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Effect of Ceramic Coating on Mechanical Properties of AZ31 Magnesium Alloy

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Abstract: In this work an investigation has been done on four samples of magnesium alloy AZ31 coated with a different percentage mixture of Al_2O_3 and TiO_2 particles by plasma spray method. The effect of the existence of ceramic particles on the surface of AZ31 substrate was studied. A homogeneous distributed microstructure of coated surface was brought to light in microscopy analysis. Densification of the surface was reported with an increasing percentage of TiO_2 content. The nanoindentation study showed that the hardness of coated surface is inversely proportional to TiO_2 percentage. XRD analysis was done, and the results of hardness tests were correlated with SEM and XRD analysis. It was observed that, increase in percentage of TiO_2 beyond 20% led to the formation of Aluminium titanate (Al_2TiO_5) as major phase in coating. Further, decreased densification and increased porosity level were observed due to formation of microcracks with this phase formation. Better densification of coating on AZ31 with less porosity was observed with addition of 20% of TiO_2 particles in Alumina. Increased percentage of TiO_2 beyond 20% decreased the coating hardness by 7%. Fabricated material can be used in places where strength to weight ratio plays an important role. Hence it may be useful in automobile and aircraft parts.

Keywords: AZ31 Mg Alloy; Plasma Spray coating; Hardness; Al_2O_3; TiO_2

1. Introduction

Magnesium (Mg) and its alloys have many performance advantages such as high strength to weight ratio, low density, good thermal conductivity, and good castability[1]. Because of low weight and high strength[2], Mg and its alloys are extensively used in marine/ air / ground transportation industry[3),4)]. Magnesium is a good choice in the areas where strength and weight play an important role. Earlier, the use of Mg in structural materials was limited but in recent years the awareness of environmental protection and fuel saving through reduced CO_2 emission makes this material even more useful in automobile industry[3),5)]. However, major drawback with Mg and its alloys is they have very low resistance towards wear and corrosion[6),7),8)], which makes its use restricted in the areas where the material has to withstand wear and comes in contact with air and moisture[9),10)].

Magnesium is an active metal and casts a layer of MgO at the surface when interacts with the atmosphere. In the existence of moisture, the MgO gets converted into $Mg(OH)_2$ which is stable at high PH[11].The use of magnesium alloy in the industrial fields such as door frames, bearings, steering wheel, gear box, rocket nozzles, turbine blades etc.[12] are unavoidably exposed to wear and corrosion attack in real service time. Lot of studies were carried out to improve the wear and corrosion resistance of magnesium alloy via methods which includes development of composite, structural modification, and surface coating techniques[13].

Surface modification and alloying are found to be effective in improving surface properties. All the properties of substrate metal may be changed by alloying the substrate with other components, but by modifying the surface, only surface properties can be altered by keeping the mechanical properties of the substrate material intact[14),15)].

The characterization of composite materials can easily be altered by varying the type of matrix material, size, shape, and distribution. of reinforcement particles. Mostly Metal Matrix Composites are fabricated at elevated temperatures to create a better bonding between matrix and reinforcement[16]. However, working on elevated temperatures is always not feasible for the matrix such as magnesium. Therefore, coating the surface of magnesium alloy is a better option to enhance its mechanical properties.

Various studies revealed that AZ31 is one of the important structural alloys due to its good mechanical properties and formability at room as well as at elevated temperatures[17]. In a study, a nano composite AZ31-Al_2O_3 -Ca was compared for dry sliding wear behavior with the alloy AZ31 and it was found that wear resistance of composite is much higher as compared to that of the alloy though the wear rate was found to be inversely proportional to the sliding speed for both the nano composite and alloy[18].

Dry sliding wear was studied for AZ31 Mg alloy and Al_2O_3 reinforced MMC and the composite exhibited better wear resistance at high sliding speed and load[19]. In a recent work, a magnesium MMC was developed by reinforcing WC particulates for the application in piston of engines. It was found that MMC increases the piston strength by 30% while reducing the weight of the piston by 35% compared to using base alloy[20]. In another research, an aluminum metal matrix composite was fabricated by reinforcing Al_2O_3, TiO_2 and SiC particles and the results exposed that hardness, tensile strength and yield strength of the samples increases with increasing percentage of SiC particles[21].

