray technique.
5. Average hardness values of Al-15% Al_2O_3, Al-20%Al_2O_3 and Al-25%Al_2O_3 composites were found out to be 51.95 Hv, 52.5 Hv and 41.5 Hv respectively. The study showed that on increasing the amount of reinforcement material from 20 to 25%, the microhardness decreases.

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