JETIR.ORG ISSN: 2349-5162 | ESTD Year : 2014 | Monthly Issue
JOURNAL OF EMERGING TECHNOLOGIES AND INNOVATIVE RESEARCH (JETIR)
An International Scholarly Open Access, Peer-reviewed, Refereed Journal

DEVELOPMENT OF THERMAL SPRAY COATING ON AISI H13 STEEL AND ITS CHARACTERIZATION

Vipan Bansal[1], Abhishek Sharma[2], Sunpreet Singh[3]

[1] Assistant Professor, Department of Mechanical Engineering, DAV University, Jalandhar, India

[2] Research Scholar, Department of Mechanical Engineering, DAV University, Jalandhar, India

[3] Assistant Professor, Department of Mechanical Engineering, LPU, Jalandhar, India

[*] *Corresponding author; e-mail: @rediffmail.com*

Abstract: In this research work thermal spray coating was developed on AISI H13 hot forming tool steel. The specimens were heat treated prior to thermal spray coating. The main thermal spray coating parameters in the process were: substrate temperature-500°C; nitriding time-24 hours; pressure-5mbar; voltage-500 V, coating composition-THERMAL SPRAY COATING/POWDER COATING 20:80; duty cycle of 80%, and the frequency used was 30-KHZ. The cross-sectional microstructures of the thermal coated specimens were examined by optical microscopy (OM). A diffusion layer was formed on the material by controlling the nitriding parameters but no white layer was observed during the optical microscopy. Microhardness measurements were performed on the surface and through the diffusion layer for each nitrided material. The as-nitrided specimens were characterized by X-ray diffraction (XRD) analysis and Scanning Electron Microscopy/Electron Dispersive Spectroscopy (SEM/EDS) analysis. The results showed the increased hardness of tool steel with nitrided layer. Further, the cross-sectional SEM micrographs showed the intact and pores free outer nitrided layer in the specimens.

Keywords: Thermal spray coating, hot working tool steel, SEM/EDS and XRD analysis.

Introduction

AISI H13 hot working tool steel combines good red hardness and abrasion resistance with the ability to resist heat checking. It is an AISI H13 hot work tool steel, the most widely used steel for aluminum and zinc die casting dies. It is also popular for extrusion press tooling because of its ability to withstand drastic cooling from high operating temperatures. H13 is produced from vacuum decoating tool steel ingots [1]. This manufacturing practice plus carefully controlled hot working provides optimum uniformity, consistent response to heat treatment, and long service life [2]. H13 is an outstanding die steel for die casting aluminum and manganese. It is used for zinc in long production runs, and also employed successfully for slides and cores in tool assemblies [3]. H13 in the hardness ranges from 45/52 RC is excellent steel for plastic molds. Consider using this grade of hot work tool steel for applications where drastic cooling is required during the operation, and where high red hardness and resistance to heat checking are important. This grade has found wide acceptance for die casting dies for zinc, white metal, aluminum and magnesium. It is also widely used for extrusion dies, trimmer dies, gripper dies, hot shear blades, casings, and other similar hot work applications [4].

Apart from all these advantageous applications when H13 tool steel is to be processed under savere loading conditions it does not retain its surface hardness and results into wear at the contact surface of steel [5]. Therefore, to overcome this limitation surface treatment and coating are developed at the surface of steel so as to increase its hardness and wear resistance properties. Thermal spray coating is one of the surface treatment processes in which nitrogen ions are introduced on the surface of material. But before starting with the thermal spray coating process the surface of the material needs to be heat treated so as to avoid the problem of decarburizing [6]. Thermal spray coating is the nitrogen diffusion process on the subsurface of the steel components. After the development of thermal spray coating over the surface of material a diffusion layer was formed and this formation of diffusion layer as a result of carbon particles present in the steel got diffused by the nitrogen atoms. The diffusion layer is having high hardness resulting into increase in hardness and improved wear resistance of steel. More over no white layer was observed on the surface of steel. The aim of the present paper was to develop the Thermal spray coating on H13 tool steel [7]. Thereafter, the characterizations of the thermal coated samples were done by X-ray diffraction (XRD) analysis and Scanning Electron Microscopy/Electron Dispersive Spectroscopy (SEM/EDS) analysis. The microhardness and surface roughness values were also evaluated. The results showed the increased hardness of tool steel with nitrided layer. Further, the cross-sectional SEM micrographs showed the intact and pores free outer nitrided layer in the specimens.