In fabrication of metal matrix composite, properties get modified uncontrollably due to inhomogeneous dispersal of metal within the matrix[23].

A group of researchers evaluated the effect of nickel coating on carbon nanotubes growth taking stainless steel as substrate material. And the results obtained showed that nickel coating on the surface of SS316 could increase the CNT yield merely by 8.4%[24].

In another study hardness behavior was studied after the surface modification of AISI 304 in the presence of scallop shell powder and ammonia as nitriding agent by varying the temperature. And the hardness was found maximum at 600^0 C for the duration of 7 hours in presence of 15.35% nitrogen[25].

While modifying the surface, only surface properties get changed. Hence, Surface modification techniques are recommended where wear and corrosion resistance of material is the main concern, as it is most economic and effective way. Applying layer of ceramic particles may provide a solution to the industrial challenges[26]. Among various ceramics, Al_2O_3 and TiO_2 based coatings are recommended as these coatings possess very high hardness and stability to chemical attack which makes them fit for corrosion and wear resistant applications[27]. Addition of rare earth oxides in the coating material have found to enhance the tribological and mechanical properties. In a study, the researchers compared mechanical and tribological properties of Al_2O_3 coated Mg alloy with Al_2O_3 doped with CeO_2 coating and they found out that doping of CeO_2 enhanced the hardness, wear resistance and mechanical properties of the substrate[28]. In a similar kind of study Al_2O_3- 13wt % TiO_2 and Al_2O_3- 13wt % TiO_2-20 wt% Y_2O_3 coating were deposited on a 304 stainless steel substrate by plasma spraying process and their properties were compared and the sample with 20 wt% Y_2O_3 showed better densification, improved fracture toughness, lower friction co-efficient hence better wear resistance with reduced hardness as compared to the sample coated with Al_2O_3- 13wt % TiO_2[29]. In another research a mixture of Al_2O_3 and TiO_2 were coated on steel discs in an alcoholic suspension by suspension plasma spray process in varying weight percentage. The result showed that the wear rate of samples decreases with an increasing percentage of TiO_2[30].

In a comparative study[31] of Al_2O_3 and Al_2O_3- Al sprayed composite coating, it was found that the splashing extent of the coating particles got decreased by addition of Al on Al_2O_3, which means the coating became denser. Also, the presence of Al phase and Al_2O_3 ceramic phase improved the strength and toughness of the coating. Wear resistance of the composite coating was also found to be superior.

Research was done on a combination of Aluminium and TiO_2 flame sprayed composite coating on carbon steel. The tests were performed on five samples, out of which samples with coating composition Al with 10% TiO_2 showed better tribological properties than other coated samples and bare Carbon steel[32].

To reduce the solid particle erosion, a composite multilayer carbide coating was applied on AISI 304 steel by high velocity oxy fuel process and the tests conducted revealed that the hardness and wear resistance of the substrate enhanced due to the high hardness and less porosity of the coated surface[33].

Another recent study investigates abradable environmental barrier coatings (EBCs) in gas turbines. Ytterbium disilicate EBCs with varying porosity levels were examined. Increasing porosity, achieved by adding more pore-forming phase, reduced erosion resistance but enhanced cutting performance by a turbine blade[34].

Resin materials suffer from poor surface characteristics. Research explores supersonic plasma-sprayed AT13/Al_2O_3-phenolic resin coatings on resin matrix.AT13 prepared by agglomeration granulation evenly dispersed TiO_2 phase enhances tribological properties, while AT13 shows better erosion wear resistance[35].

From the available research work, it has been concluded that very limited work on mechanical properties of ceramic coated Mg alloy by varying weight percentage of ceramic particle has been yet carried out, therefore the objective of this work is to obtain various coatings in varying weight percentage of Al_2O_3 and TiO_2 and to compare their mechanical properties.

