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Experimental analysis of Tribological behaviour of SS304 using Electroless Ni-Al_2O_3-WS_2 and Ni/WS_2 Coatings

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Abstract—*An attempt has been made in this paper to study the synthesis of Ni-Al_2O_3-WS_2 coating on SS304 substrate with different levels of electroless coating process parameters and optimization of the parameters in order to obtain minimum roughness friction and wear characteristics. Also cladding process has been conducted with Ni/WS_2 mixture on SS304 as the base substrate. The binder used with the mixture is polyvinyl alcohol (PVA). An electroless bath is made consisting of Nickel sulphate solution(base), makeup solution, distilled water or de-ionized water in proportions of 60ml/L for Nifoss 2500 base, 200ml/L for Nifoss 2500 makeup and 740ml/L for de-ionized water. In the case of the first substrate of SS304 in which only nickel coating has been applied the concurrent results where initial hardness was 113 HV but after coating the hardness value became 421.4 HV with the application of load value of 0.5kgf and the dwell time of 10 seconds. Similarly with same base parameters of load and dwell time and coating of Ni-Al_2O_3-WS_2 on the unchamfered side of SS304, the corresponding hardness values before and after electro less coating were 131.4 HV and 481 HV respectively. Whereas similar values for SS304 chamfered side gave hardness values of 356.5 HV and 401.5 HV respectively. Similarly for the cladding procedure on SS304 the Vickers micro hardness tester gave hardness values of 113 HV and 275.6 HV for same load application and dwell time.*

1. INTRODUCTION

Laser cladding is a process to apply metal coatings using a precision laser as a heat source. The laser melts a formulated powder and welds it to a base metal to create a very strong and durable protective coating [1-3]. Laser cladding is the fusion of a different metal to a substrate surface with a minimum of melting of the substrate. The laser cladding process uses lasers as a concentrated heat source to fabricate a coating on a substrate with a perfect metallurgical bond between them [4-5].

Electro less nickel (EN) plating is an autocatalytic process where the substrate develops a potential when it is dipped in electro less solution called bath [6-9] that contains a source of metallic ions, reducing agent, complexing agent, stabilizer and other components. Due to the developed potential both positive and negative ions are attracted towards the substrate surface and release their energy through charge transfer process. The credit for the invention of electro less method goes to Brenner and Riddell. [1]

Electro less nickel plating is an auto catalytic chemical technique and is one of the elegant ways of coating by controlling temperature and pH of bath. The performance and lifetime of engineering components can be enhanced by applying hard coatings over the surface of components. Electroplating and electro less plating deposition are the most economical processes for applying metallic coating of thickness between 10 and 500μm on many engineering components. This is mainly because their rates of deposition can provide the required product quality at relatively low capital and operating costs. Electroplating is a well-established technology but the electro less coating has emerged recently in comparison.

However, the LC becomes more and more popular because of its advantages such as the high material utilization rates, no dust pollution, compact structure and low surface roughness. It has been reported that the coatings prepared by laser cladding have the advantages of dense microstructure, strong metallurgical bonding with the substrates and notable properties compared with the substrates. Laser cladding is very effective and offers distinct advantages. Namely, the surface coatings produced by laser cladding have some obvious advantages such as lower porosity, improved microstructure [10-13] and minimal damage to the underlying substrate, as compared with the coatings produced by plasma spraying. [1-3].

2. LITERATURE REVIEW

Li et al. (2015) [1] in their paper described that multiple-layer laser cladding of 308L stainless steel was obtained by a fiber laser using a way of wire feeding to repair the surface scrapped or erosive parts of 316L stainless steel. The microstructure of the coating was measured by a metallographic microscope, and phase composition was determined by X-ray diffraction. The results show that good metallurgical bonding can be obtained between the 308L stainless steel coating and 316L stainless steel substrate. The coating is mainly composed of columnar dendrites, and there are also a few planar crystals and cellular dendrites distributed in the bonding zone

Gao et al. (2006) [4] in their present study made an attempt was made to improve the wear resistance and the corrosion resistance of AZ91HP magnesium alloy by laser cladding Al–Si eutectic alloy. The results showed that the clad layer mainly consisted of Mg_2Si, Mg_{17} Al_{12} and Mg_2Al_3 phases. The microstructure of the bonding zone changed from columnar grains to equiaxial grains along the direction of heat-flow. The heat-affected zone consisted of α-Mg and α-Mg + β-Mg_{17} Al_{12} eutectic. The formation of multiple Mg intermetallic compounds allowed the clad layer to exhibit higher hardness, better wear resistance and corrosion resistance.

Weng et al. (2015) [3] studied that metal matrix composite (MMC) coatings were fabricated on Ti–6Al–4V titanium alloy by laser cladding. Co_{42} self-fluxing powder, B_4C, SiC and Y_2O_3 were employed as the cladding materials. Microstructures and the wear properties of the different composite coatings were investigated comparatively. Results showed that the laser cladding coatings were mainly reinforced by CoTi, $CoTi_2$, NiTi, TiC, TiB_2, TiB, Cr_7C_3 and Ti_5Si_3.The wear resistance was enhanced by over 10 times compared with the substrate. However, with more SiC addition (14 wt%), a higher micro-hardness was obtained together with poor wear resistance. The wear mechanism of the coatings was discussed by referring to the microstructures and the wear morphologies.

