



Investigation on coated substrate of D-Gun sprayed Monolayer and Functionally Graded Ceramic Composite Coatings of Alumina and Alumina-Titania (AT-13)

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Abstract

The ceramic composites coated by thermal spraying process and Detonation-gun (D-gun) is one of the most important and less explored area on the high velocity thermal coating process. The coated ceramic materials are Alumina and Alumina-Titania (AT-13) as monolayer and functionally graded coatings (FGC's) by D-gun. The important aspect of the present study is to find the residual stress on the coated substrate and the hardness of the coatings and its chemistry changes by XRD analysis. XRD analysis shows the phase change in the monolayer are less compare to functionally graded coatings. The monolayer of Alumina has higher hardness compared to all other coatings and functionally graded coating ATG2 gives higher compressive residual stress.

Keywords: Hardness, Residual Stress, XRD, FGC's, Detonation-gun

1 Introduction

Ceramic are well known for its higher hardness and wear resistance, but the drawback is it has lesser toughness. To get over this problem it had been developed as composite material, still there is only a marginal improvement had been shown by [3]. Further to improve the toughness properties of mono-ceramics, it had been coated as a thin layer on a toughness material like mild-steel as a substrate. Thermal spraying is one of the versatile processes to coat ceramic materials. The present trend looks for more than one property on the surface of the component apart from their product design, such requirements are difficult to obtain some times, from monolithic, multilayer, or composite systems. This has prompted the development of a new category of material known as functionally graded material (FGM) that possess various functions simultaneously [1], facilitating the distinct multifunctional characteristics needed. FGMs are usually developed in the form of bulk materials, interfacial layers or coatings (Functionally graded coating –FGC). The paper discussion is mainly about the residual stress on the substrate surface, which is not been investigated in the due course of the researchers and it seen to be one of the very important factor of ceramic coatings by thermal spraying processes.

2 Coating materials

Alumina represents the most commonly utilized ceramic material in industry, because of its extremely superior higher hardness, abrasion, high temperature, chemical resistance and it also electrically insulating as well. Purity levels are available from 85% through 99.9%. The fused Alumina-Titania (AT-13) is grayish in color [2], resulting in coating i.e., dense and hard and resisting wear due to abrasion, fretting, cavitations and particle erosion. FGC is developed through the D-gun spraying process; D-gun is one of the high-velocity spraying processes, which gives lesser porosity with higher hardness to the coating. Using various combinations of alumina and alumina-titania; the detailed specifications are listed in Table. 1. It is preferable to use the tougher grade as a top layer to avoid any tendency to surface initiated cracking. Fig. 1 & 2 shows the SEM photograph of Alumina and Alumina-Titania powders.

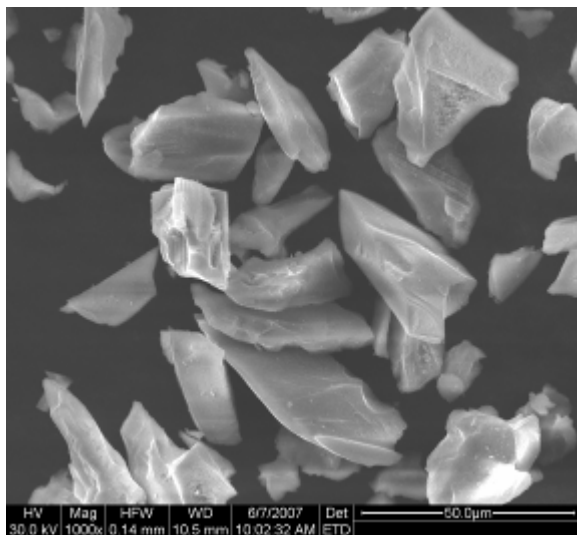


Fig. 1. SEM photograph of Alumina powder

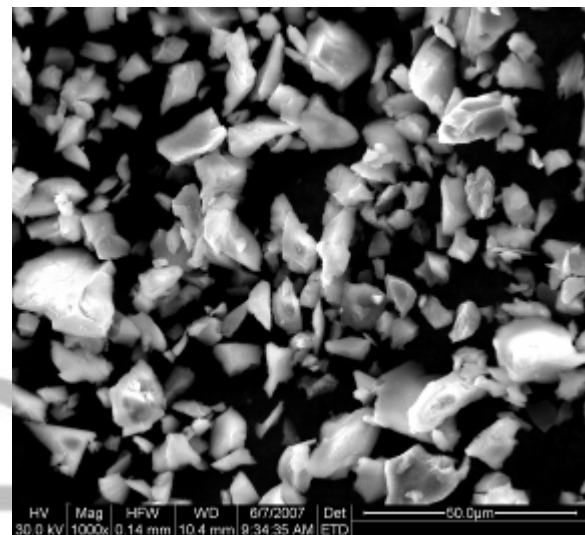


Fig. 2. SEM photograph of AT-13 powder

Table 1 Specification of Coatings

| Sl. No | Designation | Coating Details |
|--------|-------------|--|
| 1 | ALM | Alumina (Al_2O_3) monolayer 500 μ m |
| 2 | ATM | Alumina- Titania (AT-13) monolayer 500 μ m |
| 3 | ATG1 | Alumina- Titania (AT-13) 100 μ m over Alumina (Al_2O_3) 400 μ m |
| 4 | ATG2 | Alumina- Titania (AT-13) 200 μ m over Alumina (Al_2O_3) 300 μ m |
| 5 | ATG3 | Alumina- Titania (AT-13)) 250 μ m over Alumina (Al_2O_3) 250 μ m |

