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A REVIEW ON SURFACE MODIFICATION OF AZ91 MAGNESIUM ALLOY FOR IMPROVEMENT IN CORROSION AND WEAR RESISTANCE

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ABSTRACT

Magnesium alloys have very properties like low density, its high strength to weight ratio , high dimensional stability, good electromagnetic shielding characteristics, high damping characteristics, good machinability and is easily recycled makes it an ideal for automotive, aerospace , portable electronic and communication devices, sporting goods, structural materials, handheld tools, household equipment, and biodegradable implants.However the use of magnesium alloys is limited by their poor wear behaviour and low corrosion resistance for many industrial applications. This has limited its use in the automotive and aerospace industries, where exposure to harsh service conditions is unavoidable. The simplest way to avoidcorrosion and increasing wear resistance is to do surface modification of Magnesium alloys .This review paper give detailing in coating and surface modification technologies, applied to AZ91alloy for improved corrosion and wear resistance. The different techniques by which generally AZ91 surface modified include electrochemical plating, conversion coatings, anodizing, gas-phase deposition processes, thermo- mechanical treatments, laser surface alloying / cladding and organic coatings.This research covered surface modification by electroless Ni plating, thermal spray plasma deposition processes, Friction stir process for improving corrosion and wear resistance.

Keywords: *AZ91 Alloy, Surface Modification, Coatings, Corrosion Resistance, Wear Resistance,*

I. INTRODUCTION

Magnesium(Mg) is the new age light weight metal. Mg and its alloys are used as lightweight structural material in automobile, aerospace, and aviation industries. It has good strength to weight ratio[1]. It has density 1.8 gm/cc, which is 2/3 of Aluminium; 1/4 of steel and 1/5 that of copper and nickel alloys[2]. Mg and its alloys have good stiffness; high castability; machinability; 100% recyclability and other numerous advantages. Although, these good properties, hitherto Mg and its alloys does not find its wide use as an independent structural material due to its poor corrosion and wear resistance. Mg and its alloys come at comparatively the most anodic end of galvanic series. Hence, it forms a galvanic coupling with any metal/alloy and sacrifices itself[3].It has high affinity towards oxygen, hydroxide, etc. It also produces the protective film like Al and Cr,but the film produced by oxides/hydroxides is very thin and poor. This film peels off with minor load and causes pitting corrosion. [4]

In order to improve corrosion and wear resistance, the addition of only alloying elements or change in design is not ample remedy. The high corrosion resistance alloys of Mgare AZ31, AZ61 AZ91 and AM50 have same challenges. To overcome these challenges and provide a good lifespan myriad research had been done. One of the most effective ways is to insulate the surface layer by providing various types of coating. [3]In the past, many researchers have reported many coating techniques, which criticized by many researchers. One of the best critical papers was by J.E. Gray, B. Luan. In their critical review, they included almost all developed surface coating techniques for Mg and its alloys until 2002. We are working on surface modification of AZ91 alloy. Hence, in this critical review, we have reviewed practically possible and important coating methods of AZ91 like, Electroless Ni coating, Friction stir processingon AZ91 by pure Al as well as ceramic coating by thermal Atmospheric plasma spray process.

II. ELECTROLESS Ni COATING ON AZ91:

Electroplating, a subtype of electrochemical plating is one of the widely used coatings. But, it possesses some limitations, like it produces a less uniform coating especially in recess and holes. The uneven distribution of current density is the main factor. It can be resolved by the electroless plating techniques. Hence, it is mostly used for high reactive Mg and its alloys as a substitute of electroplating. The Electroless coating does not use electrical property, hence produces a more uniform layer. Many materials like Ni, Cu, and Ni-Cr etc. find its suitability with AZ91 in this process[3].

The desire in technology is to develop an easy process which produces a proper surface for electroless Ni plating. This direct electro/electroless plating is one of the old techniques. To plate Mg substrate, interaction between electrolyte and base metal should like,

1. The dissolution of the Mg alloy must not occur in the electrolyte.
2. The naturally formed hydroxide and oxide films must be removed in the electrolyte.

