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Enhanced Droplet Erosion Resistance of Laser Treated Nano Structured TWAS and Plasma Ion Nitro-Carburized Coatings for High Rating Steam Turbine Components

B.K. Pant, Vivek Arya, and B.S. Mann

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This article deals with surface modification of twin wire arc sprayed (TWAS) and plasma ion nitro-carburized X10CrNiMoV1222 steel using high power diode laser (HPDL) to overcome water droplet erosion occurring in low pressure steam turbine (LPST) bypass valves and LPST moving blades used in high rating conventional, critical, and super critical thermal power plants. The materials commonly used for high rating steam turbines blading are X10CrNiMoV1222 steel and Ti6Al4V titanium alloy. The HPDL surface treatment on TWAS coated X10CrNiMoV1222 steel as well as on plasma ion nitro-carburized steel has improved water droplet resistance manifolds. This may be due to combination of increased hardness and toughness as well as the formation of fine grained structure due to rapid heating and cooling rates associated with the laser surface treatment. The water droplet erosion test results along with their damage mechanism are reported in this article.

Keywords control valve, diode laser, LP bypass valve, plasma ion nitro carburizing, stainless steel, steam turbine, TWAS, water droplet

1. Introduction

The shape and design of a steam turbine moving blades determine how much of kinetic energy is converted into power and conversion of this kinetic energy becomes a criterion for the measurement of efficiency. Lower kinetic energy leads to lower losses and higher steam turbine efficiency. This loss is proportional to the square of the ratio of the volume flow rate of steam through the last stage and annulus areas of the exit of the turbine. To minimize the losses, a large exit annulus area of the steam turbine is needed. Increase in last stage blade annulus area can be accomplished either by using longer blades mounted on smaller rotor diameter or using shorter blades mounted on a larger diameter (Ref 1). GE-Toshiba has optimized these blade design based upon aerodynamic and mechanical considerations. For 1310 MW super critical steam turbine Mitsubishi Heavy Industries Ltd has optimized new 74 inches for last stage moving blades based upon high strength steel and titanium alloy. GE-Toshiba is working on optimizing hybrid titanium and steel alloy blades. Due to their large size and high rpm (3600), the droplet erosion of these blades is a challenging problem. Attempts to reduce erosion damage either by laser hardening, by hard thermally sprayed coatings or by stelliting are being made and success up to a certain level has been achieved (Ref 1-4). At present edges of low pressure steam turbine (LPST) blades are also protected either by induction or flame hardening at 980 ± 5 °C or by fixing stellite plates of length up to 320 mm from the blade tip toward the root by brazing (Ref 3, 4). These plates are generally 320 mm long, 12 mm wide, and 1.50 mm thick and made from stellite 6 alloy having a hardness of 421 HV_{10} for a typical 200 MW steam turbine. During operation there are chances that these plates may come out due to the difference in thermal expansion of stellite with the base materials. In addition to LPST moving blades, the droplet erosion is a significant problem in the control valves and LP bypass valves, generally on the inner side of the casing (Ref 5). The material generally used for the casing is X10CrMo910 (0.14C, 0.28Si, 0.65Mn, 2.4Cr, 0.91Mo, P&S < 0.014 bal. Fe). Significant corrosion in combination with droplet erosion in control valves is due to the formation of larger droplets impinging at high speed exceeding 240 m/s.

Among various hard thermal spray coatings, twin wire arc using cored wires are gaining interest for application of erosion and corrosion due to their high spray rate, reduced set-up time, higher deposit efficiency (60 to 70% as against 30 to 35% for HP-HVOF coating), reduced coating cost (20 to 30% cheaper as compared to HVOF coating). Advanced liquid based HP-HVOF systems generally accelerate WC particles more than 750 m/s leading to such lower deposit efficiencies (Ref 6). These coatings have low

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porosity and high hardness as compared to conventional HVOF sprayed coatings (Ref 7-12). Recently, Nano steel has reported an achievement of nano scale coating by twin wire arc spraying process that compares well with HVOF coatings (Ref 7). This new development with SHS 7170 cored wire was enabled by extensive alloy design studies which revealed specific combination of atom ratio that readily form metallic glass structures at cooling rate in the range of thermal spray (10^4 to 10^5 K/s). During second heat treatment above 700 °C these glass precursors readily transform into nanoscale composite structure. The ability to develop nano scale composite coating while processing in air is well known (Ref 7, 9-11) and results in coating with excellent combination of properties including high bond strength, hardness, wear, and corrosion resistance. Recently developed coatings to resist high temperature erosion using cored wire are Tafa 140 MXC (Cr < 25, Mo > 6, Nb < 12, Mn < 3, W < 15, Si < 2, C < 4, B < 5, bal. Fe), SHS 7170 alloy (Cr < 25, Mo < 10, W < 10, Mn < 5, B < 10, C < 5, Si < 5 bal. Fe), Alpha 1800, Armcore M, and Ducor (Ref 7, 12). The melting temperature of SHS 7170 alloy is around 1200 °C.