3 Experimental Details

3.1 Grid blasting

The grid blasting process was carried out to the specimen before coating. The air compressor feed air at 3.5atm pressure, through a drier and a pressure regulator to a suction-feed grid blasting gun mounted inside a 1m³ cabinet. The plane carbon steel ASI-1018 is held in a vice at a distance of 0.15m by the alumina brown grid of mesh size 24 (840 μ m) for a process duration of 15 seconds.

3.2 Detonation Gun

The D gun coating system, shown in Fig. 3, consists of 25.4mm inner diameter, 2.25 m long stainless steel detonation tube positioned at one end, near to a substrate holder and at the other end has a detonation venting section. The substrates in the form of 50 x 50 mm square piece with 5mm thickness mild steel samples were held in vice. The ceramic powder was

propelled along the detonation tube placed at the specified distance i.e., stand off distance from the substrate location (5). The ceramic powder particles were entrained into the convection flow behind the passing detonation wave. The detonation wave was initiated by igniting the acetylene-oxygen i.e., fuel oxygen gas mixture at the closed end of the tube by an electric spark. The entrained particles experienced the thermodynamic conditions behind the wave for a certain residence time, by the carrier flow gas and splashes on the substrate at a predetermined detonation frequency. The advantage that a detonation wave provides is the uniformity of the thermodynamic and flow conditions created behind the wave that the particles are exposed to, which provides a unique advantage over other processes [4]. The gun operating parameters are listed in the Table.2.

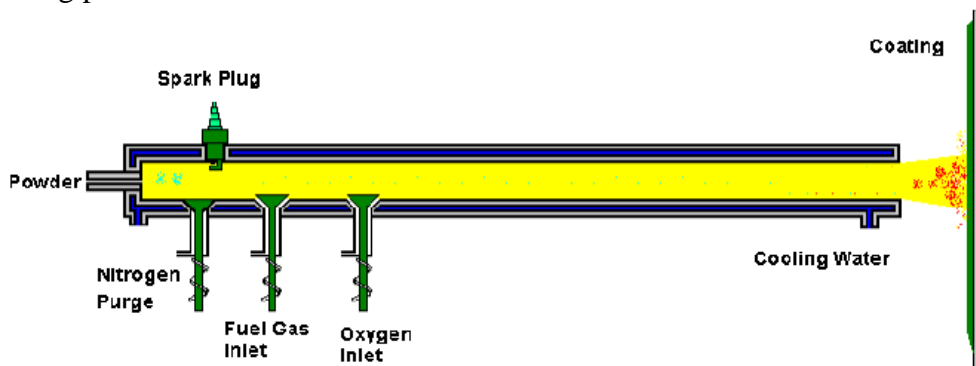


Fig.3. Schematic of a Detonation gun

Table 2. Operating parameters of Detonation – gun

| Sl No | Ceramic powders (Size 5 – 25 μm) | Fuel content (C_2H_2) Splh | Oxygen content (O_2) Splh | Carrier gas flow (N_2) Splh | Stand off distance (mm) | Detonation Frequency Shots/s |
|-------|--|--|--------------------------------------|--|-------------------------|------------------------------|
| 1 | Alumina (Al_2O_3) | 1920 | 4800 | 900 | 200 | 3 |
| 2 | Alumina Titania (AT-13) | 1920 | 4500 | 800 | 180 | 3 |

3.3 Coating Analysers

The coated monolayer and FGC's are tested for its hardness, XRD and residual stresses on the coated surface. Hardness is been test by the standarad microhardness testing machine MATSUZAWA MMT-7 with 300gms load for 5sec duration at different points of the specimen. The material phases are identified by the standard Shimadzu XD-D1,XRD phase analyser with copper radiation ($\lambda=1.5405 \text{ \AA}$) and it scan at 0.050/ sec. The residual stresses of the plane carbon steel specimens were analysed by the standarad two exposure x-ray diffractometry technic by Rigaku Strain tester, with chromium $\text{K}\alpha$ radiation ($\lambda=2.2909 \text{ \AA}$) was used to detect angular changes, induced by residual strains, for the 211 ferrite diffraction line at ψ value from 0° to 30° in steps of 5° and the angle of 2θ from 140° to 170° . Fig. 4 illustrates the specimens surface after different processes are mentioned as follows: machined surface (MS), grid blasted surface (GBS), coated surface (CS) and surface after pealed-off coating (SAC), the surfaces roughness (R_a) are measured and the details are mensioned in Table.3.