Zheng et al. (2007) [2] Ni/hBN coating was successfully prepared on $1Cr_{18}Ni_9Ti$ stainless steel substrate by means of laser cladding. The micro hardness profile of the composite coating along the depth direction was measured, while its cross-sectional microstructures and phase compositions were analysed by means of scanning electron microscopy and X-ray diffraction. Moreover, the friction and wear behaviour of the composite coatings sliding against Si_3N_4 from ambient to 800 degree Centigrade was evaluated using a ball-on-disc friction and wear tester, and the worn surface morphologies of the composite coatings and counterpart ceramic balls were observed using a scanning electron microscope.

Dumitru et al. (2003) [5] described the friction and wear reduction in applications that allow only a minimal use of liquid lubricants is done with solid lubricant films or with protective coatings, such as diamond-like carbon (DLC). Further improvements are possible if the geometries of the contact surfaces are modified in a controlled way, as we have already demonstrated it for TiN and TiCN. In this work, the possibilities to generate patterned DLC coated low wear tribological surfaces by means of laser processing were investigated. The results showed that the friction coefficient did not increase, as compared with the unstructured and DLC coated surfaces, and that the structure pores trapped the debris particles produced when the DLC film eventually broke.

Meng et al. (2005) [6] researched that in order to improve wear resistance of titanium alloy, a process of laser cladding Ni-Co-Cr-Al-Y coating on Ti-6Al-4V substrate with pre-placed Ni-Co-Cr-Al-Y powder was studied. A good coating without cracks and pores was obtained in a proper laser process. The microstructure of the coating was examined using SEM and EDS. There is a metallurgical interface bonding between the coating and the substrate. The microstructure of Ni-base alloy coating is homogeneous austenite g-Ni in cell and cell-dendrite morphology. The result of Vickers hardness tests that the average micro-hardness of the coating is HV800¨HV1000, and it is two times higher than that of Ti-6Al-4V substrate.

Lu et al. (2015) [7] described that laser clad Ni60/h-BN self-lubricating anti-wear composite coating on 304 stainless steel were heat treated at 600 ◦C (stress relief annealing) for 1 hour and 2 hour, respectively. Effects of the phase compositions, microstructure, micro hardness, nano-indentation and tribological properties of the composite coatings with and without heat treatment had been investigated systemically. Results indicated that three coatings mainly consist of the matrix (Ni, Fe) solid solution, the CrB ceramic phases and the h-BN lubricating phases. Compared with the coating without heat treatment, the friction coefficients of the coating after heat treatment were decreased obviously. Effects of the heat treatment time on friction coefficient were negligible, but were significant on wear volume loss. Comparatively speaking, the laser clad self-lubricating anti-wear composite coating after heat treatment for 1 h presented the best anti-wear and friction reduction properties.

Sivandipoor et al. (2012) [13] described that WS_2 particles, as solid lubricant, were incorporated in electro less nickel–phosphorous to produce a new composite coating with enhanced tribological properties. Coating deposition was carried out in a laboratory electro less bath followed by heat treatment at 400 °C. The process included pre-treatment of steel substrate by grinding and polishing to reach an appropriate roughness, surface activation by degreasing and acid cleaning and preparation of WS_2 powder before addition to the electro less bath. Evaluation of friction coefficient and wear resistance was performed by a pin-on-disc tribotester at room temperature.

3. EXPERIMENTAL PROCEDURE

Laser cladding

SS304 of dia 6 mm and length 3.5 cm is used as the substrate material. The substrate surface was cleared with acetone before laser cladding. The Ni/WS_2 mixed powder, a mixture of 60 wt% Ni powder and 40 wt % WS_2 was taken. The particle size of Ni-powder was 50 μm and particle size of WS_2 was 200 nm. The powder was pasted on steel substrate using an organic binder (4 wt% polyvinyl alcohol, VA) to form a layer of approximately 0.7 mm thick. Then the specimens with the mixed powder layer were placed in an oven and heated at 40˚C for 2 h. After cooling in air, 304ss coating was performed using the fibre laser operating at 1070±10nm wavelength. The laser processing was conducted in an argon shielding atmosphere. The process parameters are shown in Table 1.The schematic diagram of the laser cladding process is shown in Fig. 1.

Table 1: Process Parameters of Laser Cladding process

Sample	laser power (w)	scanning speed (MM/min)	thickness of coating	spot diameter (mm)
1	210	240	0.7	1
2	100	240	0.7	1

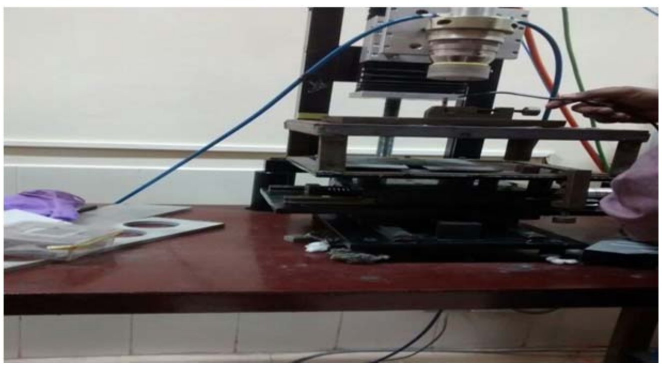

Fig. 1: Laser Cladding Process

Electroless coating

SS304 substrate with diameter of 6 mm and 3.5cm length was prepared by performing cutting on stainless steel rod by Wire EDM, followed by grinding and other machining operations.