Magnesium alloys are one of the futuristic materials for various applications such as automobile, aerospace, biomedical, etc. High strength to weight ratio entices their applicability in various automotive and aircraft components by replacing the high-density materials such as steels, cast iron and even Al alloys. The objective of this work is to develop a new coating on magnesium alloy which can be used in various automobile and aircraft parts and hence reducing the weight of the part and whole assembly.

2. Experimental Work

Material selection and methodology of experimentation procedure is discussed in upcoming sections.

2.1 Materials

AZ31 Mg alloy was chosen as substrate material to sediment coating on the surface and was procured in the form of a sheet of thickness 6 mm. Four samples of dimension 100 mm X100 mm X 6 mm were prepared by using wire EDM for coating purpose. Powders of TiO_2 and Al_2O_3 with average particle size -53+15 µm and -45+22 µm respectively were procured for coating purpose. AZ31 magnesium alloy was procured from Nufit Piping Solution Pvt. Ltd. And ceramic powders are procured from Standard Chemicals.

Composition of procured AZ31 alloy is shown in Table 1:

Table 1. Composition of AZ31

Element	Weight %age
Al	3.069
Zn	1.133
Fe	0.019
Mn	0.486
Cu	0.001
Si	0.131
Mg	95.278

2.2 Coating Formulation and Sample Preparation

The composition of three coated samples (S1, S2, and S3) and one uncoated sample of AZ31 (S4) are shown in Table 2.

Table 2. Samples with Coating Compositions

Sample	Composition
S1	100% Al_2O_3
S2	80% Al_2O_3-20% TiO_2
S3	60% Al_2O_3-40% TiO_2
S4	Uncoated AZ31 alloy

A high energy ball mill was used to mix the powders of Al2O3 and TiO2 weighed in required composition. Powders were milled for 3 hours at 300 RPM to achieve an uniform and homogenous mixture for coating. Ball to powder ratio used was 10:1 for milling process.

Samples were cut by wire EDM for coating AZ31 by plasma spray process. This process involves a plasma source which converts the coating material into high velocity molten particles. The schematic of plasma spraying Process is shown in Fig.1.

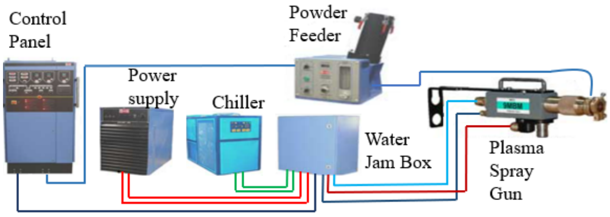


Fig. 1: Schematic of Plasma Spray Process

Samples prepared for coating were cleaned with acetone and polished with 100 to 1000 grit size emery papers to make the surface rough to improve the cohesion between substrate and coating. After that the samples were coated with Al_2O_3 and TiO_2 in different compositions by plasma spray method at Metalizing equipment company, Jodhpur. The thickness of coating applied is 250 µm on all the three samples. Plasma spray coating process using MF4 torch as the plasma source was used in coating process with the parameters shown in table-3. The thickness of coating was selected based on the available literature.

Table 3. Parameters for coating

Power	21-23 KW
Electrode	W/ Cu
Argan Flow Rate	38 lit/min
H_2 Flow rate	8 lit/min
Arc Current	575A
Arc Voltage	69 V
Poder feed disc speed	6 rpm
Spraying Distance	75 mm

2.3 Surface Characterization

The presence of coated ceramic particles and pores were studied using (FESEM) FEI QUANTA 200 F. SEM system equipped with EDS and elemental analysis mapping were used to study various coating on submicron size particle and dispersion of ceramic particles.

The crystalline structure of the coatings was studied by XRD Rigaku MiniFlex 600 in the continuous scanning mode from 0° to 90° and rate 4° /min.

The microhardness was determined using the Vickers Hardness tester (SICMVHT-02). Vickers indentation method (load 300g, dwell time 10s) was used to determine micro hardness. The indenter in this case was square base diamond pyramid.

3. Results and Discussion

XRD test for phase characterization, porosity test, SEM micrograph and micro hardness tests were performed and results of these are discussed in upcoming sections in detail.