These coating materials especially SHS 7170 produced by twin wire arc sprayed (TWAS) technique gives bond strength more than 450 kg/cm^2, micro-hardness values more than 1000 HV_{300} and has excellent wear and corrosion resistance (Ref 7). The cored wire SHS 7170 and 140 MXC coatings are meant for application at high temperature to resist erosion due to abrasive coal and fly ash. The 'as-sprayed' coatings contain a high fraction of two-dimensional defect phase boundaries. To eliminate these defects, these coatings are recommended to be heat treated above 700 °C for a short duration (Ref 7, 12) and it has been reported that carbide and boride type nano hard phases of 66% volume fraction are formed $(Fe\text{-}Cr\text{-}W\text{-}Mo)_{23}C_6$ and $(Fe\text{-}Cr\text{-}Mo\text{-}W)_3B$, which are responsible for improved performance. As-sprayed coatings give rise to tensile residual stress, in fact with increased thickness these values exceed the bond strength and failure in the form of detachment takes place. However, after post spray heat treatment, these materials cause the devitrification of the metallic glass contents and this results in very low residual stresses, excellent wear resistance, and better impact properties (Ref 7). Twin wire arc cored wires coatings treated with high power diode laser (HPDL) will be effective in tackling such problems of droplet erosion for these big components and will be economical because of high component and outage cost.

In the present study SHS 7170 coating by TWAS system and nitro-carburizing by plasma ion process have been selected to combat droplet erosion. Plasma ion nitro-carburizing is a process by which the surface hardness of steels can be increased by diffusion of carbon and nitrogen atoms up to around hundred micron depth (case) under the surface. It is carried out in the ferritic region without any phase transformation. The solubility of carbon and nitrogen in ferrite steel is small and most of the carbon and nitrogen that enters this steel forms hard carbides and nitrides. Plasma ion nitro-carburizing is generally carried out on steel substrates using ammonia and LPG gases. Plasma ion nitro-carburized and TWAS-coated steel samples have been treated using HPDL and their water droplet erosion test results are reported in this article.

2. Experimental Procedure

2.1 Twin Wire Arc Spraying

Using Hobart Tafa 9000 twin wire arc spraying system, the SHS 7170 cored wire (1.6 mm diameter) was sprayed on steel round (∅ 12.7 × 40 mm) samples. Spraying parameters such as arc voltage, current, spray distance, rotation, and transverse speed were optimized and manipulation of the samples during coating was done by six plus two axis robot. The samples were grit blasted using 24 mesh alumina grit at an air pressure of 5 to 5.5 kg/cm^2 (g). The spraying parameters used were as per those given in the manual. Figure 1 shows the twin wire arc gun mounted on a robot. Table 1 gives the TWAS details and parameters (Ref 11).

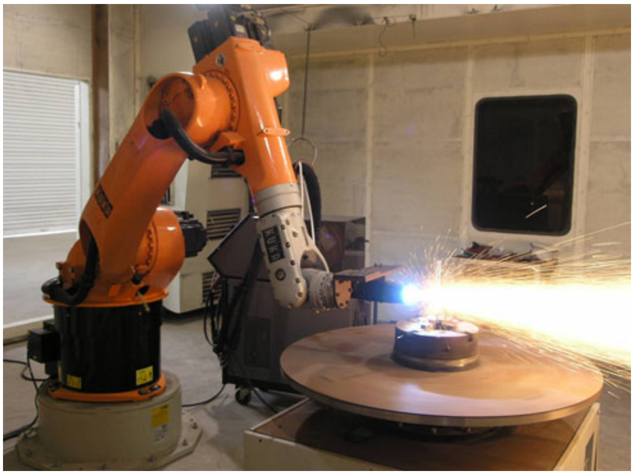

Fig. 1 Set-up showing twin wire arc spraying (TWAS) on round samples using robot

Table 1 Twin wire arc spraying parameters (Ref 11)

Equipment type	Hobart Tafa, TWAS 9000
Primary air pressure	5.44 kg/cm^2 (g)
Nozzle cap/positioner	Green/Long C
Arc voltage	33 V
Arc current	200 A
Round sample size	12.70 dia × 40 mm length
Spray angle	90°
Circumferential velocity of round samples	40 mm/s
Linear movement of TWAS Gun	20 mm/s
No. of passes	3
Stand off distance	110 mm
Coating thickness:	350-400 micron

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2.2 Plasma Ion Nitro-Carburizing

Plasma ion nitro-carburizing is a process by which the surface hardness of steels can be increased by diffusion of carbon and nitrogen atoms up to a few hundred micron depth (case) under the surface. It is carried out in the ferritic region without any phase transformation. The solubility of carbon and nitrogen in ferrite is small and most of the carbon and nitrogen that enters the steel forms hard nitrides and carbides. In typical gas nitro-carburizing, the outer layer formed is called the ‘‘white layer’’ which is hard and brittle. This layer has to be removed by grinding. However, in plasma ion nitro-carburizing this white layer can be avoided by a proper choice of plasma parameters including temperature, and LPG, NH_3 gases flow rates (1-1.5% LPG in the LPG/NH_3 mix) as well as the applied and bias voltage (480VDC). Plasma ion nitro-carburizing of the steel samples was carried out in a furnace in which the white layer was completely eliminated. The following plasma ion nitro-carburizing parameters were used (Table 2).