2. Experimental Details

2.1 Substrate Material

In this experiment commercially available steel AISI H13 which is mostly used for hot working applications was considered as a substrate material. The chemical composition (weight%) of the steel is 0.38 C, 0.30 Mn, 0.03 P, 0.02 max S, 1.0 Si, 5.3 Cr, 0.2 Ni, 1.3 Mo, 0.4 V and 0.22 Cu. The tool steel specimens in the dimensions of 8mm diameter and 50mm length were machined from the base material with the help of linear precision saw at a blade speed of 900rpm (BUEHLER Isomet) Fig. 1.

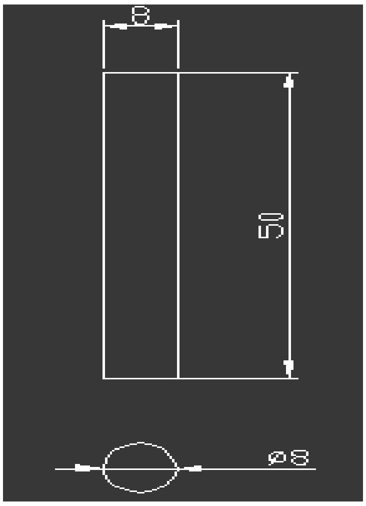


Figure 1- Macrograph of AISI H13 tool steel

2.2 Heat Treatment Details and Procedure

It was found from various studies that heat treatment is required before Thermal spray coating. The same was ensured after discussions with actual industries in Ludhiana. Therefore, to increase the hardness to some extent, heat treatment was performed. This was done at Central Tool Room, Ludhiana. The specimens were austenized at 1020˚C for 30 min, quenched in air and then tempered at 520˚C for 1hour in air. The hardness of the substrate was 22HRC before heat treatment and it increased to 48/49 HRC. Afterwards, the surface of the pins was ground by using SiC emery papers to 240, 320, 400, 600, and 1000 grit size. The final mirror polishing of the specimens was done by using 6 µm and 1µm diamond paste in the presence of water on double disc polishing machine prior to Thermal spray coating.

2.3 Thermal spray coating

Thermal spray coating is the nitrogen diffusion process on the subsurface on the steel components. Typical applications include gears, crankshafts, camshafts, cam followers, valve parts, extruder screws, and pressure-die-casting tools, forging dies, cold forming tools, injectors and plastic-mould tools, long shafts, axis, clutch and engine parts [8]. It was developed at Facilitation Centre for Industrial Plasma Technologies, Institute for Plasma Research (IPR) Gujarat, India. The samples were nitrided in a hot well thermal spray coating apparatus with 80% duty cycle nitriding time-24 hours; pressure-5mbar; voltage-500 V, coating composition-THERMAL SPRAY COATING/POWDER COATING 20:80; and the frequency used is 30-KHZ and the coating temperatures is taken as 500˚C. The Thermal spray coating hot wall system specifications are given in Table 1. The schematic diagram of Thermal spray coating set up is shown in Figure 2.