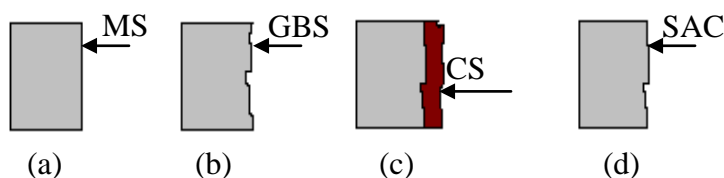


Fig.4 Specimens surface after different processes

Table 3 Surface Roughness

| Sl | Coatings | M S | G B S | S A C | C S |
|----|----------|---------|--------|--------|--------|
| No | Ra (μm) | Ra (μm) | Ra(μm) | Ra(μm) | Ra(μm) |
| 1 | ALM | 0.30 | 5.74 | 5.33 | 6.19 |
| 2 | ATM | 0.26 | 5.49 | 4.64 | 5.95 |
| 3 | ATG1 | 0.25 | 5.55 | 4.93 | 5.87 |
| 4 | ATG2 | 0.27 | 5.82 | 3.63 | 5.60 |
| 5 | ATG3 | 0.25 | 5.78 | 4.63 | 5.90 |

4 Results and discussions

4.1 Hardness

Fig. 5 shows the hardness of the coatings. ALM has higher hardness compare to other coatings and ATM has a lower value and indicated a lowest trend line. The remaining coatings hardness' are in between the ALM and ATM coatings. Among all ATG1 trend seems to be almost a straight till 300μm depth of coating from the substrate and then it falls as the top layer is alumina-titania (AT-13). ATG3 has a marginal increase from 100μm to 200μm then it falls drastically. ATG2 trend show a intermediate trend between ATG1 and ATG3. Comparing the different coatings hardness, it seems that ATG1 is very near to the monolayer ALM coating. As the alumina coating is very brittle compare to the AT-13 ceramic composite coating, the composite on the top surface improves the toughness of the coatings [5]. So, it is preferable to use the tougher grade as a top layer to avoid any tendency to surface initiated cracking and to improve its flexural strength.