An electro less bath is made consisting of Nickel sulphate solution (base), makeup solution, distilled water or de-ionized water in proportion given in table;

Table 2

COMPONENTS	CONCENTRATION
Nifoss 2500 Base	30 ml/0.5litre
Nifoss 2500 Makeup	100 ml/litre
De-ionized water	370ml/litre

The tank was filled with this quantity of solution and temperature and pH were noted of the solution. The temperature is found always +2 degree of the room temperature. Generally, pH value was observed less than 4.8, which is optimum for the bath. pH was increased by adding 10% potassium hydroxide solution and in order to lower the value of pH 10% sulphuric acid was added.

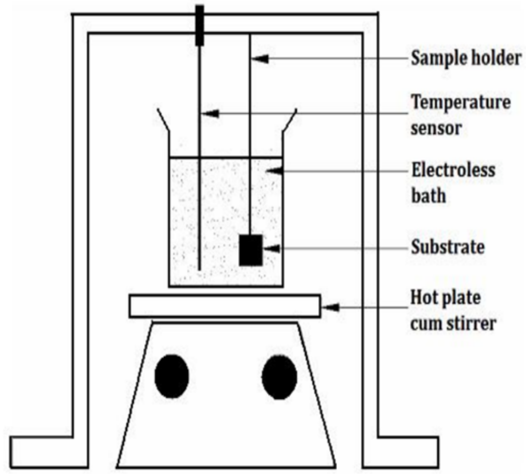

Fig. 2: Sketch of electro less setup

Then electro less bath tank is kept on magnetic stirrer for heating purpose for attaining the desired temperature as required by the electro less bath.

Mild steel plate of any dimension is kept inside the electro less bath just as the heating process starts in order to start bubble formation within the bath and thus chemical reaction starts within the solution. After 20–30 minutes, mild steel plate is taken out and substrate (SS304) is dipped in bath with help of specimen holder.

Reducer and Base should be mixed in same proportion in the bath in order to complete the reaction. It is added every 30 minutes from time SS304 is dipped in the electro less bath.

As due to continuous heating, level of solution keeps on decreasing with time, so it should be regularly topped up with de-ionized or distilled water to maintain the same level of bath as was initial.

By changing the parameters, coatings have been done on the substrates as:-

SUBSTRATE 1:

SS304 (Substrate) is one side chamfered and one side surface roughened and only nickel plating is carried out on surface roughened side. Parameters used are same as mentioned in table 2.

Fig. 3

SUBSTRATE 2:

Electro less nickel plating was carried on unchamfered side of SS304 with WS_2 and AL_2O_3.

Base	30ml/500ml
Make up	100ml/500ml
De-ionized water	370ml/500ml
Al_2O_3	0.7g
WS_2	0.3g

Fig. 4

Initially, 1 hour only substrate was dipped in bath so that nickel coating starts from discrete ends and then lateral growth occurs. After 1 hour, WS2 and AL2O3 were added and heated for next 2 hour.

SUBSTRATE 3

Electro less nickel plating was carried on chamfered side of SS304 with WS2 and AL2O3 .

Base	15 ml/250ml
Make up	50ml/250ml
De-ionized water	185ml/250ml
Al2o3	0.5g
WS2	0.3g

Fig. 5

4. EXPERIMENTAL RESULTS

The surface hardness is measured by the UHL VMHT microhardness tester with an indentation load of 0.5 kg and dwell time 15 seconds before and after the coating. Hardness measurement on the electro less Ni-Al_2O_3-WS_2 coatings and laser cladding Ni/WS_2 coatings is repeated for five times at different locations and the average of the measurements is taken as the response for the actual experiment. The hardness of the sample which is measured in Vickers hardness (HV) scale. However the HV value can be converted to Rockwell (HRc), Knoop (HK) as well as Brinell hardness scale. The Hardness values of Electroless and Laser cladding has increased. Hardeness values after Electroless coating on substrate 1, has increased from 131HV to 421.4HV, on substrate 2 has increased from 131HV to 481.4HV and on substrate 3 it increased from 356 HV to 409.5HV and in Laser cladding hardness its value has increased from 131HV to 278.5HV respectively.

5. CONCLUSION

From the result of hardness it is revealed that the deposition of Nickel, Aluminium oxide and the Tungsten di sulphide on SS304 substrate increases the hardness of the coated sample than the base metal. The hardness of the deposits could be increased by adding Aluminium oxide either in an amorphous or crystalline state. By varying the percentage of the Tungsten di sulphide and Aluminium oxide present in the coating, the hardness of the coating can be varied.

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