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Experimental investigation of tribological properties of laser textured Tungsten doped Diamond like Carbon coating under dry sliding conditions at various loads.

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Abstract:

Laser micro texturing technique has shown its potential in reducing friction and wear at various mechanical interfaces such as automotive and cutting tools etc.. Automotive parts are coated with Diamond-like Carbon (DLC) coatings to enhance their performance. Due to stringent condition at the automotive contacts and demand for performance enhancement, increase in performance of DLC coatings is required. In this study laser micro texturing is being combined with tungsten doped DLC coating. In order to analyze the benefits of laser micro texturing on tungsten doped DLC coating. Tribological testing was conducted on a reciprocating test rig at various loading conditions. The results indicated that laser textured tungsten doped DLC coating showed the lower coefficient of friction compared to un-textured tungsten doped DLC coating at a load of 15 N, 25 N and 35 N. Higher graphitization was observed in the case of un-textured coating at 35 N load.

Keywords:

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3 Laser surface texturing, Tungsten doped DLC, Friction, Wear, Dry sliding
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6 **1. Introduction:**

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9 Reduction in friction to enhance automotive fuel efficiency is an important research topic
10 for several decades. Several techniques have been devised over the years such as surface
11 modifications and lubricant oil modifications. Laser micro texturing is a relatively new technique
12 for reducing friction at automotive components. Surface modification in the form of asperities
13 was introduced in 1966 by Hamilton et al. [1, 2]. They found that a seals load carrying ability
14 can be improved with micro asperities[1, 2]. Since then several analytical and experimental
15 studies have been conducted to analyze tribological effects due to change in shape of textures,
16 size of textures, sliding direction, placement angle, etc.. Surface modification has been useful for
17 reducing friction and wear at various mechanical interfaces. Various types of textures have been
18 created such as micro dimples[3] and micro grooves[4].
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32 Various applications of micro surface texturing are automotive[2, 5-15], cutting tool[16-
33 20], mechanical seals[21, 22], sports equipment [23] and biomedical applications [24, 25]. The
34 basic mechanisms of micro texturing are as follows: they behave as a micro hydrodynamic
35 bearing, they act as wear debris traps, they supply lubricant at the contact [26]. Micro textures
36 can be created by lasers[27], Vibro-mechanical texturing[28], Micro EDM[29], chemical
37 etching[30], micro grinding[31] etc..
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47 Various coatings have been used in automotive engines to lower wear/tear and reduce
48 friction; thus increasing the life of a component and reduce fuel consumption. Among them are
49 carbide coatings such as CrN, Ti-TiN, ZrN, CrAlNi [32] and Diamond-like Carbon coatings such
50 as a-C:H, ta-C, and a-C:H:W. DLC coatings are famous because they possess excellent
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3 tribological and mechanical properties in rolling sliding contacts [33] [34]. Various components
4 in IC engines such as piston rings, bearings, valves, and tappets are coated with DLC [35].
5 Applications of DLC coatings include automotive[35-37], cutting tools[38, 39] and medical
6 applications[40].
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13 To further enhance the service life of components, researchers have doped DLC coatings
14 with Hydrogen, various metals, carbides and nitrides [41]. W-DLC is one such coating in which
15 DLC coating is doped with tungsten. W-DLC coatings show high hardness, adhesion to the
16 substrate, low friction and wear[42]. Tribological properties have been evaluated for gears by
17 Joachim et al. [43]. They showed that W-DLC coated gears have higher macro pitting and load-
18 carrying capacity[44].
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27 A combination of laser micro texturing and DLC coatings has recently been found to
28 provide higher wear resistance than un-textured DLC coatings [8, 9, 45, 46]. Researchers have
29 combined amorphous hydrogenated carbon coating (a-C:H) [45], amorphous carbon (a-C) [47],
30 Silicon doped Diamond-like Carbon coating (Si-DLC) [46] and tetrahedral amorphous carbon
31 (ta-C) [8] with laser texturing. To date, the effects of laser texturing on W-DLC coatings have
32 not been studied. W-DLC is being used to coat automotive components, it is important to
33 analyze whether micro texturing plays a positive role in the overall performance enhancement or
34 not. The purpose of this article is to analyze whether micro texturing is beneficial in enhancing
35 the tribological properties of W-DLC coatings or not. Dry sliding was conducted so that the
36 positive or negative effects of micro-textures can be observed without the addition of complex
37 mechanisms of automotive lubricant or the additives that are present in them.
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53 **2. Methodology:**

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2.1 Sample Preparation:

The configuration used for Tribological testing was a ball on flat. The ball was made of AISI52100 steel and the plate was coated with W-DLC. The ball material was selected as it is used to make automotive parts. The properties are mentioned in Table 1. The substrate was made of hardened steel, the substrate was cut from a bulk material according to test rig specimen size (thickness, length, and width of 4-5 mm, 15 mm and 15 mm, respectively). After cutting, the samples were hardened to achieve a hardness of 58-62 HRC. For the ease of cutting, samples were hardened after cutting. To reduce the surface roughness of substrate various grit size sandpapers (100 to 3000) were used. After using sandpapers, polishing was conducted using a diamond suspension (size- 6 μm to 1 μm). After polishing, laser micro surface texturing was conducted. Dimples were created using Picosecond laser. Laser power, pulse duration, beam position speed and wavelength were 10 W, 10.3 ps, 1000 mm/s and 1640 nm, respectively. Ultrafast lasers such as (picoseconds and femtosecond) are mostly used for micro texturing[48]. They can be used to make well controlled micro and nano textures [48]. The lower cost of picosecond lasers make them suitable for several commercial application[49]. The scope of this paper is limited to wear behavior of textured coatings rather than effects of laser on material properties. The depth and diameter of micro-texture were around 6-8 μm and $\sim 50 \mu\text{m}$, respectively. These texture parameters have been found previously to enhance tribological properties[9]. After the micro texturing, the bulges were removed by light polishing with diamond suspension (0.5 μm). According to Amanov et al.[46], bulges are formed around the dimples, additionally, areas nearby to dimples experience melting, thermal and rapid re-solidification. After laser texturing W-DLC was deposited. The deposition was conducted by Oerlikon Balzers Ltd. The properties can be found in the paper [50].

2.2 Tribological testing and wear track characterization:

Tribological testing was conducted on a reciprocating test rig at loads, frequency and temperature of 15, 25 and 35 N, 10 Hz and 28-30 °C, respectively. Reciprocating test rig (DUCOM TR 282) was used for tribo testing. The duration of test was 1 hr. Each test was repeated three times to ensure repeatability. The wear of counter surface balls was measured using an optical microscope. After tribological testing, the samples were cleaned with n-heptane and placed in desiccators for further investigation. To analyze the surface morphology and elemental composition, scanning electron microscope (SEM) (Hitachi Model No. TM3030) and Energy dispersive spectroscopy (Hysitron, Inc., Model No. TI750 UBI™) was used.

3.1 Results and Discussion:

Figure 1 shows that in the start, the increase in friction is abrupt, however after 500 seconds; the increase in friction became smoother. This can be related to unsteady friction behavior in the running-in period. Similar behavior has been observed by Kummel et al. [51]. As this test has been conducted without lubricants, the steady-state average friction coefficient is higher.

At a load of 15 N, in the case of W-15t (textured coated sample at 15 N load) sample, the average friction coefficient is 0.147 compared to W-15 (coated sample at 15 N load) sample which showed average friction coefficient of 0.165. This shows that in dry sliding condition, micro-textures can help in the reduction of friction coefficient. Previously, reduction of COF had been observed in the case of lubricated sliding condition for DLC coated samples [46, 47].

At a load of 25 N, in the case of the W-25t sample, the COF value is 0.1495. Whereas in the case of the W-25 sample, the COF value is 0.1665. At a load of 35 N, in the case of the W-

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3 35t sample, the COF value is 0.149. Whereas in the case of the W-35 sample; the COF is 0.166.
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5 The COF results at 25 and 35 N load shows that micro-textured samples show lower COF
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7 compared to un-textured cases in dry sliding conditions. Another indication that can be obtained
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9 from Figures 1a, 1b, 1c is that in the case of textured samples, friction trend after 2500 s is
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11 smoother compared to un-textured cases. This behavior can be attributed to the capture of wear
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13 debris by micro-textures, which reduced the friction instability[45]. This has been observed by
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15 He et al. in the case of H-DLC coating[45]. Takeno et al. [52] observed that due to the rolling
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17 effect of tungsten oxide debris the friction coefficient is unstable. They proposed the mechanism
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19 that due to the reaction of tungsten and oxygen, tungsten oxide debris is formed. The debris
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21 comes in and out of the sliding track, which causes unstable friction [52]. Mustafov et al. [35]
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23 also observed instable friction of W-DLC in the case of dry tests of untextured samples. The
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25 authors deduced that the formation of the transfer layer is responsible for friction variation in dry
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27 sliding conditions [35]. Figure 2 shows the average COF of various samples. It can be seen that
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29 textured samples showed lower average COF than un-textured samples.
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36 **3.2 Wear track characterization:**