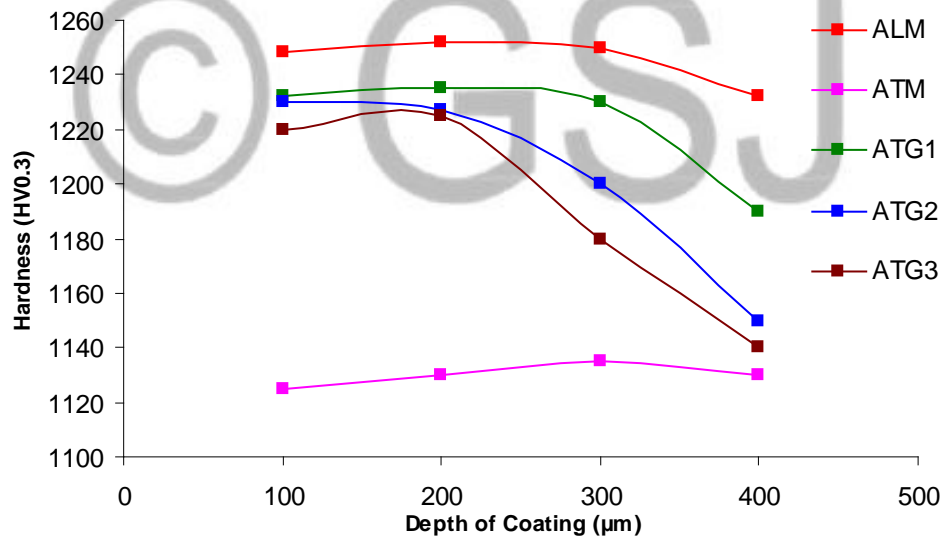


Fig.5 Hardness versus depth of the coatings

4.2 XRD Analysis

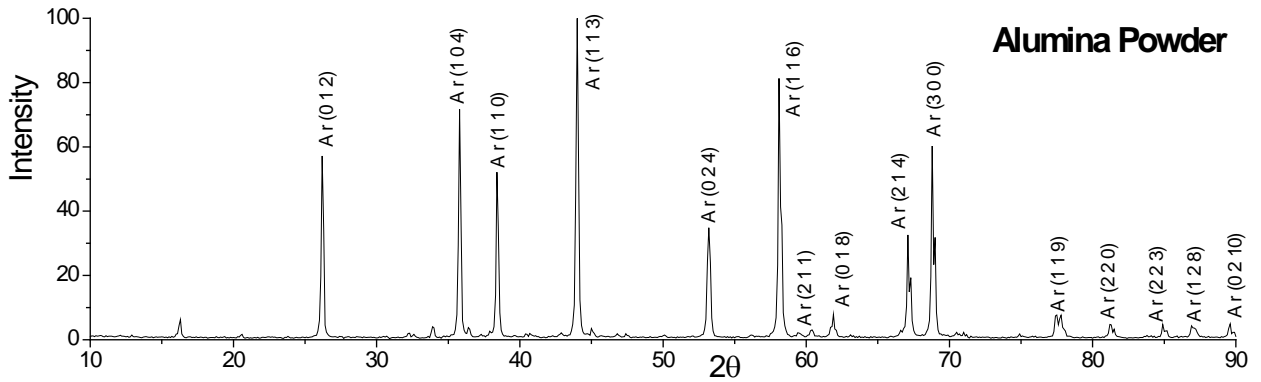


Fig.6 XRD profile of Alumina powder

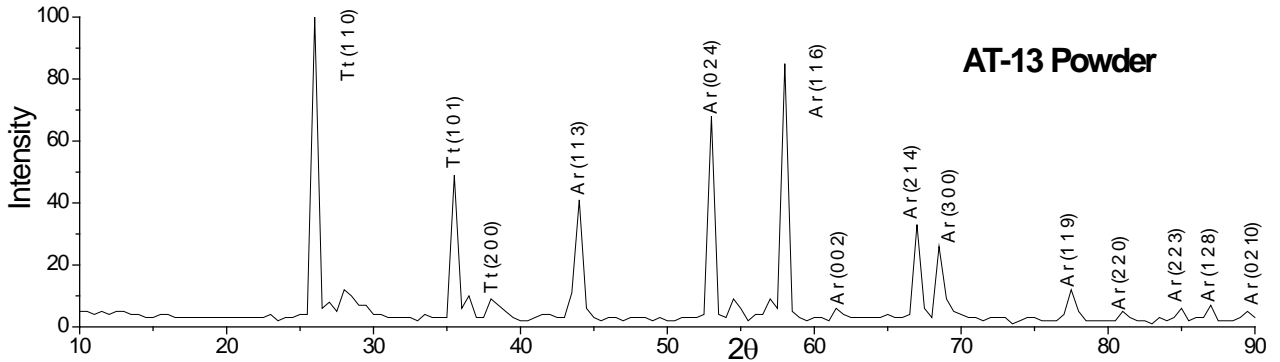


Fig.7 XRD profile of Alumina-Titania (AT-13) powder

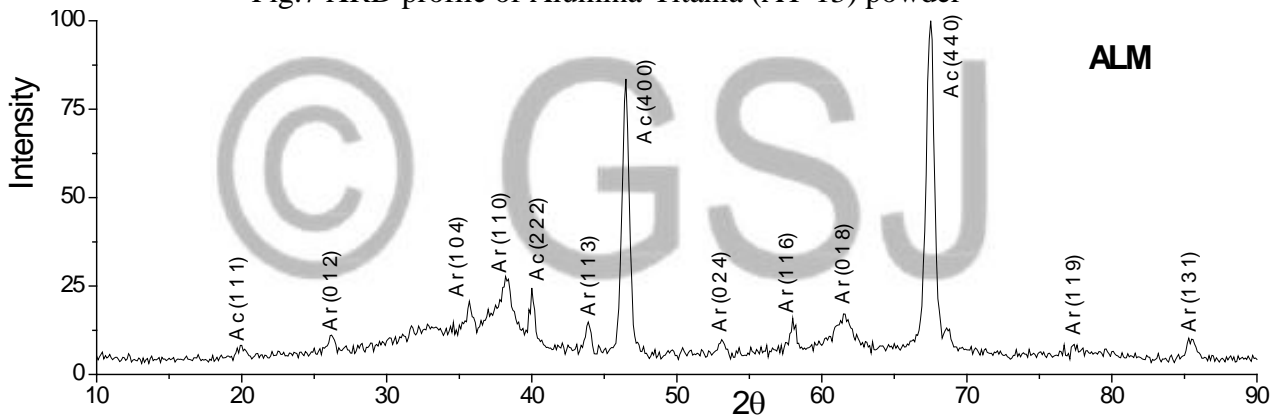


Fig.8 XRD profile of Alumina coating

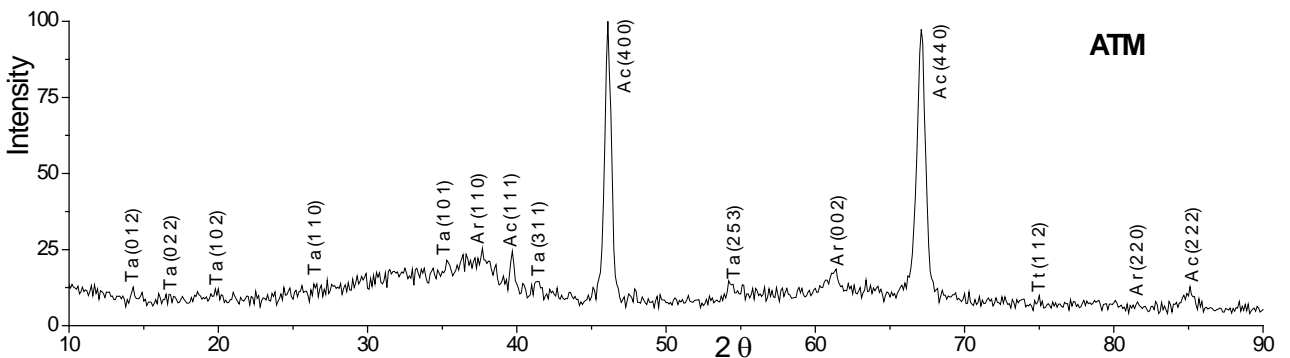


Fig.9 XRD profile of Alumina-Titania coating

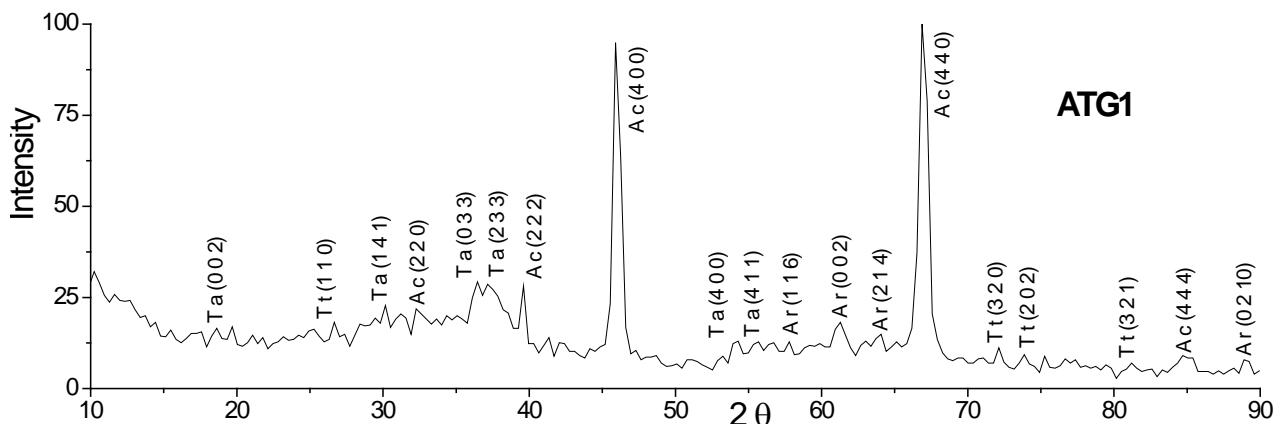


Fig.10 XRD profile of ATG1 coating

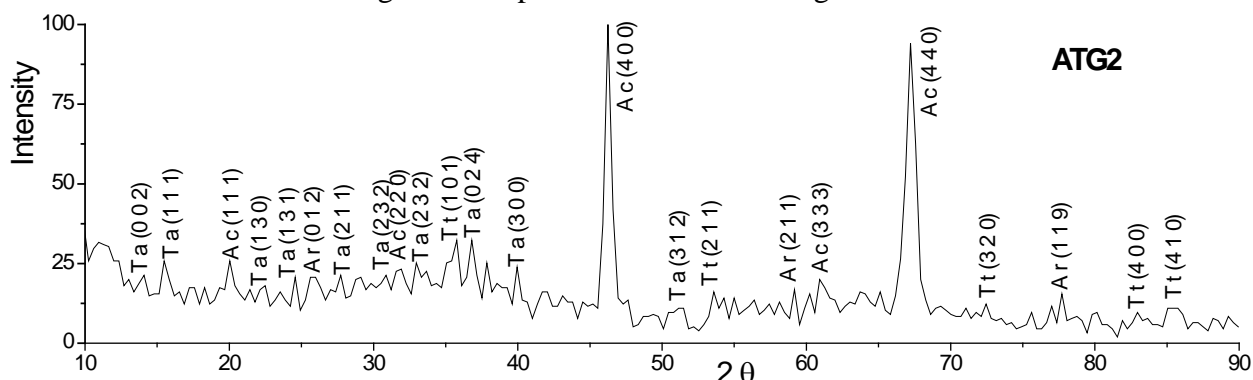


Fig.11 XRD profile of ATG2 coating

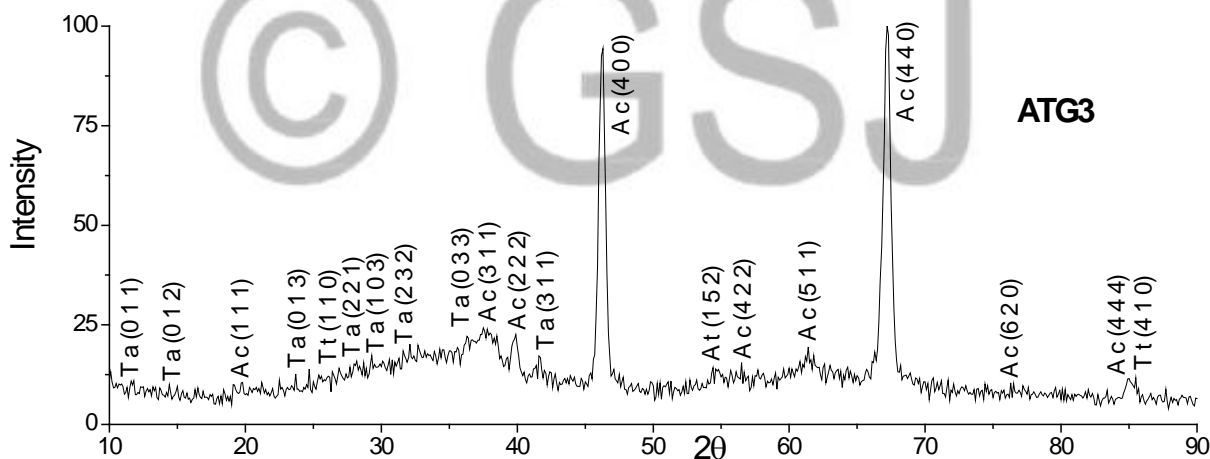


Fig.12 XRD profile of ATG3 coating

Note: A-Alumina, T-Titania, a- anorthic, r-Rhombohedral, c- Cubic, t- Tetragonal

Fig. 6&7 shows the XRD profile of Alumina and Alumina-Titania powders and Fig.6-10 indicates the different profiles of ALM, ATM, ATG1, ATG2 and ATG3 respectively. In Fig.6 it is observed that, while the