Ni is most important among all becausenot give good corrosion resistance, but also gives good wear resistance as well as conductivity with maintaining the metallic property. [3,5] The most important benefit of Electroless Nickel (EN) coatings among the few elements which finds adherent ability with Mg is that provide a hard surface finish, good anti-corrosion performance, good solder ability with maintaining metallic properties . The research interest of EN coating on the AZ91 alloy help to promote the application of AZ91 alloys in industry. Due to the distinct behaviour of AZ91 in the coating solution, careful pre-treatment methods can be conducted to get the good quality coating [5]. Applying EN coating on AZ91 Mg alloy components can enhance the wear and corrosion performance of the components. [5]In general, it was studied since 1970s on steels, Cu, Al and later on Mg alloys. Most of studies were focused on the relationships among the plating bath compositions, temperature, additives, pH valueand the deposition mechanisms [6].

As Mg and its alloys are highly reactive they require care full pre-treatments, which do not forms any oxide or hydroxide layer during intermediate steps. The main reason behind elimination of any oxide or hydroxide layer is it perturbs the interaction between base metal and coating layer and results into delamination of layer. The careful pre-treatment steps include cleaning, etching and fluoridisation before EN plating. It ensures clean and protective layer free surface.

Efforts have been done mainly in two aspects for pre-treatment of Mg alloys:

1. To overcome the use of hazardous chemicals in traditional pre-treatment routes

2. To enhance the adherence and uniformity of EN coating [6].

The first pre-treatment process in which electroless nickel is plated directly onto magnesium alloy AZ91 die castings was developed by Sakata et al. The authors state that uniform, adherent coatings were obtainedalthough; this process was failed due to corrosion formed due to porosity. The effective porosity was in Ni strike layer [3].In general the pre-treatment is as follows[5]:

Pretreat→ Degrease→ Alkaline Etch→ Acid Activation→ Alkaline Activation→ Alkaline Electoless Ni Strike→ Acid electroless Ni plating

To overcome these difficulties a simpler process has been developed by PMD (UK) The basic sequence of this pre-treatment is as follows [3]:

Pretreat→ Alkaline clean→ alkaline activation→ Fluorides Activation→ Electroless Ni Plating

In this case the adhesion was strong. The only problem is the short bath life for industrial application due to fluoride contain. The probable remedy is addition of a complexing agent like glycine.H.K. DeLong proposed a process. In this process the use of toxic pre chromium ions avoided and the chemical solution was made up of pyro phosphate, nitrate and sulfate[7]. The process sequence is as follows:

Chemical Etching→ Fluoride Treatment→ Neutralization→ Electroless Ni Plating

RajanAmbat and W. Zhou studied the effect of plating parameters like addition of thiourea, Fluoride, mercaptobenzothiosole (MBT). Also they have studied the effect of ligand to metal ion ratio and the distribution of Ni during plating. The pre-treatment they have used is as follows[8]:

Ultrasonic Degreasing using acetone→ Rinse in 10% NaOH at 60°C for 5 min→ Water Rinse→ 6% Chromic acid+ 5% Nitric acid pickling for 45s→ Water Rinse→ Fluoride Activation in HF(250 mL 70% HF/L) for 10 min→ Water rinse $\xrightarrow{Immediately}$→ Solution Bath(500ml).

Table-1 Optimized Bath Composition and Coating Parameters

Bath constituents and parameters	**Quantity**
Basic nickel carbonate($NiCO_3$ $2Ni(OH)_2 4H_2O$)	9.7 g/l
Citric acid	5.2 g/l
Ammonium bifluoride	7.5g/l
Hydrofluoric acid	11 ml/l
Thiourea (TU)	1 mg/l
Sodium hypophosphite	20 g/l
Ammonium hydroxide	To adjust pH
Temperature	80°C
Agitation	Mild-mechanical