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39 The wear scar diameter of the counter surface ball is shown in Figure 3. The trend is
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41 similar to the trend of COF. At a load of 15 N, W-15t counter ball showed wear scar diameter of
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43 321.7 μm . Whereas W-15 counter ball showed wear scar of 618.7 μm . At a load of 25 N, the
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45 counter ball which rubbed against W-25t and W-25 showed wear scar diameters of 360.53 μm
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47 and 594.3 μm , respectively. At a load of 35 N, the counter ball which rubbed against W-35t and
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49 W35 showed wear scar diameter of 403.9 and 703.9 μm . The results show that with the increase
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51 in load, counter ball wear also increases. Additionally, the counter ball which rubbed against
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3 textured W-DLC samples showed lower wear scar compared to un-textured W-DLC samples.
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5 Figure 4 shows optical microscope images of wear scar of counter balls.
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9 Figure 5 shows the SEM images of various micro-textured and un-textured W-DLC coated
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11 samples at various loads. The W-DLC generally showed a nodular structure as observed by
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13 other authors [50, 53]. Figure 5 (h) shows the W-DLC before testing. Table 2 shows the EDX of
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15 samples. The SEM result of W-15 shows small localized scratches; additionally, the nodules on
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17 the surface were also removed. The surface of W-15t shows wear induced smoothening
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19 (polishing wear). The wear induced smoothening has also been observed by Ronkainen et al.
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21 [53]. At the wear track of W-15t, the dimples were full of coating material; one example is
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23 shown in Figure 5 b. This is one of the reasons that abrasive wear marks were not observed on
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25 the surface of W-15t, as they were trapped by the dimples. He et al. [45] in the case of textured
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27 H-DLC observed similar behavior, where micro dimples stored the wear debris to reduce wear.
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33 Sample W-25 shows a thick layer of coating materials on the surface of wear track at
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35 some points, as shown in the figure. 5 c. In the case of W-25t (figure 5 d), the coating material
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37 can be seen to be trapped by the dimple. The wear track of W-35 (figure 5 e and f) shows a thick
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39 coating material layer at some points and wear induced smoothening at other points. SEM image
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41 of W-35t (figure 5g) showed that dimple trapped wear debris. SEM images of all the samples
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43 show that the delamination of coating didn't occur in the case of textured or un-textured W-DLC
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45 coating. This indicates that coating adhesion to the substrate was excellent.
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49
50 Table 3 shows the EDX results of tested samples. At a load of 15 N, W-15 and W-15t
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52 shows a reduction of C content and increase in W and O content compared to as-deposited
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54 sample. At a load of 25 N, W-25 showed higher O content compared to the W-25t, this can be
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56 due to the presence of oxide rich films at the wear track. At a load of 35 N, sample W-35 showed
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3 higher O content similar to the W-25 sample compared to W-35t. Takeno et al. [52] suggested
4 the formation of tungsten oxide debris. Formation of beneficial tribolayer has also been
5 suggested by Ronkainen et al. [54].
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10 Raman spectroscopy was conducted to analyze the structural changes of W-DLC coating
11 before and after testing. The graphitization of W-DLC coating has been observed by other
12 authors [50]. Before wear testing, the Id/Ig ratio was 1.49. After wear testing, W-35 and W-35t
13 showed the Id/Ig ratio of 4.39 and 2.92. W-35 showed higher Id/Ig compared to W-35t. The
14 results indicate that higher graphitic transformation in the case of un-textured W-DLC compared
15 to textured W-DLC at 35 N load. Graphitization can be induced due to micro-wear debris present
16 at the wear track. Due to these wear particles, the load per unit area increases at the contact point,
17 which lowers the temperature at which graphitization occurs [47]. In the case of the textured
18 sample, figure 5g shows that the dimple is full of wear debris. The lower wear induced
19 graphitization in the case of textured samples can be due to wear debris entrapment by the
20 dimples. The results indicate that the depth of dimples maybe increased in the case of dry sliding
21 conditions, as 6-8 μm may be not enough to entrap all the wear debris during operation. This
22 may further reduce the wear induced graphitization in the case of textured samples.
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41 **4. Conclusions:**

42 The effect of laser texturing was investigated on tungsten doped DLC coating under dry sliding
43 conditions under various contact loads.
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- 48 1. At a load of 15 N, sample W-15t showed lower COF compared to sample W-15. This can
49 be attributed to the entrapment of micro-wear particles by the micro-textures.
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2. At a load of 25 N, COF increased in the case of both W-25t and W-25 compared to W-15t and W-15. However, even at higher load W-25t showed lower wear compared to W-25.
3. COF increase didn't occur at a load of 35 N compared to 25 N. This can be because of graphitization phenomena. Even at high load, micro-textured W-DLC showed lower COF than un-textured W-DLC.
4. Counter ball wear scar reduced in the case of micro-textured W-DLC coating compared to counter balls which rubbed against un-textured samples.
5. Laser micro texturing can be used to enhance the tribological performance of W-DLC coated automotive parts.