alumina powder contains predominantly rhombohedral (corundum-synthetic) alumina phase and in Fig.7 the AT-13 powder contains rhombohedral alumina and Tetragonal titania phases. Fig.8 shows ALM XRD profile, the rhombohedral alumina phase changes to cubic alumina. Fig.9 indicates ATM XRD profile, the spray deposits consist cubic alumina and some part of Tetragonal titania changed to anorthic titania phase. The phase transformation is induced by the high temperature during spraying. Owing to the high rate of cooling ($10^6 - 10^8$ K/s) leading to rapid solidification in the spray deposition, high temperature metastable cubic alumina and anorthic titania did not have enough time to transform into stable rhombohedral alumina and tetragonal titania. However, the material has undergone lattice straining. Which has a great cause of residual stresses on the specimen surface, the detail are discussed in the following residual stress analysis part.

Fig.10 indicates the XRD profile of ATG1. ATG1 has little identifications of anorthic and tetragonal phases, this is due to very thin coating of AT-13 and the thickness to rapid cooling ratio is very less. Fig.11 indicates ATG2 XRD profile and the phases are comparatively significant than ATG1 and the above statement, i.e., the rapid cooling ratio is significant. The same trend is shown in ATG3 XRD profile indicated in Fig.12.

All the above coatings show a similar trend in the XRD profile i.e., Alumina in cubic structure phases (4 0 0) and (4 4 0) are predominant in the profile. In ALM coating rhombohedral alumina phases has lesser intensity and behaves like a matrix with cubic alumina. The remaining coatings, has traces of titania in Tetragonal and Anorthic phases with lesser intensity. Which leads to a conclusion of matrix structure formation, i.e., the 13% of titania is melted completely to form a matrix and the alumina particles are presented like a whiskers reinforcement in a matrix.