This process does not requires electricity,sufficient temperature (80 ^{0}C) is maintained by using hot plate. The time for plating was varying with the desired coating thickness. After removing from the bath ,specimen were rinsed with water and acetone followed by air drying. Coating rate was decided after measuring weight as coating thickness is directly proportional to plating time. They analyzed the deposit layer on specimen withSEM-EDS. The microstructure got is shown in fig 1. In AZ91 α and β to phases are present .β has the composition of $Mg_{17}Al_{12}$, which provides nucleation site for the Ni plating and spreads it on the α-phase. Final weight of the specimen was determined and the coating rate in mm/h was calculated from the weight gain. The optimum ligand to metal ion ratio was found to be 1:1.5, while the safe domain for thiourea (TU) was in the range of 0.5–1 mg/l. Fluoride was found to be an essential component of the bath to plate AZ91D alloy with an optimum value of 7.5 g/l. Within this range, the phosphorus content in the coating remained the same. Below 0.5 mg/l, the bath decomposes spontaneously; correspondingly a higher amount of phosphorus content can be seen in the coating, possibly due to the formation of nickel phosphide. The presence of 0.25–0.5 mg/l mercapto-benzo-thiosole (MBT) found to accelerate the plating process. Addition of 0.5 mg/l of MBT gave the best deposition with maximum hardness, although the effect was similar in the concentration range of 0.25–0.5 mg/l. The hardness they obtained was 600-700 VHN. This illustrates the strong wear resistance along with corrosion behaviour of the Electroless Ni coating [8].

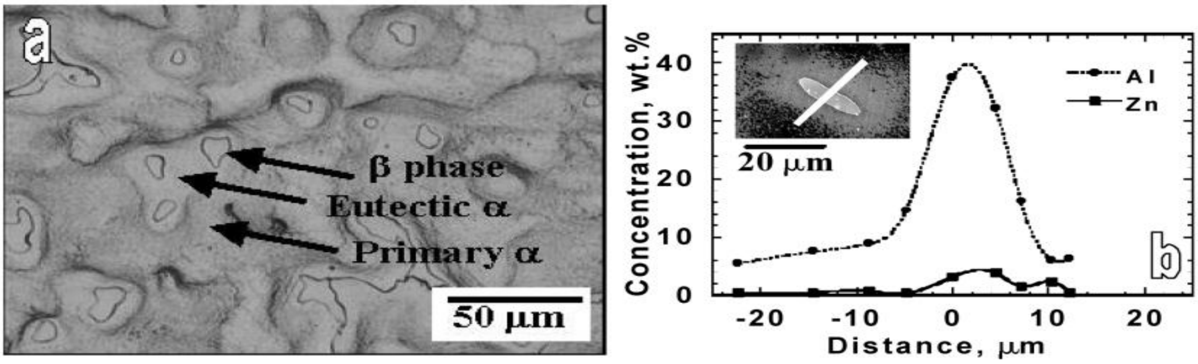

Fig 1 AZ91 substrate: (a) microstructure and (b) Al and Zn concentration across the β-phase along the line shown in the picture [8].

In recent investigation by Mohd. Imran Ansari &Dineshsingh G. Thakur the bath pH was investigated to properly coat the AZ91 Mg alloy. They used NaOH as pH adjuster. Pre cleaning steps were as follows:

Mechanical Cleaning→ Rinse with Distilled water→Degreasing in Acetone→ Rinse with Distilled water→ Rinse in 10% NaOH at 60^{0}C for 5 min→ Rinse with Distilled Water→ 6% Chromic acid-5% Nitric acid pickling for 45s→Rinse with Distilled water→ Fluoride activation (250ml/L HF) for 10 min→ Rinse with distilled water $\xrightarrow{Immediately}$ Electroless Ni Bath.

The bath constituent is shown in Table-2.

Table-2- Bath constituent for Ni-P coating on AZ91 Mg alloy.