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Figure No. 1. Coefficient of friction of textured and un-textured W-DLC coating at various loads

(a) 15 N (b) 25 N (c) 35 N

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Figure No. 3. Wear scar diameter of the counter surface at various loads.

Figure No. 4. Optical microscope images of Ball wear scar at various loads. (a)W-15(b) W-15t

(c)W-25 (d) W-25t (e)W-35 (f) W-35t

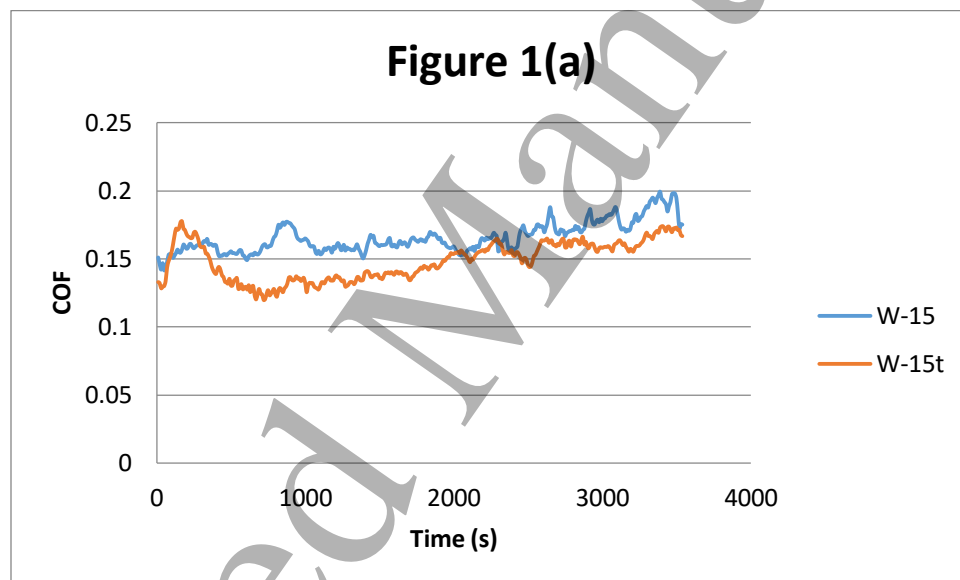
Figure No. 5 Scanning electron Microscope images of wear track of textured and un-textured W-DLC at various loads. (a)W-15 (b) W-15t (c)W-25 (d) W-25t (e)W-35 (f) W-35 (g) W-35t (h) as-deposited

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Table No. 2. Wear track EDX analysis of various textured and un-textured W-DLC coatings

Table No. 3. Raman Spectroscopy of textured and un-textured W-DLC samples at a load of 35 N