4.2 Macrographic observation

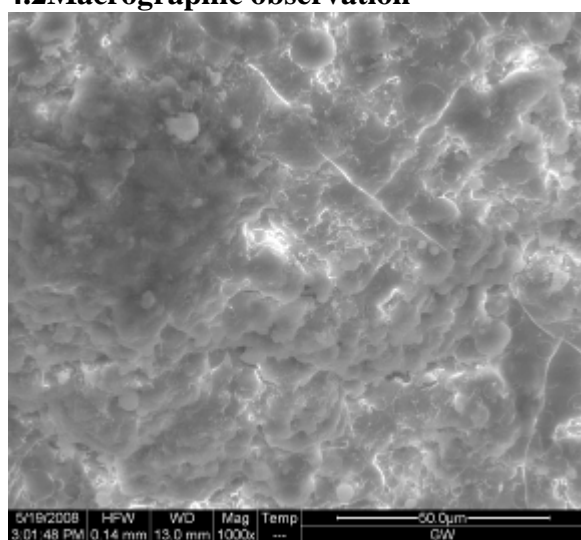


Figure 13 SEM photograph of ALM

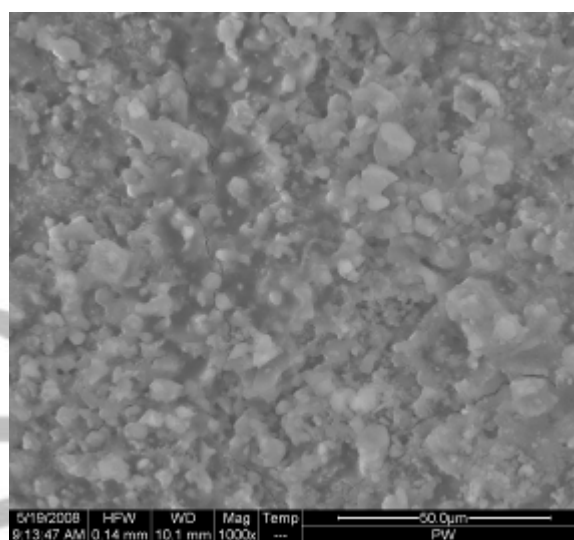


Figure 14 SEM photograph of ATM

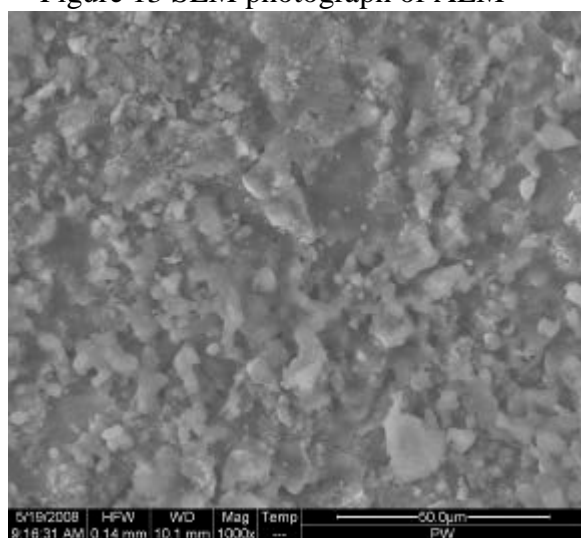


Figure 15 SEM photograph of ATG1

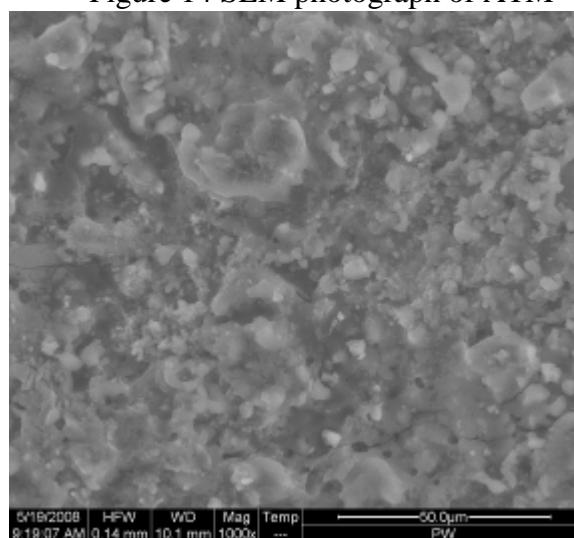


Figure 16 SEM photograph of ATG2

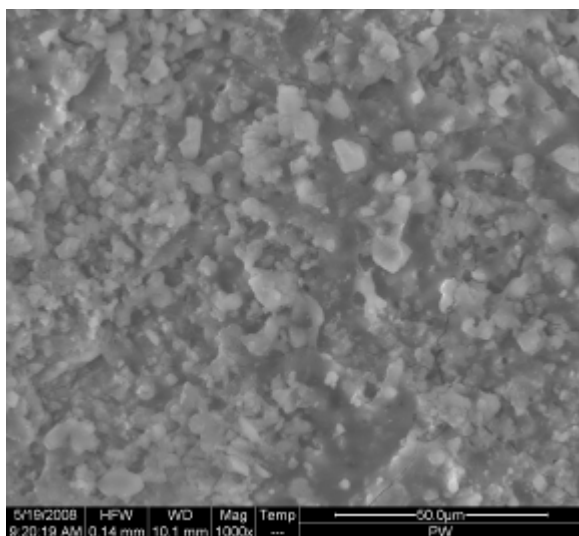


Figure 17 SEM photograph of ATG3

Fig. 13-17 shows the SEM photograph of ALM, ATM, ATG1, ATG2 and ATG3 respectively. Fig.13 shows ALM SEM photograph shows small cracks on the surface, this may be due to higher hardness of the alumina powder and higher operating temperature and velocity with larger thickness (500µm). There are also some solids and spread locals surrounded the surface, which looks like a sprayed matrix structure. Fig. 14 illustrates ATM SEM photograph and it also shows cracks but comparatively lesser than ALM coating, this also due to the larger thickness (500µm) and the spray matrix are very clear as the 13% of titania melts and surrounded the cubic alumina. ATG1, ATG2 and ATG3 coatings SEM photographs follows the same trend as ATM, but the cracks are not present on the surface this may be due to the lesser coating thickness as specified in Table1..

4.3 Residual Stress analysis

4.3.1 Longitudinal Residual Stress

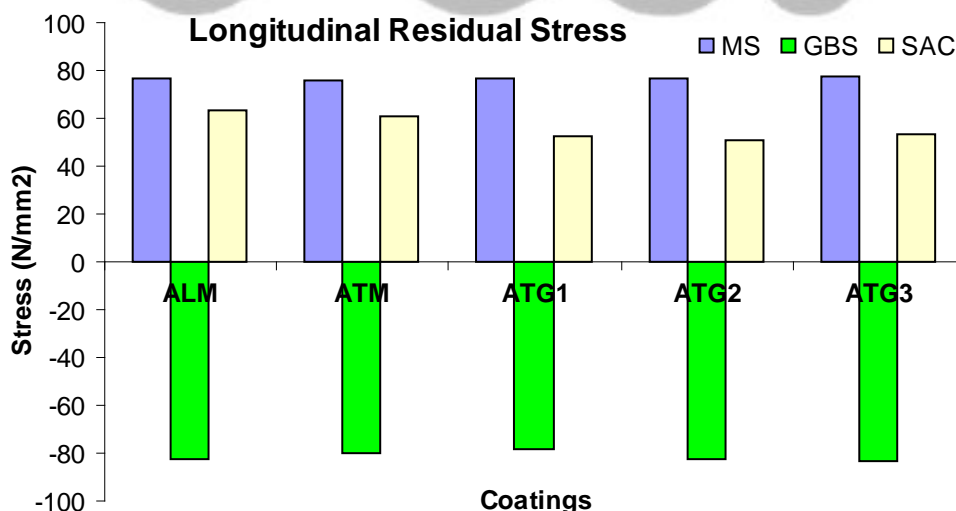


Fig. 18 Longitudinal residual stresses at surfaces after different processes of ALM, ATM, ATG1, ATG2 and ATG3 coatings