Bath constituents	**Bath A**	**Bath B**	**Bath C**
Nickel sulfatehexahydrate (g/l)	18	18	18
Sodium hypophosphite monohydrate (g/ l)	22	22	22
Citric acid monohydrate (g/l)	5	5	5
Sodium acetate (g/l)	13	13	13
Ammonium bifluoride (g/l)	8	8	8

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Hydrofluoric acid, HF (40% v/v) (ml/l)	12	12	12
Operating conditions			
pH (NaOH)	3.5	6.5	10
Temperature (C)	80°C±1	80°C±1	80°C±1

In the case of 6.5 pH, the produced surface was smooth (As shown in fig-3). It had wear resistance 478HV on Rockwell scale. This was much higher than original value of 91HV hardness of pure AZ91. Also the hardness obtained from bath A and C (3.5 and 10 pH respectively) was not much deviated from original value. The wearand friction behaviour is shown in fig-4, which says that bath pH 6.5 is optimum in order to obtain corrosion and wear resistant Electroless Ni coating on AZ91 Mg alloy [9].

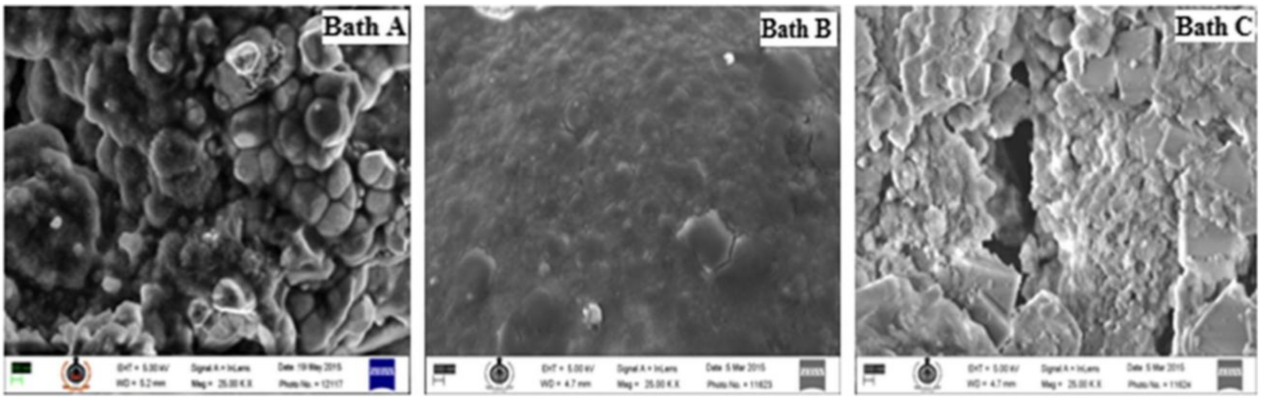

Fig-2-FESEM morphologies of as-deposited condition ENi–P deposit obtained from bath A, bath B, and bath C

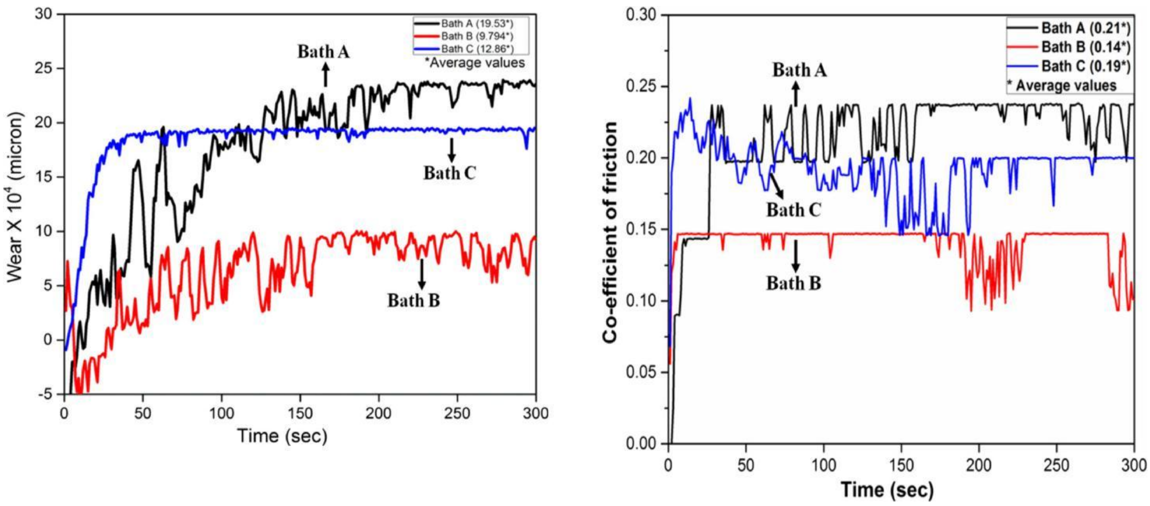

Fig-3-Wear & Co-efficient of friction of as-deposited condition ENi–P deposit obtained from bath A, bath B, and bath C [9].