2.3 HDPL Surface Treatment

Laser treatment of materials using HPDL is a very versatile technique for surface treatment of materials (Ref 13, 14). HPDL surface treatment was carried out on each sample using 4.6 kW diode laser with the laser head mounted on a six plus two axis robot. Optics of ‘30 by 3.0 mm’ was used to produce the laser beam at focal length of 275 mm. Laser beam power was controlled in a closed loop by a two color pyrometer and a uniform surface temperature of 1550 °C was maintained at which temperature the TWAS coating gets remelted and metallurgically modified (change in porosity, fracture toughness, hardness). The complete system was controlled by the robot controller. The robot was programmed in such a way that the laser beam tracked the round sample at a speed ranging from 1 to 5 mm/s ensuring that hardening of the sample was completed in one pass. A wide area having a span of 30 mm on the outer periphery of the round was laser hardened. Uniform compressed air cooling of the round sample was maintained throughout the HPDL treatment.

Droplet erosion samples of size ∅ 12.7 by 40 mm length having internal threading M8 were made of X10CrMoV1222 stainless steel. A fixture was fabricated to hold and rotate these samples while carrying out HPDL surface treatment. Each sample was fixed in a self centered three jaw chuck at one end and supported on a fixture on the other end. The fixture has a rotating seal, so that the samples can rotate freely and air used for cooling the samples does not leak. Rapid cooling of the sample was carried out during HPDL treatment by introducing compressed air having volumetric flow rate of 15 to16 m^3/h through the M8 tapped hole. This air is capable of removing heat at a rate of 160 to 180 W which is comparable to the heat removed in a bulk stainless steel during laser surface treatment. The complete setup is shown in Fig. 2. The samples were thoroughly cleaned using acetone before the start of the experiment to make the surface free from dust, oil etc.

Table 2 Nitro-carburizing parameters

Sample for plasma ion nitro-carburizing	Nitro-carburizing temperature	Chamber pressure	Process duration	Bias DC voltage
X10CrMoV1222	530-550 °C	1.8 Torr approx.	8 h	480 V

2.4 Conventional Heat treatment of X10CrNiMoV1222 Steel

Conventional heat treatment of X10CrNiMoV1222 steel samples (LPST blade material) was also carried out to make a comparison with the HPDL treated X10CrNiMoV1222 steel samples. Precisely controlled heating of these samples was carried out at a temperature of 980 ± 5 °C in a conventional electric furnace followed by oil quenching to achieve hardness in the range of 450-500 HV which is generally obtained either by flame or high frequency induction hardening techniques. These samples along with the HPDL surface-treated X10CrNiMoV1222 steel samples were studied for water droplet erosion.

2.5 Droplet Erosion Testing of HPDL Surface Treated Samples

Erosion caused by liquid droplet impact has similarities to that arising due to cavitation bubble collapse. The details of erosion damage mechanism for a particular material due to cavitation or liquid droplet impact are given in Ref 15, 16. The forces causing this deformation and erosion are micro-jet impacts occurring when the bubbles collapse. In addition to micro-jets, high pressure shock waves are also generated which weaken the materials. Erosion is mainly caused by micro-jet impacts arising due to bubble collapse similar to that caused by liquid droplet impact. The erosion has mainly four stages, i.e., incubation, accelerated period, maximum-rate period, and

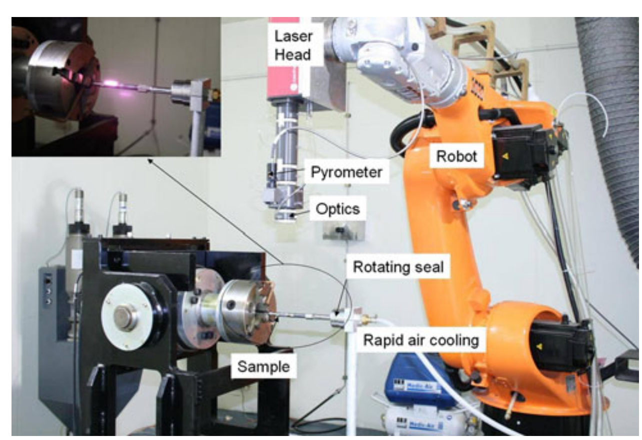

Fig. 2 Set-up showing laser hardening (LH) on round samples using robot

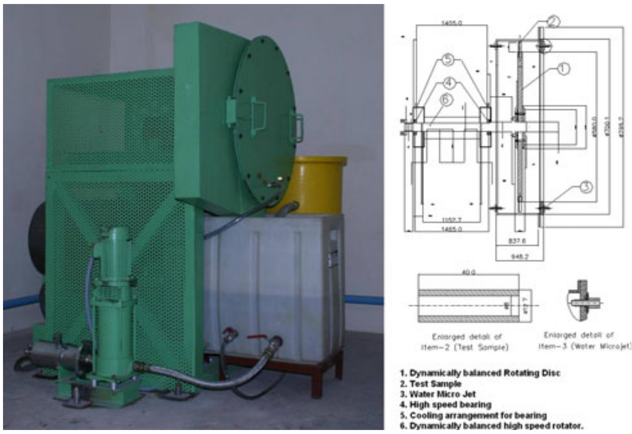

Fig. 3 Water droplet impingement erosion test facility

Table 3 Experimental test conditions

Conditions	Test parameters
Volume of water impacted per cycle	0.035 mL
Water impact energy (1/2 mV^2)	0.380 J
Water energy flux, J/m^2s	57.167×10^6
Water mass flux	4.0 m/s
Relative water velocity	147.6 m/s
Test sample size	∅ 12.70 × 40 mm
Number of specimens used	12
Test duration cycles	8.55×10^6
Angle of impact	0-90°
Impact frequency	79.166 cycles/s
Experimental accuracy	±15.5%

steady state. During incubation period, the erosion rate is negligible compared to the other stages.