3.1 Phase Characterization

XRD spectra of the samples is shown in Fig. 2. It was observed that Alumina and TiO_2 coated AZ31 Mg alloy shows high-intensity peak of Alumina and Aluminium Titanate with low intensity peak of TiO_2. With increase in percentage of TiO_2, Aluminium titanate (Al_2TiO_5) phase formation can be observed to a greater extent. This phase in the coating led to microcracks formation and increased the porosity in the coating. Which directly affects interfacial strength of the coating surface. Formation of such oxides in coating introduce porosity and does not contribute to enhance any desired property which is confirmed by porosity results as obtained. Also, it is observed that the formation of magnesium oxide phase in coating is because of substrate internal oxidation which adversely affects the interfacial bond between the coating and substrate and hence reducing the coating strength[34].

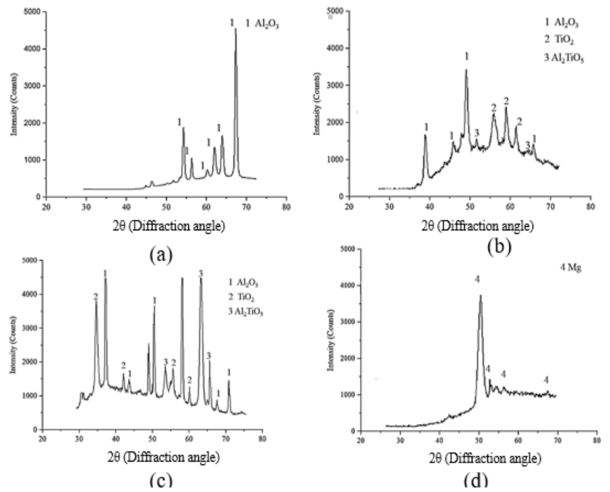


Fig. 2: XRD spectra of Samples (a) S1 (b) S2 (c) S3 and (d) S4

3.2 Porosity and Microstructure

Porosity of the samples were observed by area percentage porosity method on 24 mm coated length. The values of porosity of different samples are shown in Fig. 3.

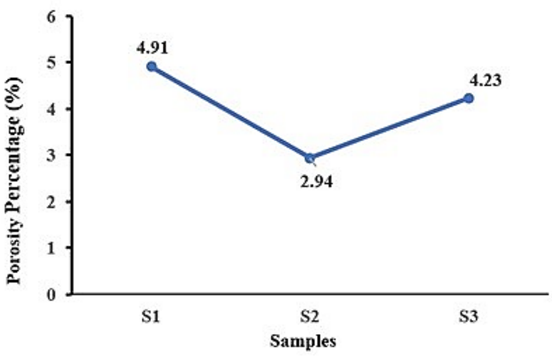


Fig. 3: Porosity Plot of Coated Samples

The coating exhibit lamellar structure with some porosity. Plasma sprayed coating was applied by the effect of stream of molten ceramic on to the surface and hence the nature of the deposited coating is channelized by different factors like temperature, velocity, particle size and their distribution, distance between the torch and substrate etc. Due to thermal stresses developed during plasma spraying, curling of splats occurs which leave some gaps partially filled, resulting in formation of pores. SEM images of coated surface were taken to identify the presence of pores and particles of coating material. It was found that presence of TiO_2 particles up to a certain limit makes the surface denser and on further adding TiO_2 particles the surface became porous due to crack formation same as depicted in the porosity test performed.

SEM images of cross section of coated samples S1, S2 and S3 are shown in Fig. 4.

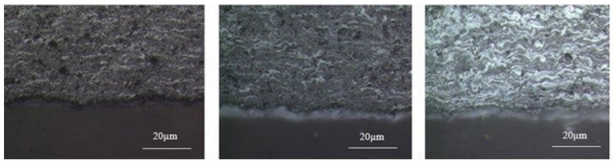


Fig. 4: SEM images of cross-section of Samples S1, S2 and S3

SEM micrograph of S1 is shown in Fig. 5. To study the morphology of coated surfaces, SEM Images were taken on 200X, 500X and 1000X and 5000X. Through microstructural analysis of the samples, it was observed that the sample S2 is denser with less porosity than the other samples.