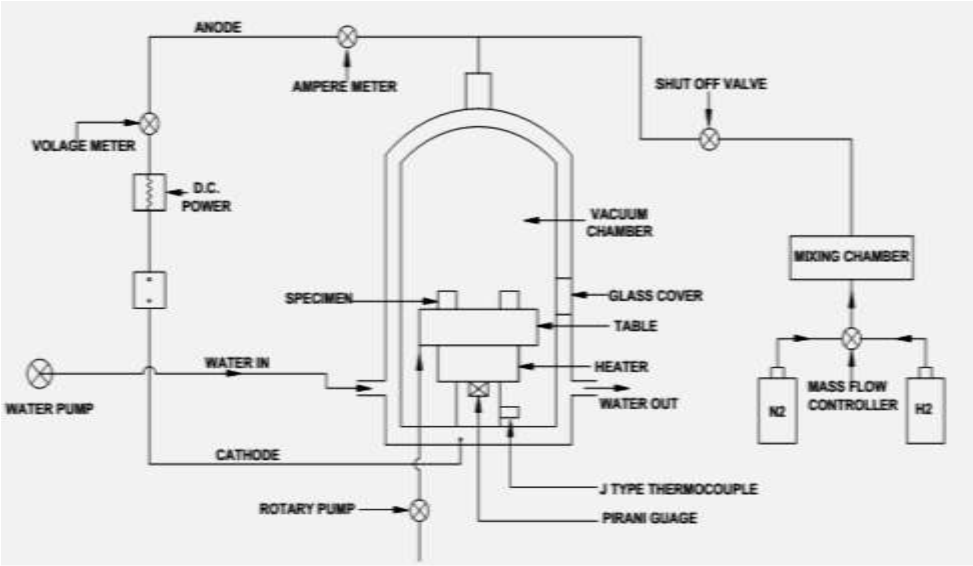


Figure 2- Schematic Diagram of Thermal spray coating Set Up

Procedure:

Step 1- Loading of the samples was dome inside the chamber. The samples were cleaned in acetone, placed in proper sequence and at exact distance. The chamber was closed properly.

Step2- The vacuum chamber was evacuated. The pressure inside the chamber should be below 0.09 mbar.

Step 3- Sputter cleaning was done to remove the surface contaminates from the surface of the specimens for 30 minutes by introducing POWDER COATING coating inside the chamber at temperature 250°C and pressure 0.9mbar.

Step 4- Introduce THERMAL SPRAY COATING and POWDER COATING inside the chamber at 20:80 till pressure 5 mbar is attained.

Step 5- Reaching to the required temperature-500˚C by slowly increasing the voltage and duty cycle up to 80%.

Step 6- After reaching the required temperature, hold for Thermal spray coating for required time. The time is this study was planned for 24 hours. Maintain the temperature by regulating the voltage of the D.C. source.

Step7- After completion, the D.C. power source is off and the chamber left for cooling.

Step8- POWDER COATING flow was stopped for hazards and continued the THERMAL SPRAY COATING flow for fast cooling at the time of cooling. The chamber was not opened before 180°C temperature for avoiding oxidation at atmospheric coatings. The Figure 3 shows the Thermal spray coating inside chamber.

Table 1- Thermal spray coating System Specifications

Coating temperature	500˚C
Diameter	500mm
Height	500mm
Working diameter	400mm
Working height	300mm
Working capacity	50kg
DC pulse power supply	20KW
Heater power capacity	5KW
Cooling	only for O ring

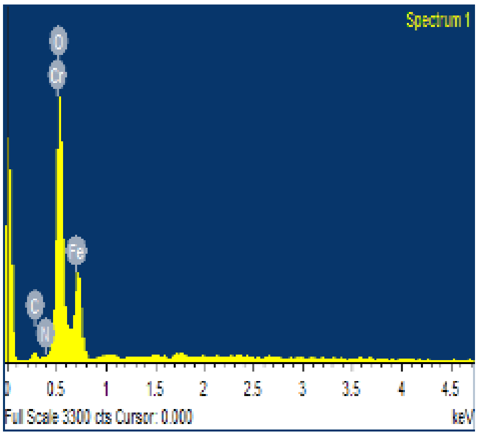
Figure 3- Thermal spray coating inside chamber