The details of droplet erosion test facility are given in Ref 2. In short the test facility consists of a 700 mm diameter chamber and a round stainless steel disc where the test samples are positioned. Samples, 40 mm in length and 12.7 mm in diameter are affixed on the periphery of the disc. The disc is rotated at 79.166 cycles/s to obtain the test sample tangential velocity of 147.0 m/s. Two water jets impinge on the cylindrical test samples and cause impingement erosion. As such, a relative velocity of 147.6 m/s is obtained. The mass of water impacted from these jets per cycle is 0.035 mL equivalent to energy flux values of 57.167×10^6 J/m^2 s, respectively. The details of the test set up are given in Fig. 3 and experimental values of test parameters are given in Table 3.

Water droplet impact energy of 800 micron droplet (1/2 mV^2) for 74 inches size moving blade of 1310 MW supercritical steam turbine rotating at 3000 rpm may exceed 0.15 J. A precision balance (±0.1 mg) was used for measurement of mass loss after testing. The test duration depending upon energy and mass fluxes was selected in such a way as to achieve steady-state erosion in limited cycles. The accuracy and repeatability of the tests have been established on X10CrNiMoV1222 steel sample which is taken as a reference material. The extent of erosion damage is calculated as cumulative volume loss

Table 4 Materials used for droplet erosion testing

Materials	Composition, wt.%
X10CrNiMoV1222	0.1C, 0.25Si, 0.7Mn, 12Cr, 2.5Ni, 1.75 Mo, 0.3 V, bal. Fe
SHS 7170 wire	Cr < 25, Mo < 10, W < 10, Mn < 5, B < 10, C < 5, Si < 5, bal. Fe

(cumulative mass loss divided by the material density). The results have been plotted in the form of cumulative volume loss versus number of cycles. Table 4 gives the materials used for HPDL treatment and for droplet erosion evaluation.

2.6 Testing and Characterization

The Vicker's micro-hardness of HPDL treated and untreated samples was measured by using Tukon 2100 Macro/Micro-hardness tester by applying a load of 300 g with a dwell time of 13 s. Five measurement of hardness at different locations were taken per sample and average of the results has been reported in the article. The fracture toughness (k_{1c}) of the surface coating plays a significant role in the droplet erosion resistance and it was evaluated by the surface indentation technique (Ref 17) using the same Tukon 2100 hardness tester. Loads up to 30 kg were applied on the coatings and the indent diagonal (a) and crack length (c) were measured. X-ray diffraction was carried out by Philips X-pert system using copper K alpha radiation and nickel filter in the present study.

3. Results and Discussion

3.1 Micro-Hardness and Fracture Toughness

The micro-hardness values of TWAS 'as-sprayed' (TWAS AS) SHS 7170 coating is 1118 HV_{300} (standard deviation 12 HV_{300}). After laser treatment, it was observed that the hardness of the sample (TWAS LH) dropped to 910 HV_{300} in the laser melted region. The micro-hardness values of nitro-carburized X10CrNiMoV1222 NC sample was 1139 HV_{300} (standard deviation 43 HV_{300}). After laser treatment, these values dropped significantly to 816 HV_{300} (standard deviation 34 HV_{300}) throughout the entire nitro-carburized case having depth of around 80 micron. Laser treatment has helped the coatings to develop a better combination of hardness and toughness as compared to the untreated sample and has hence given a much better performance in the droplet erosion testing.

Both these coatings did not show any cracks on application of load after laser treatment, whereas in samples without laser treatment, showed the development and propagation of cracks. Figures 4 to 7 shows the indentations on the coatings before and after laser treatment. The k_{1c} values, calculated by the Evans-Charles formula (Ref 18), for "TWAS SHS 7170 LH" is 7.15 MPa√m (average of 3 readings) at 30 kg load whereas for "TWAS SHS 7170 AS", this value is 3.77 MPa√m at a load of 5 kg.

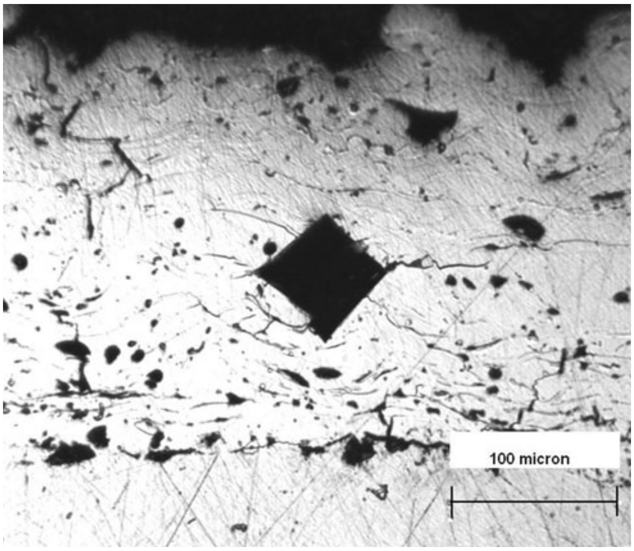

Fig. 4 Diamond pyramid impression on TWAS SHS 7170 sample for fracture toughness at 5 kg load and 100× magnification