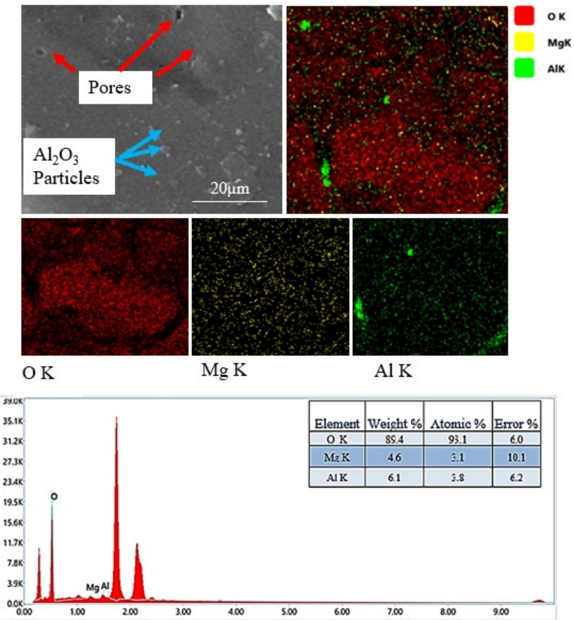
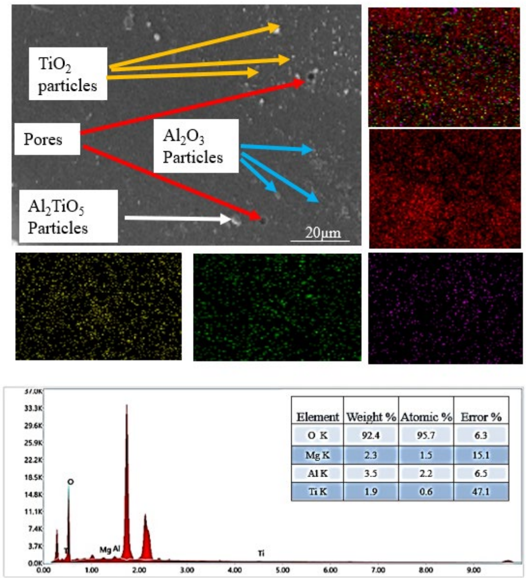


Fig 5: SEM Micrograph EDX and line scannig of Sample-1

SEM micrograph of the coated surfaces show some non-melted particles present on the surface, which may be destructive for tribological applications. SEM images of plasma sprayed ceramic coated surfaces show that the surface of the coating is uneven, and many pores were present. These pores are the results of factors like volume shrinkage[35)] of molten ceramic particles, overlapping of particles, deposition of gas in the gaps created between the molten particles and especially sudden and rapid cooling of sprayed particles. These deposited particles on rapid cooling creates pores between the adjacent layers.

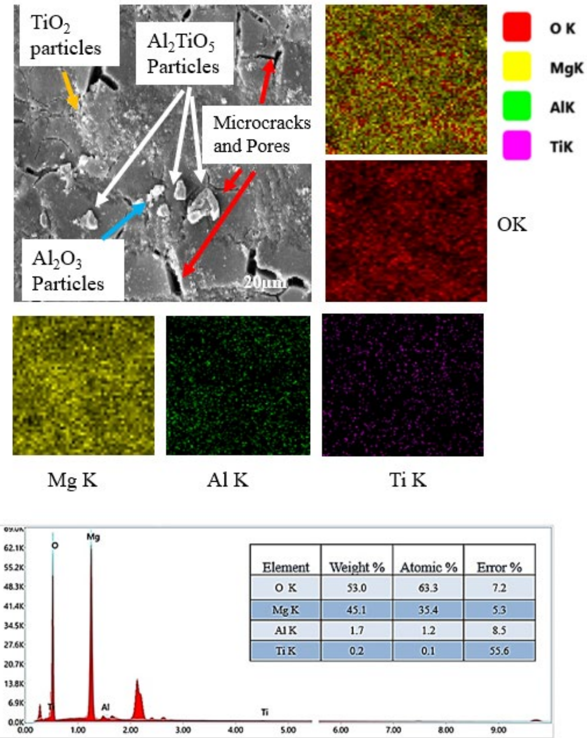
SEM micrograph of S2 is shown in Fig.6.