Fig. 18 illustrates the longitudinal residual stress at surfaces after different processes of the coatings. The surface roughness (Ra) values are listed in the Table 2. The MS surface has a lesser Ra value and the GBS has a higher Ra value and SAC has a lesser Ra value compared to the GBS, this is due to the higher process temperature and velocity of D-gun. The D-gun coating process induces velocity (1300m/s) and temperature (3420°K) at the time of deposition, which smoothes the SAC. The MS has tensile stress and after grid blasted

operation the residual compressive stress is induced and after the coating process, the SAC surface turned once again to the tensile residual stress, but the tensile residual stress after machining is comparatively larger than SAC for all the coatings. This is also due to the above mentioned D-gun coating process temperature and velocity. Among all the coatings the residual tensile stress on SAC is lesser in ATG2, which shows a positive aspect in the coating area and also good flexural strength [5].

4.3.2 Transverse Residual Stress

Fig. 19 illustrates the transverse residual stress at surfaces after different processes of the coatings. MS has residual compressive residual stress and after grid blasting the residual compressive stress increases to a larger extent. After coating the tensile residual stress are induced, but the values are comparatively lesser than the longitudinal residual stresses. This may be due to the travel of the gun on the longitudinal direction of the specimen and also due to the dimension of the coated sample width 10mm and length 75mm. The width to length ratio is a considerable value and this may also result in the residual stress.

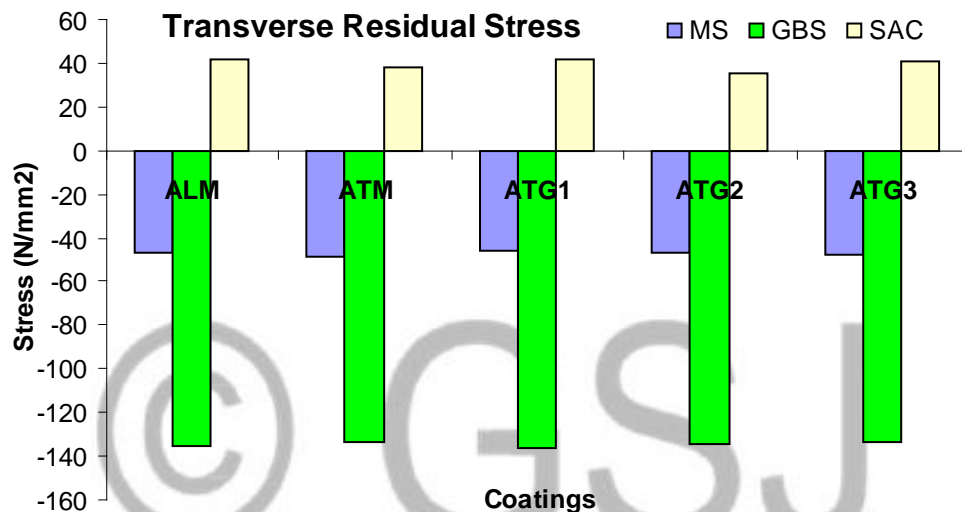


Fig. 19 Transverse residual stress at surfaces after different processes of ALM, ATM, ATG1, ATG2 and ATG3 coatings

5 Conclusion

- This tendency to cracking can be arrested by suitable selection of composite material and proper graded deposition.
- It is preferable to use the tougher grade as a top layer to avoid any tendency to surface initiated cracking.
- Ra is lesser for ATG2 in SAC and CS compare to other coatings, this is due to the higher temperature and velocity in the operation of D-gun coating process.
- ATG1 shows the hardness trend very nearer to the ALM coating.
- The XRD profile shows the lattice strain on the coating due to the rapid cooling by D-gun process.
- The XRD profile of all the coatings shows the same trend in predominant peaks of the cubic Alumina and the remaining are peaks has lesser intensity, which gives a conclusion of formation of spray matrix structure on the coated surface.
- The SEM photographs conforms that the larger thickness leads to small cracks and lesser thickness in the FGCs' arrests the cracks on the surface.
- The SEM photograph also inferences that the spray matrix on the surface by D-gun coatings processes. This may be due to sprayed particles are in semi-solids and partially liquids state, as the powders are range from 5µm to 25µm.

- The longitudinal and transverse residual stress value conforms that the residual stress are changed from compressive to tensile on the coated specimen surface in the D-gun coating process.
- Among all coated substrate ATG2 coated substrate has lesser tensile residual stress in longitudinal and transverse direction.

6 References

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