III. FRICTION STIR PROCESSING

Friction stir processing (FSP) is a solid state process known for its ability to modify microstructures and provide improved properties over conventional processing technologies. Friction Stir processing(FSP) is the new emerging technology to make s/c composites. It started its journey from Al alloy and now extended it to most of all metals and their alloys. It is fact that the light alloys are more readily fabricated using this method. To produce S/c composite base metal either grooved or holed followed by filling of desired filler material.

Filler/ reinforcing is material most probably ceramic .This grooved or holed surface stirred by high velocity of non consumable tool and produces S/c composites. This tool posses a pin supported by shoulder. The new research is mainly based on the tool design; however it affects the metallurgy of the component. The rotating and linear motion of tool gives desired weld pool, i.e. s/c composite. [12, 13]. From the literature it is evident that reinforcement element like Al_2O_3, SiC, Pure Al, C, TiC is possible for Mg and its alloys [14-18].

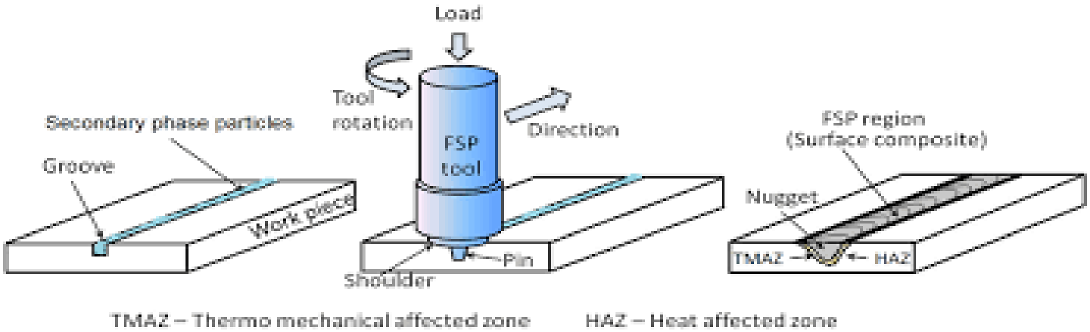

Fig-4 Schematic representations of friction stir processing (FSP) [13]

Jugaraj Singh and et al. have studied effect of TiC reinforcement in AZ91. They successfully made s/c composite using AZ91 plate having dimensioned 5mm×5mm×10mm and 40µ TiC powder. They had developed small holes in a row having dimension: - 2mm dia and 0.5mm depth. The dimensions of toolsand the condition for tool rotation had been used by them are as follows:

Table-2- Dimension of Tools& process parameters for TiC filled AZ91 S/c Composite

PIN Dia.(mm)	**PIN Length (mm)**	**SOULDER Dia.(mm)**	**MATERIAL**	**SHAPE OF TOOL**	**ROTATION SPEED**	**LINEAR SPEED**
4	0.5	15	Mild Steel	Cylindrical(without threads)	900 rpm	40mm/min

The result they derived from microstructure investigation and wear behavior that reinforcing TiC is beneficial. Due to grain refinement both strength and wear resistance increases. [15]

AZ91 alloy has highest amount of Al(10%). The amount of Al more than this level does not fevers the metallurgical properties of Mg. In fact by conventional method Al rich Mg cannot be developed. A study by CHEN Ti-Jun and et al. opens the door of Al rich surface layer of Mg. They developed Al rich surface by Friction stir processing. Process parameters and Tool dimensions are shown in table-3. They first formed a plate having dimension 100mm×40mm×15mm by thixoforming process. This plate was grooved with 4.2 mm depth and 1.25mm width along the long axis. There were 3 grooves each with 6mm distance. It was filled with commercially pure Al having size 10µm size.