Element	Weight %	Atomic %	Error %
O K	92.4	95.7	6.3
Mg K	2.3	1.5	15.1
Al K	3.5	2.2	6.5
Ti K	1.9	0.6	47.1

Fig 6: SEM Micrograph,EDX and line scannig of Sample-2

Better densification of coating was observed with addition of 20% of TiO_2 particles in Alumina. This effect is ascribed to the lesser melting point of TiO_2 as compared to Al_2O_3. TiO_2 behaves as a binder for Al_2O_3 due to its lesser melting point (1843^{0}C) as compared to Al_2O_3 (2054^0 C). Most of the micro pores get sealed due to fusion of molten TiO_2 particles. Crystalline and amorphous TiO_2 particles tried to build up a cloud in and around the voids and made the voids semi sealed. SEM micrograph of S3 is shown in Fig.7.



Element	Weight %	Atomic %	Error %
O K	53.0	63.3	7.2
Mg K	45.1	35.4	5.3
Al K	1.7	1.2	8.5
Ti K	0.2	0.1	55.6

Fig 7: SEM Micrograph, EDX and line scanning of sample-3

Micrograph of sample S2 and S3 show the presence of Al_2O_3, TiO_2 and Al_2TiO_5 phases with some voids and microcracks. Uneven distribution of various phases led to the formation of voids on the surface. With the increase in percentage of TiO_2 from 20 to 40, Al_2TiO_5 phase is identified as major phase with Alumina. Aluminium titanate phase in the coating led to the formation of microcracks and increased the porosity level in the coating. Grain growth of aluminium titanate and less dense microstructure decreased the mechanical strength (microhardness) of coating. The voids on the surface are mainly due to the volume shrinkage of the molten particles, accumulation of gas in molten particles and overlapping of sprayed particles. Rapid cooling and solidifying of sprayed particles also led to the formation of voids in coating.

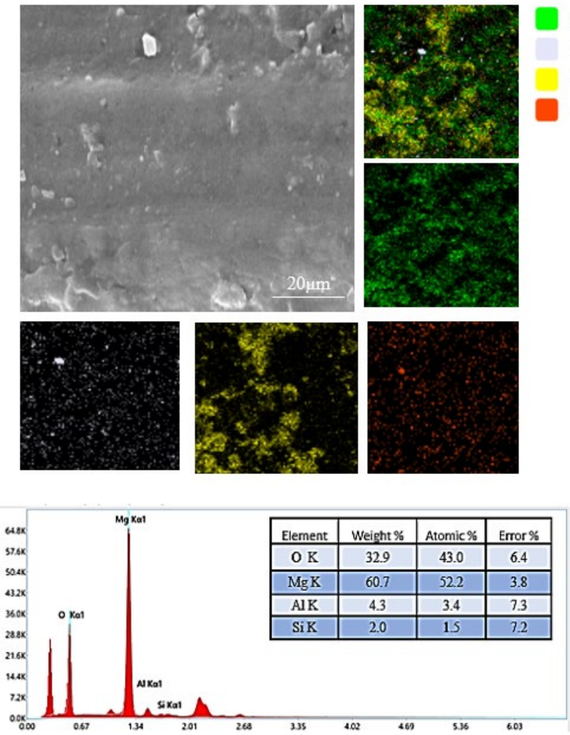


Fig. 8: SEM Micrograph,EDX and line scannig of sample-4

Figure 8 presents the SEM and EDX appearance of uncoated sample-4. The surfaces of all the coated samples appears to be rough as compared to the uncoated samples. Reason may be the quenching stress developed during the plasma spray process. Which results because of the super quick cooling of the sprayed particles on the surface.

3.3 Microhardness

Micro Vickers hardness values of uncoated and coated samples were studied under the load of 300 gram for 10 seconds duration. An average hardness value of five equidistant indentation points was taken for reporting micro hardness of each sample. Samples prepared for the hardness test are shown in Fig. 9.