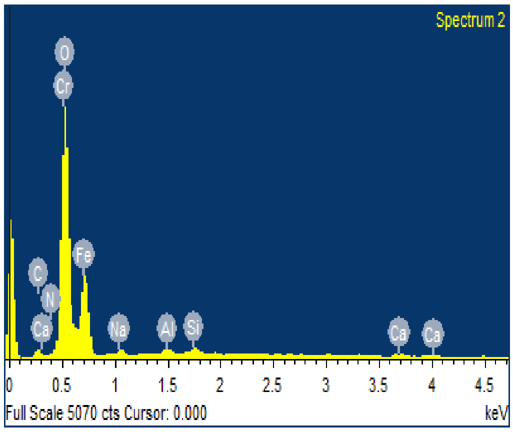
3. Results and Discussion

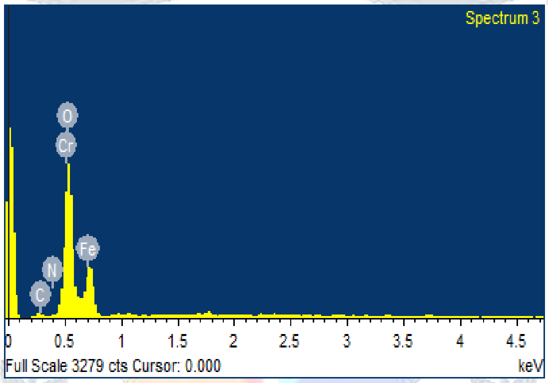
3.1 Optical Microstructure

The optical microstructure of thermal coated layer showed the presence of nitrided layer which is approximately 125 µm thick. This is called case depth of the nitrided layer [9]. It showed formation of diffusion layer on the surface. There was no formation of white layer or compound layer on the cross section of the sample. In the thermal coated sample, the presence of diffusion layer is due to the formation of various phases like $F_{e4}N$ and C_rN etc during thermal spray coating process [10]. Surface and cross-sectional microstructures of the nitrided specimens were examined by optical microscopy (OM). The nitrided specimens were sectioned with a diamond cutter (BAINCUT-LSS, Metallography Low Speed Saw, Chennai Metco Pvt. Ltd., Chennai, India). Thereafter, the cut sections were hot mounted in BAINMOUNT-H (Hydraulic Mounting Press, Chennai Metco Pvt. Ltd., Chennai, India) with transoptic powder so as to show their cross-sectional details. This was followed by polishing of the mounted specimens by a belt sanding machine having emery belt (180 grit). The specimens were then polished manually down to 1000 grit using SiC emery papers. Final polishing was carried out using cloth wheel polishing machine with 1 µm lavigated alumina powder suspension.

Specimens were then washed and dried before being examined under Inverted Optical Microscope interfaced with imaging software Envision 3.0. The same microscope was used to obtain surface microstructures of the coatings.







3.2 SEM/EDS Analysis of Thermal coated H13 Steels

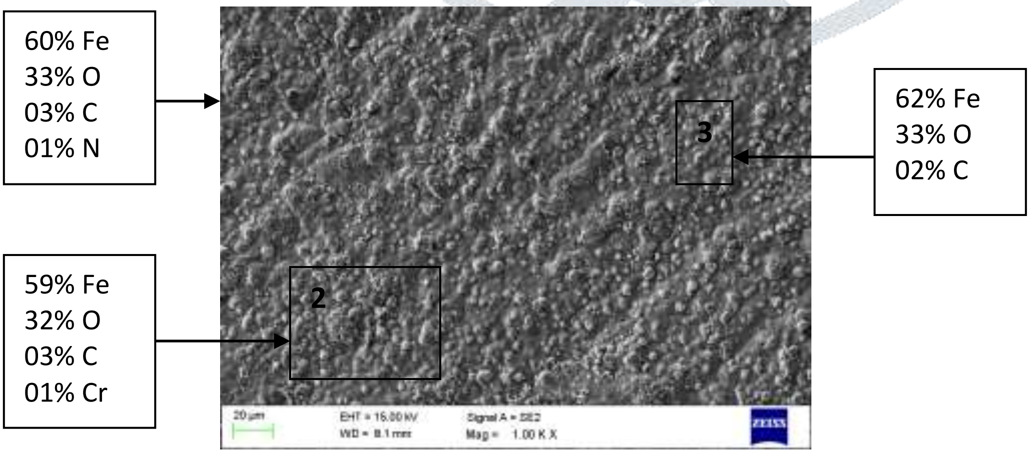


Figure 4- The SEM morphology and EDS analysis of top thermal coated surface layer

The FE-SEM-EDS micrographs are shown in Fig. 4. The nitride layer depicts the morphology consisting mainly spongy nodules homogeneously dispersed in the gray matrix. The EDS analysis indicates that the nodules are rich in O. N, Cr and C are found in minor percentages.