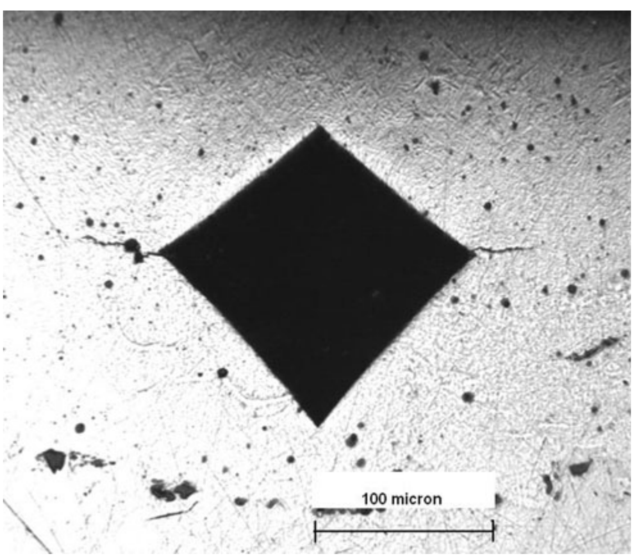

Fig. 5 Diamond pyramid impression on TWAS SHS 7170 (LH) sample for fracture toughness at 30 kg load and 100× magnification

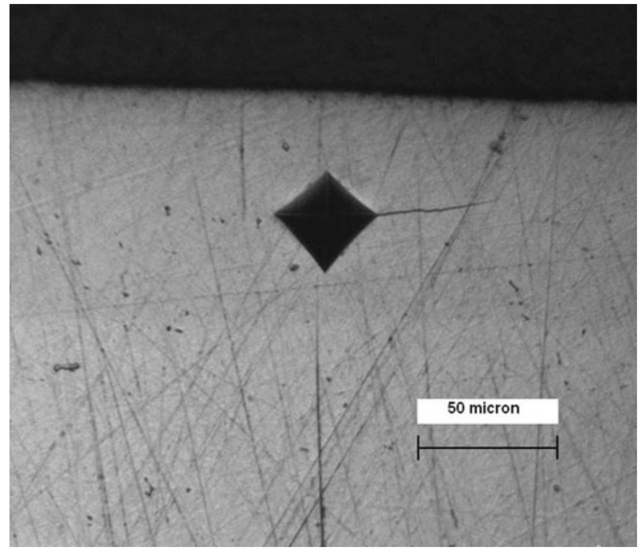

Fig. 6 Diamond pyramid impression on X10CrNiMoV1222 (NC) sample for fracture toughness at 1 kg load and 200× magnification

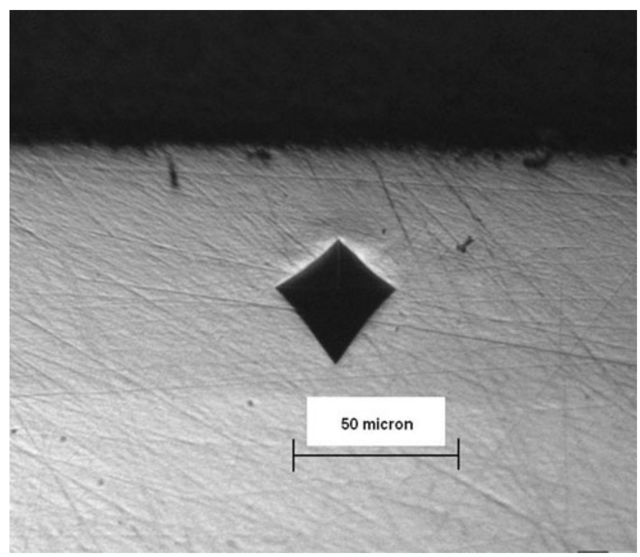

Fig. 7 Diamond pyramid impression on X10CrNiMoV1222 (NCLH) sample for fracture toughness at 2 kg load and 200× magnification

Measurement at 30 kg load for "TWAS SHS 7170 AS" coating could not be carried out since the coating got shattered at this load.

Similar observations were made for plasma ion nitro-carburized X10CrNiMoV1222 steel sample before and after laser treatment. Cracks begin to develop in X10CrNiMoV1222 NC samples at 1 kg load itself whereas there is no such crack initiation in X10CrNiMoV1222 NCLH samples even at 2 kg load. However, the k_{1c} values could not be calculated for these samples because at the load required for reasonable crack formation, the indentation diagonal was found to have dimensions similar to the nitro-carburizing case depth (80 micron). These results suggest that a significant improvement in fracture toughness has been achieved due to laser treatment. This is one of the main reasons of improved erosion performance of both types of coatings (TWAS and plasma ion nitro-carburized layers) after laser surface treatment.