Table-3-Tool dimension and Process parameters for Al reinforced AZ91Base S/c Composite

PIN Dia	**PIN Length**	**SOULDER Dia**	**MATERIAL**	**SHAPE OF TOOL**	**ROTATION SPEED**	**LINEAR SPEED**
6 mm	4 mm	18 mm	H13 tool steel	Cylindrical (without threads)	350rpm	45mm/min

This results in a 3.5mm thick stirred zone (As shown in fig-5). They haveanalysed its corrosion property. One was corrosion tested and others were annealed at 415°C for time limit ranging in 1h to 4h.The corrosion test gives the favourable result. The present results show that the average corrosion rate of the alloy with such Al-rich surface in 10% NaCl solution is about 0.429 mg/(cm2 ·h) while that of the thixoformed alloy is about 0.549 mg/(cm2 ·h), i.e., the corrosion resistance of the Al-rich surface is increased by about 22% compared with that of the thixoformed alloy. By comparing the microstructural characteristics of these two alloys, it can be found that the microstructural evolution of the PMC alloy with the stir pass is quicker than that of the TF alloy and the grain refinement of the FSP is more effective for the dendrite PMC alloy. Although for more than 6 pass the microstructure result was same.

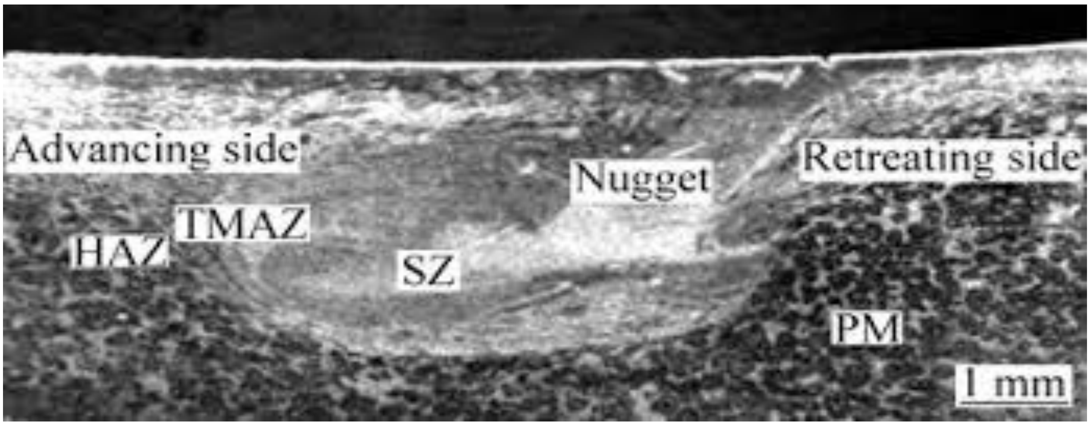

Fig-5-Cross-sectional OM macrograph of TF AZ91D alloy FSPed for four passes [16]

They had shown that the surface composite increases Al solubility in α-phase, which cannot create by conventional techniques. The solubility increased from 3.5% to 8.32 %(mass fraction). Although by producing uniform distribution (post annealing) corrosion resistance increased almost 30% [16].

P. Asadi and et al. contributed in the production of AZ91- SiC surface composite by FSP. They had used 5µ 99.8%pure SiC powder in 5mm thick AZ91 plate, which has groove of 0.8×1.2mm^2. As their investigation was on the process parameters they used following tool parameters:

Table-4- Tool Dimension and process parameters for SiC reinforced AZ91 based S/c composite.