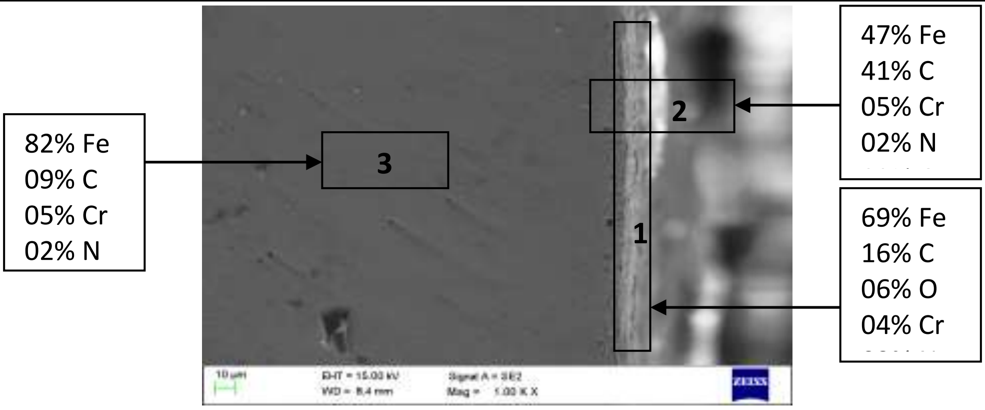


Figure 5- The SEM morphology and EDS analysis of cross section

3.3 Microhardness Measurement

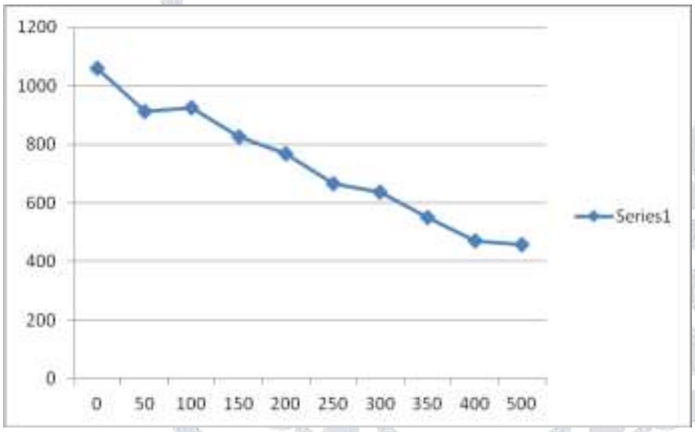


Figure 6- Microhardness profile of Thermal coated layer along the cross-section on AISI H13 steel at 500°C with 80:20- POWDER THERMAL SPRAY COATING

Figure 6 shows the microhardness profile of Thermal coated layer along the depth. The microhardness of the nitrided H13 sample was measured along the depth of the nitrided layer along the cross-section. Microhardness value1045 HV was achieved in the outermost layer. Thereafter, the value goes on decreasing towards the inner core of the sample. Therefore, from this case-depth analysis, it is clear that the nitride layer is developed with increased properties. The decreasing value of microhardness from outermost layer to the core is due to the reduction in the percentage of nitrogen from surface to core during the diffusion process. The thickness of the case depth was measured as 375 microns. The core hardness was measured as a maximum value of 429 HV.

Microhardness of the thermal coated sample was measured by SHV-1000, Digital Micro Vikers Hardness Tester, Made by Chennai Metco Pvt. Ltd., Chennai, India. 2.942N load was applied on the square pyramidal diamond indenter for penetration and the hardness values were based on the relation $Hv = 0.1891 \times \frac{F}{d^2}$ (Where F is load in N and d is the mean of the indentation diagonal length in mm). Each reported value of the microhardness is a mean of three observations. These microhardness values are plotted as a function of distance from the nitrided/substrate interface.

3.4 X-Ray Diffraction (XRD) Analysis

The Thermal coated specimens were subjected to XRD analysis to identify various phases formed on their surfaces. Diffraction patterns were obtained by Bruker AXS D-8 Advance Diffractometer (Germany) with CuK_α radiation and nickel filter at 20 mA under a voltage of 35 kV. The analysis was carried out at Institute Instrumentation Centre (IIC), Indian Institute of Technology Roorkee (IITR), Roorkee (India). The specimens were scanned with a scanning speed of 1 kcps in 2θ range of 20 to 120° and the intensities were recorded at a chart speed of 1 cm/min with 2°/min as Goniometer speed. Assuming height of the most prominent peak as 100%, the relative intensities were calculated for all the peaks. The diffractometer interfaced with Bruker DIFFRAC^{plus} X-Ray diffraction software provides‘d’ values directly on the diffraction pattern. These‘d’ values were then used for identification of various phases with the help of inorganic ASTM X-Ray diffraction data cards.

Figure 6 shows XRD profiles of the nitriding samples with 24h and 500˚C. It can be seen from Figure 5that the nitrided layer consist of Fe3N (ε), Fe4N (γ′), phases in all samples which were thermal coated at 24h. The amount of Fe3N phase increases in the depth direction from the surface. However, at the depth greater than 125 μm, the volume fraction of Fe3N decreases in the same manner as Fe4N phase.