3.2 X-Ray Diffraction Test Results

XRD analysis of plasma ion nitro-carburized (NC) and TWAS-coated X10CrNiMoV1222 (SHS) steel samples before (AS) and after laser treatment (LH) are shown in

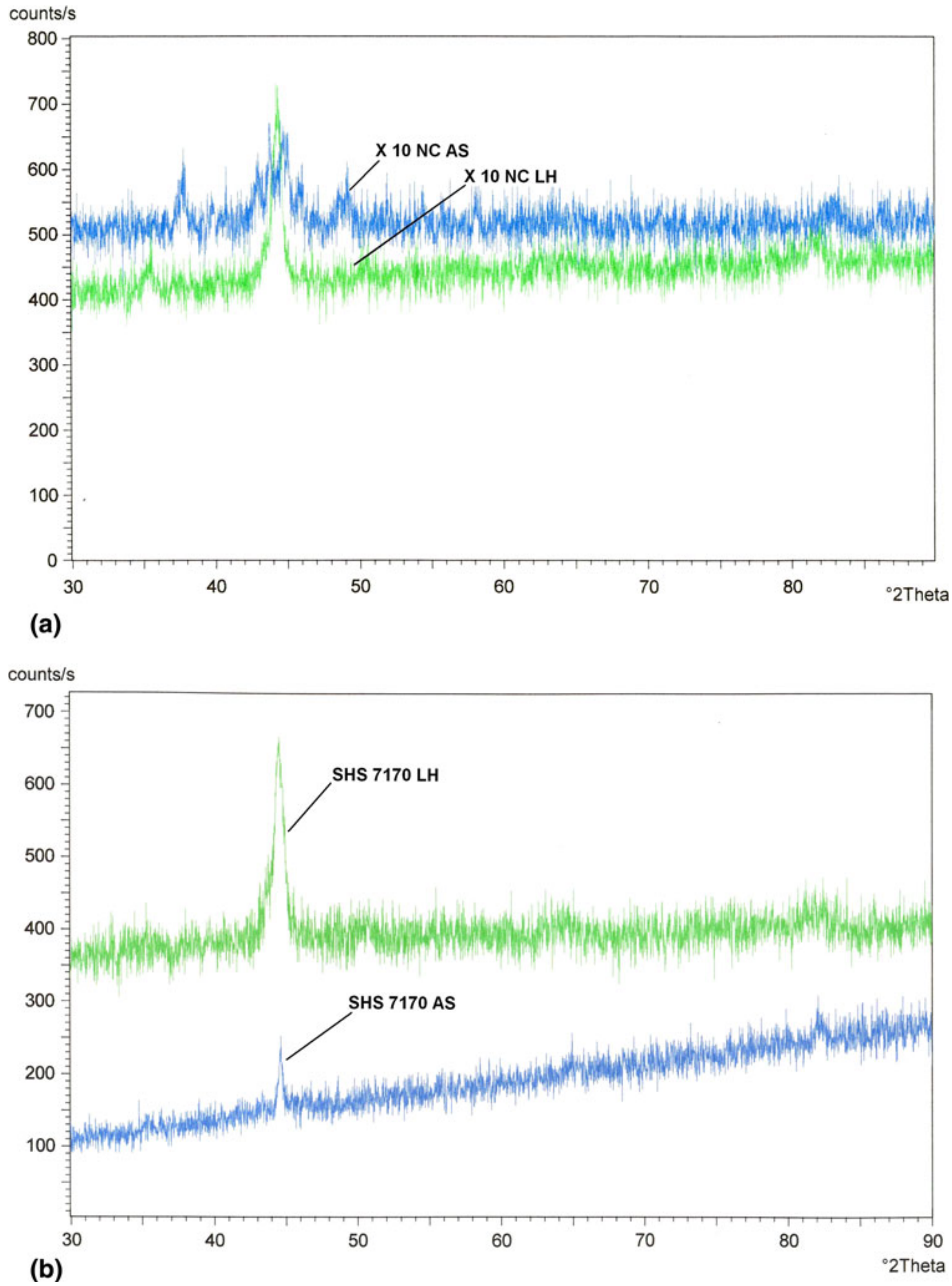

Fig. 8 (a) XRD of X10CrNiMoV1222 (NC) and X10CrNiMoV1222 (NCLH) sample. (b) XRD of TWAS SHS 7170 and TWAS SHS 7170 (LH) sample

Fig. 8(a) and (b). XRD analysis confirms that there is no significant change in phases before and after laser treatment.

3.3 Droplet Erosion Test Results

The droplet erosion test results of HPDL surface treated and untreated plasma ion nitro-carburized as well as TWAS-coated X10CrNiMoV1222 along with uncoated X10CrNiMoV1222 steel sample are given in Fig. 9. It is seen from above figure that plasma ion nitro-carburized after HPDL treatment is much superior to untreated ones for water droplet energy flux levels of 57.167×10^6 J/m^2 s. The incubation period for these coatings after HPDL treatment has also got prolonged. Details on the water analysis used for the study is given in Ref 5.

The TWAS SHS 7170 as-sprayed coating peeled off within 10 min (47500 revolutions) of droplet erosion