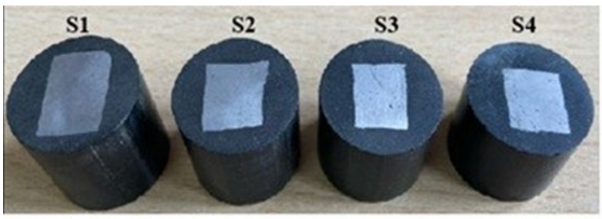


Fig.9 Prepared samples for Micro Hardness test.

The values of hardness of each sample are shown in Fig. 10.

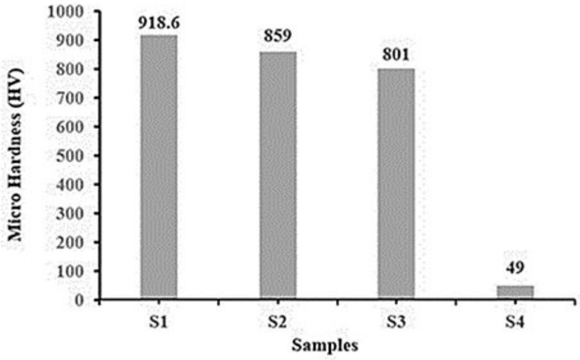


Fig. 10: Micro Hardness of Samples

It was seen that the coated samples (S1,S2 and S3) are much more harder than the non-coated sample (S4). Hardness of samples increases with increasing percentage of Al_2O_3 particles. There is a nominal difference in the hardness of S1, S2 and S3, coated with Al_2O_3 and TiO_2 on the other hand, almost 94% difference between the coated and uncoated samples. Higher microhardness of the sample implies large number of dislocations of interparticle boundaries resulting in the denseness of coating with addition of TiO_2 particles. The results of the hardness test are in sync with the results of phase characterization.

Indentation images of coated samples are shown in Fig. 11.

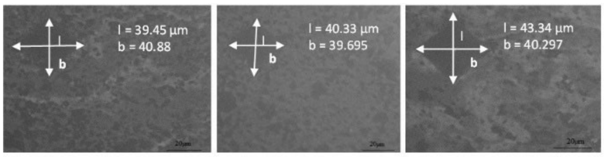


Fig.11: Indentation Images of Coated Sample

4. Conclusions

A layer of 250µm thickness of Al_2O_3 and TiO_2 in varying weight percentage was deposited on Magnesium alloy AZ31 by plasma spray technique and their mechanical properties of coated samples were compared. Following conclusions can be drawn from the work.

1. XRD analysis and microstructural study revealed the formation of aluminium titanate (Al_2TiO_5) as major phase with increase in percentage TiO_2 with Alumina. Aluminium titanate phase in the coating led to the formation of microcracks and increased the porosity level in the coating. Better densification of coating was observed with addition of 20% of TiO_2 particles in Alumina with less porosity as compared to the other three samples.
2. Firstly, the porosity of the samples decreases with TiO_2 addition but beyond 20% by weight percentage of TiO_2 particles the porosity increases gradually. This effect is ascribed to the lesser melting point of TiO_2 as compared to Al_2O_3. TiO_2 behaves as a binder for Al_2O_3.

Most of the micro pores get fixed due to the introduction of molten TiO_2 particles. Crystalline and amorphous build up a cloud in and around the gaps and made them semi sealed. And on further increasing the percentage of TiO_2 particles, a new phase Al_2TiO_5 was observed which causes more cracks and pores on the surface hence increasing porosity.

3. Micro hardness test showed a significant improvement by coating the bare substrate with Al_2O_3 and TiO_2 ceramic. It can be concluded that hardness of the sample decreases with increasing percentage of TiO_2. Aluminum Titanate grain growth and a less dense microstructure reduced the microhardness of the coating.
4. Hardness was found to be enhanced by almost 94% by coating the surface. Higher microhardness of the sample implies large number of dislocations of interparticle boundaries resulting in the denseness of coating with addition of TiO_2 particles.

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