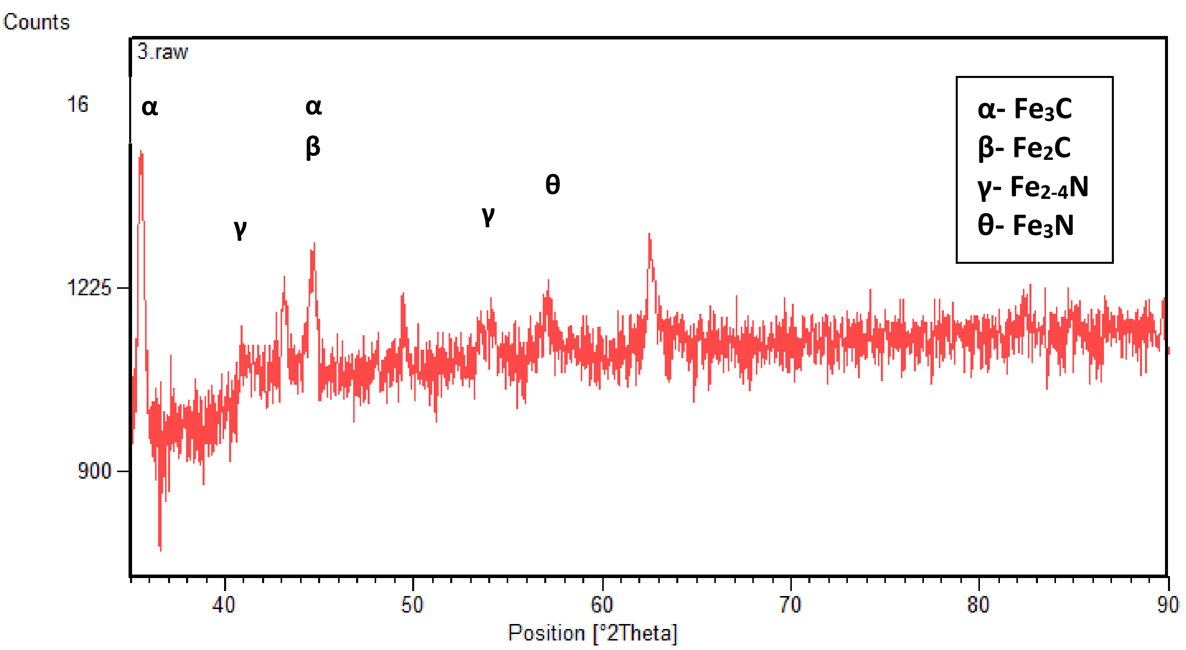


Figure 7. X-ray diffraction pattern for thermal coated H13 tool steel at 500˚C for 24h.

3.5 Surface Roughness Measurement

The surface roughness of the substrate material when measured with standard surface roughness tester was having average surface roughness value as 0.96µm. After the process of thermal spray coating average roughness was again measured with standard surface roughness tester and it was observed that after thermal spray coating process the average surface roughness value was 0.25µm, so the thermal spray coating process improves the surface roughness of material.

The aim of this work was to improve the wear properties of the AISI-H13 hot-work tool steel, so that the life enhancement of hot working tool steels could be achieved [11. For 24 hours of plasma-nitriding treatment hardness versus nitriding time data given in Figure 6. This suggests that the life of hot working tool steel is controlled by their surface hardness. This is not surprising, since under repeated high impact loads and varying temperatures, which lead to mechanical and thermal fatigue, wear is controlled by the surface hardness of the materials [12]. Figure 4 shows the SEM image of the material ion-nitrided for 24 hours).The approximate case depth which was obtained from distance from the edge of the surface observed as 125µm.The hardness versus distance from surface data, Figure 6, can also be measured. The surface roughness of the thermal coated material was improved after the thermal coated process; therefore, it is very much clear that nitriding helps to improve the surface properties of the material [13].

4. Conclusions

- From the results it can be concluded that thermal spray coating of AISI H11 steel improved the surface properties of material.
- The hardness of the as received material was about 260 HV and after the heat treatment process it was about 540 HV.
- Surface hardness of material reached to a maximum value of about 1089 HV0.1 after a time period of 24 hrs and at a temperature of 500˚C.
- Thermal spray coating results into formation of diffusion layer only without the formation of white (compound) layer.

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