No.	PIN Dia.(mm)	PIN Length(mm)	SOULDER Dia	SHAPE OF TOOL	ROTATION SPEED(rpm)	LINEAR SPEED(mm/min)
1	-	-	15mm		710	12.5
2	5-mm	2.5-mm	15mm	Square	1400	80

Tilt angles were 2.5°, 3°, 3.5° and 4°.They proceed with diff tilt angle atdepth (0.1mm, 0.2mm, and 0.3mm) .But acceptable was only 0.3mm. In the case of tool without pin gives roughened s/c due to insufficient developed temperature. Also at low travel speed the developed temperature was not enough to soften the material. The optimum rotational and traverse speeds were 1,120 rpm and 63 mm/min, respectively. Here, grain size reduced from 150 to 7.17µm. The hardness was improved from 63Hv to 96Hv [17].

In recent investigation by D. Ahmadkhania and et al. on AZ91 Al_2O_3usedfor FSP. In their investigation, plate of AZ91 was grooved. The dimension of groove was 60mm×1mm×2mm. This groove was filled with dried 50nm Al_2O_3 particles having purity 99.8%. First it was FSPed with pin less tool to avoid scattering of powder. Then FSPed with the tool having following dimension:

Table-5- Tool Dimension and Process Parameters for Al_2O_3 Reinforced AZ91 based S/c composite.

PIN Dia. (mm)	**PIN Length (mm)**	**SOULDER Dia.(mm)**	**MATERIAL**	**SHAPE OF TOOL**
4.5	4.5	20mm	Hardened H21 steel	Cylinder (without thread)

No.	**ROTATION SPEED(rpm)**	**No.**	**LINEAR SPEED(mm/min)**
1.	500	**1.**	20
2.	800	**2.**	40
3.	1600	**3.**	80

The result which they got concludes that the traverse speed 40mm/min and rotation speed 800rom is optimum for defect free processing. Also the optimum tilt angle was 3^0 . The hardness was increase from 70Hv to 95Hv. [18]

IV. ATMOSPHERIC PLASMA SPRAY COATING (APS):

Thermal sprayed ceramic coatings have been widely employed to offer alternative for modifying the component surface properties in a broad range of industrial applications, primarily for wear resistance, thermal barrier and corrosive environment [19].Thermal spray technology offers a variety of techniques, which allow the deposition of a wide range of functional coatings designed for specific environments. Recent studies have shown the enormous potential of thermal spray techniques for the surface modification of Mg alloys.

Plasma spraying , one of the good surface modification techniques, has been well-established to deposit various ceramic coatings like Al2O3, TiO2, ZrO2 and Cr2O3 as typical thermal sprayed ceramic coatings, have attracted increasing interests for developed industrial applications.The conventional plasma spray process is commonly referred to as air or atmospheric plasma spray (APS).Atmospheric plasma spraying (APS) is traditionally the most common spray process for the preparation of ceramic coatings because of their high melting points. The plasma spraying method is an optimum process for producing hard coatings because of its lower cost, plasma effect, intense deposition rate, and adaptiveness in almost all work piece dimensions. [20]

A strong electric arc is generated between a positively charged pole (anode) and a negatively charged pole (cathode). To generate the plasma, an inert gas-typically argon – helium or an argon-hydrogen mixture is superheated by a dc arc. This ionizes the flowing process gasses into the plasma state. The powder material is added in this flow and creates the surface.Powder feedstock is introduced via an inert carrier gas and is accelerated toward the work piece by the plasma jet. Plasma temperatures in the powder heating region range from about 6000 to 15,000 °C (11,000 to 27,000 °F), significantly above the melting point of any known material.

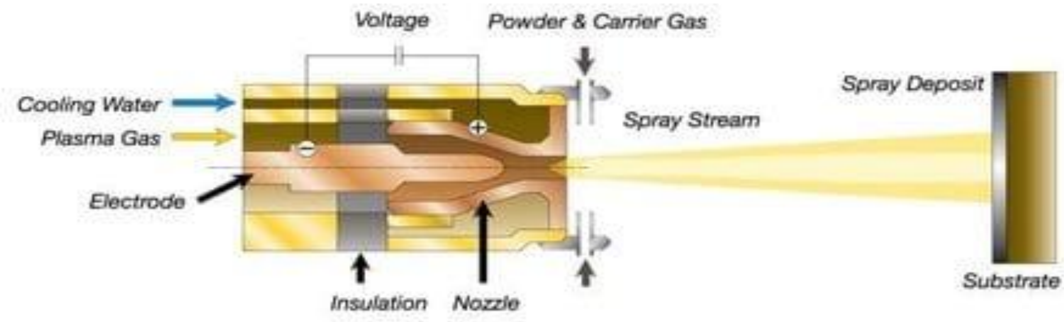

Fig-7-Schematic of APS

In research by Maria Parco and et al.APS process was utilized for Al_2O_3 coating as well as $NiAl_5$ and pure Al coating. The process parameters were as follows:

Table-6-Process parameter for APS of Pure Al, $NiAl_5$, Al_2O_3 on AZ91.

Method	Atmospheric Plasma Spray
Gun	F4 SulzerMetco AG
Atmosphere	Air
Plasma Gas	Argon, Hydrogen
Current [A]	450 ,550, 600
Powder Feed material	Al (99%) −90 + 45 µm, NiAl5 −90 + 45 µm, Al2O3 −45 µm + 5.5 µm

The ability to melt the AZ91 alloyand adherence with surface was in decrease in order as Pure Al, $NiAl_5$ and Al_2O_3. Although, the process was success in order to achieve a coating layer of ceramic powders [21].

In a study by S.W. Lee and et. al. the distance between gun and working substrate and power optimised for wear resistant s/c. As a powder feeder they used Cr_2O_3 having size -45±15µm with irregular shape. The AZ91D plate was grit blasted with alumina grit (80 mesh) in order to achieve proper adhesion. The plate was pre-heated at about 120°C. The plasma flam was used to heat the feed powder and to make it molten for coating purpose. The variable parameters were working distance, i.e., distance between plate and gun and gun power. They used three working distances 5cm, 7cm and 9cm and three variation in arc power 32.5 kW, 35 kW and 37.5 kW at constant flow rate of 70/50 l/min. The final coating thickness was 200µm. According to their results, the optimum working distance was 7cm and the optimum gun power was 35kW. At these parameters they got highest hardness 1080 Hv. Hence, good wear resistance[22].

V. CONCLUSION

Different surface modification techniques described in previous section for improving corrosion and wear resistance of AZ91 alloy. However, no single coating technology has been developed for protection of Mg and its alloy from corrosion in harsh environment. The s/c modification skims which are mentioned in our review paper are complex which incorporate many different technologies. The techniques like Electoless Ni coating, Friction stir process to make s/c composite and atmospheric plasma spray coating must be conducted very carefully in order to achieve optimum results.

For developing a coating for industrial application, Factors like- capital investment, ease of manufacturing coating performance and environmental issue have to be consider.

In S/c modification of AZ91 by electroless Ni coating, the capital investment is small but has serious concern over waste disposal. In this method the use of toxic chemicals such as fluoride and chromide compounds in pre-treatment and plating bath also necessitate further research into the development of "green" plating technology. The challenge associated with Electoless plating on AZ91 Mg alloy is the narrow gape window for operating condition in order to obtain optimum coating.Electoless Ni coating technology has wide potential to produce uniform wear and corrosion resistance coating. Despite, the challenges of different s/c chemistry.

There is several number of coating technologies available for protecting AZ91 Mg alloy. The wide spread use of AZ91 in automotive industries, aerospace industries, electronic, bio implant application is still demanding appropriate coating which can withstand harsh service condition for developing better, simpler, cheaper coating technologies still great research is required. By developing this technologies, we can able to take advantage of the lower weight and good mechanical, physical and chemical properties of this AZ91 alloy.

From these paper it is evident that Mg can be new era light metal. We can overcome its unwanted properties. As a firm step, Electroless Ni is the promising one. It can promisingly improve corrosion and wear properties. The only care is bath constituent and accordingly bath pH. We can easily use this method for mass production.

The Friction stir processing(FSP) is most beneficial for the AZ91 but mass production still requires new technology. It also produces desired surface properties, the only lacking point is the economy for mass production.

In the APS method for ceramic coating ,improvement in hardness , corrosion and wear resistance have observed compare to AZ91 alloy.

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