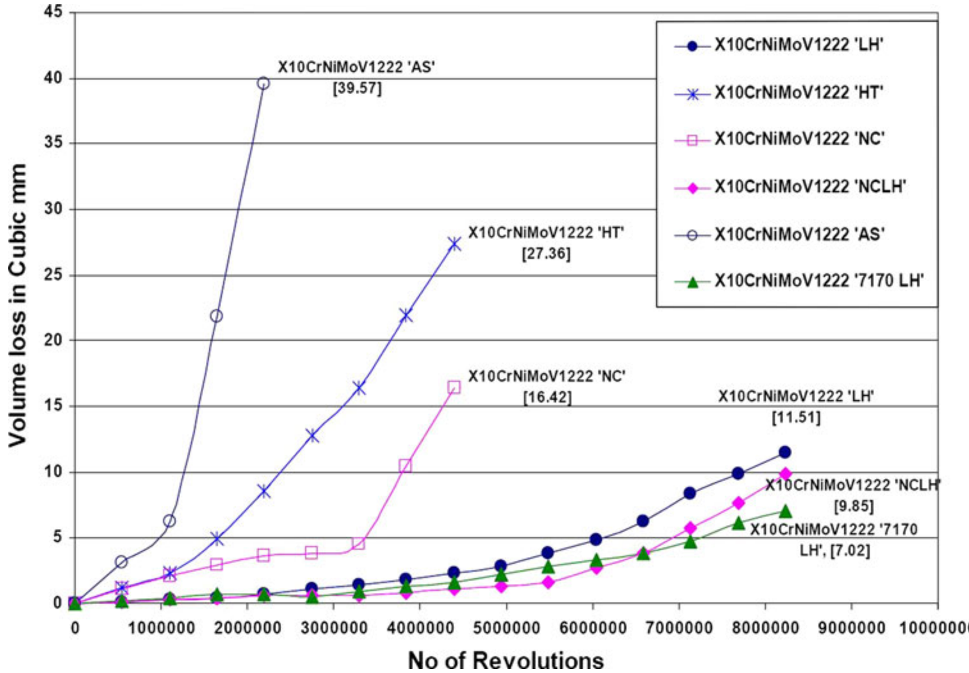

Fig. 9 Volume loss for various surface treatments at energy flux of 57.167×10^6 J/m^2 s

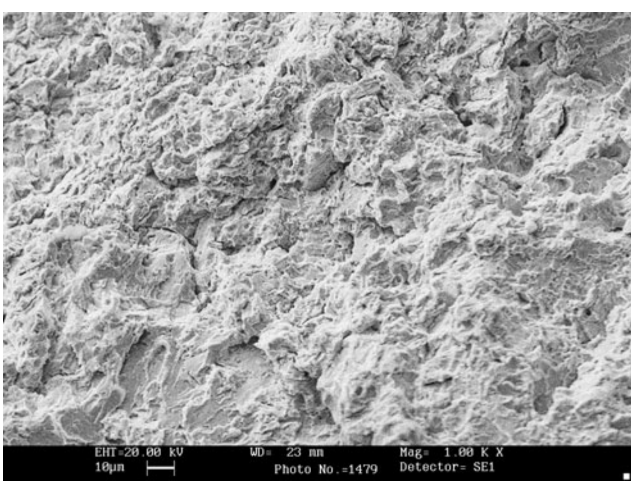

Fig. 10 SEM of heat-treated X10CrNiMoV1222 sample showing presence of cracks

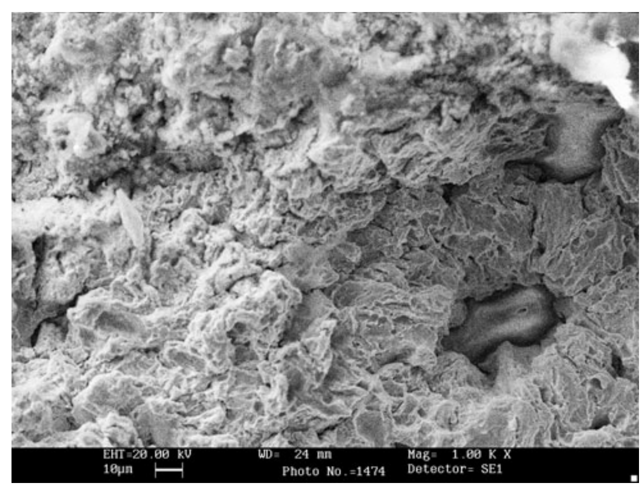

Fig. 11 SEM of X10CrNiMoV1222 (NC) sample showing coarse grains and cleavage fracture

testing whereas the TWAS SHS 7170 coating after laser treatment remained intact even after 8 million revolutions. This indicates greatly enhanced bond strength and much superior droplet erosion resistance. The bond strength of the coating (as-sprayed TWAS and after laser treatment) has been measured using adhesion tester as per ASTM C 633-01(2008). It was recorded as 20 MPa for the as-sprayed which is the main reason for its peel-off failure in droplet erosion testing whereas after laser treatment the bond strength exceeded 42 MPa (adhesive peeled off before coating) indicating that laser treatment has substantially enhanced the bond strength.

3.4 Scanning Electron Micrographs

The scanning electron micrographs of droplet erosion tested X10CrNiMoV1222 steel and TWAS SHS 7170 samples are shown in Fig. 10 to 14. After the combined treatment of nitro-carburizing and laser, it is observed that in the X10CrNiMoV1222 steel there is a clear reduction in grain size as compared to the untreated one. The X10CrNiMoV1222 NCLH sample has performed very well in the droplet erosion testing showing a direct bearing of grain size on droplet erosion performance. The microstructure of the sample which was first coated with SHS 7170 by TWAS process and subsequently laser treated

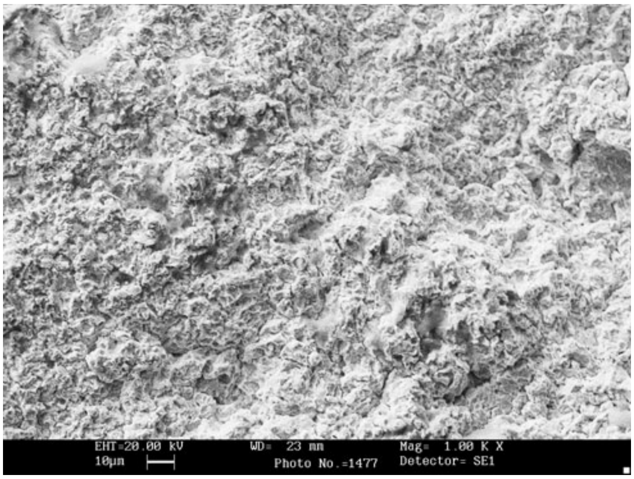

Fig. 12 SEM of X10CrNiMoV1222 (NCLH) X10 sample showing refinement of grains

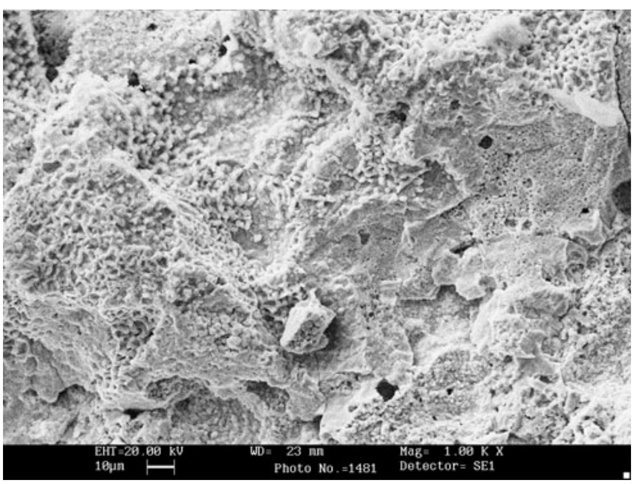

Fig. 13 SEM of TWAS SHS 7170 (LH) sample showing the presence of fine nodules

(Fig. 13) shows the presence of fine nodules and very fine pores. This sample has also performed very well in the droplet erosion testing. The SEM of conventional heat treated sample (X10CrNiMoV1222 HT) is also shown (Fig. 10).

4. Discussion

A cross-sectional view of a polished TWAS AS coating shows the presence of internal cracks and high degree of porosity. On laser hardening at 1550 °C, the coating gets melted and rapid cooling during the process results in a vastly improved microstructure (nanostructure) that is largely free of cracks and porosity. The XRD pattern indicates that there is no phase change after laser treatment and therefore the improved droplet erosion properties are mainly due to these micro-structural changes.

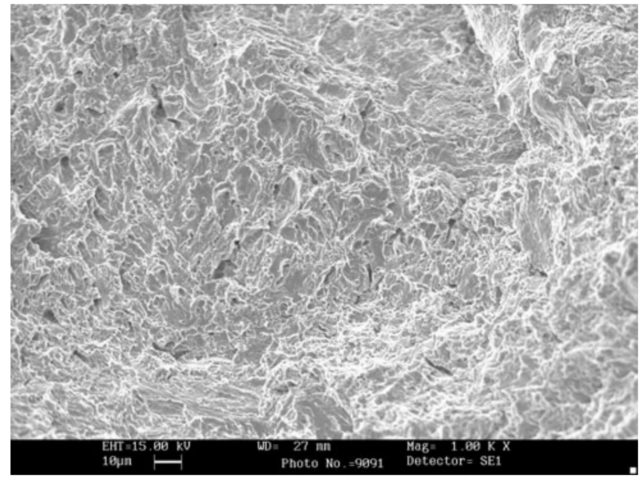

Fig. 14 SEM of X10CrNiMoV1222 (LH) sample showing fine grains

In the case of plasma ion nitro-carburized X10CrMoNiV1222 steel sample, the nitrogen and carbon atoms diffuse into the steel matrix up to around 70-80 micron inside the surface and result in the formation of nitrides/carbides. These nitrides/carbides get dissolved during laser hardening and re-crystallize to form very fine dispersions thereby resulting in marked dispersion strengthening. This is the reason for superior performance in droplet erosion resistance.

5. Conclusions

1. The droplet erosion results show that the X10CrMoV1222 steel sample after TWAS coating and laser treatment has shown a superior performance as compared to uncoated ones.
2. The micro-hardness values of TWAS 'as-sprayed' SHS 7170 coating lies between 1120 and 1150 HV_{300} and after laser treatment these values have dropped significantly (910 HV_{300} in the laser melted region). The micro-hardness values of plasma ion nitro-carburized layers lies between 1055 and 1247 HV_{300} and after laser treatment these values have also dropped significantly (644 to 856 HV_{300}).
3. While evaluating coatings for fracture toughness using the surface indentation technique, plasma ion nitro-carburized layer and TWAS SHS 7170 coated sample after laser treatment did not show any cracking. Laser treatment has helped the coatings to develop a better combination of hardness and toughness as compared to the untreated ones and hence give a much better performance in the droplet erosion testing.
4. The SEM study of conventional heat-treated sample (X10CrNiMoV1222 HT) shows relatively large grains. In samples with combined treatment of nitro-carburizing and laser, there is a clear reduction in the

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grain size. This combination has improved the droplet erosion resistance manifold showing a direct bearing of grain size. The microstructure of laser-treated TWAS SHS 7170 sample after droplet erosion testing shows the presence of fine nodules and very fine pores. This has also performed very well in the droplet erosion testing.

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