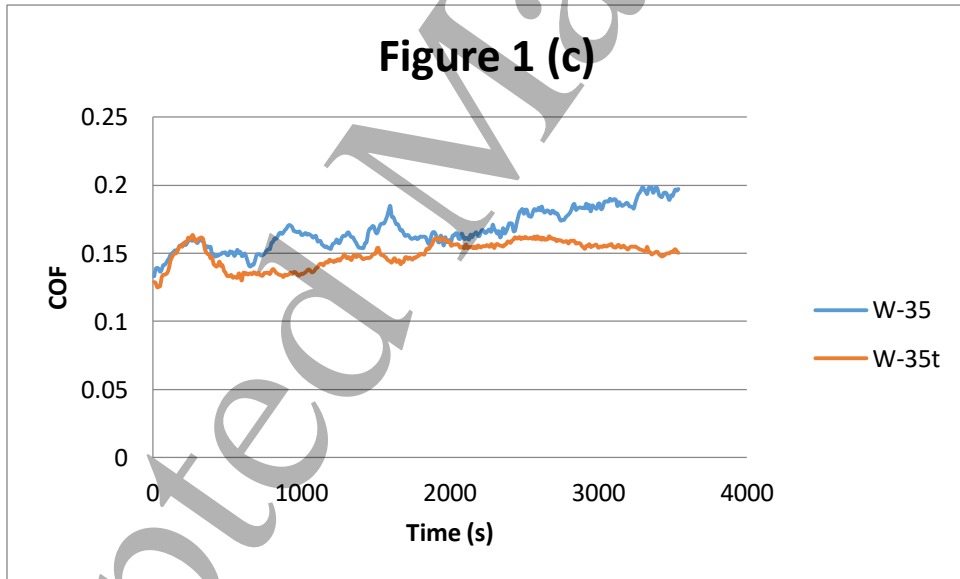
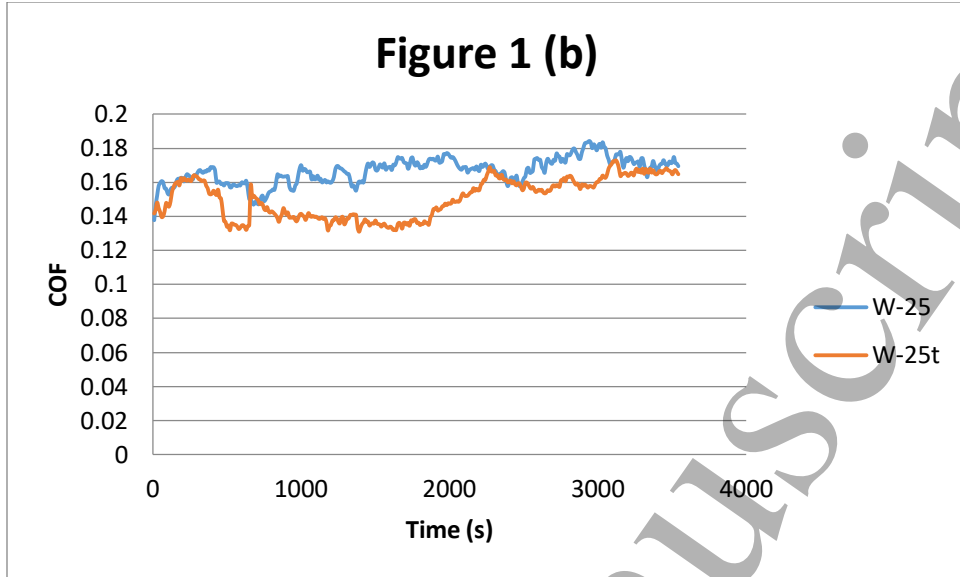


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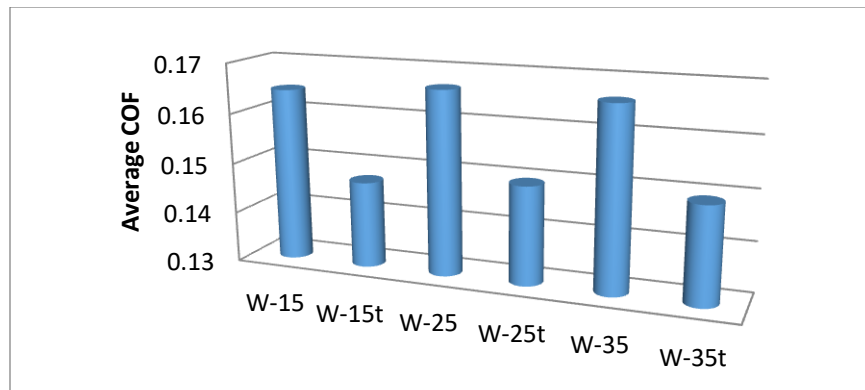


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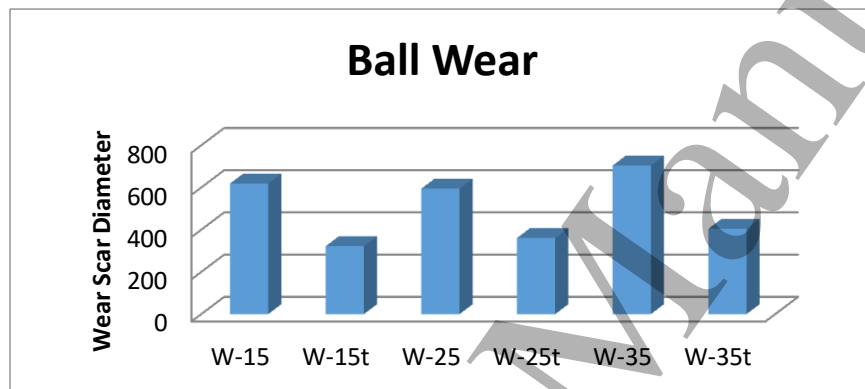
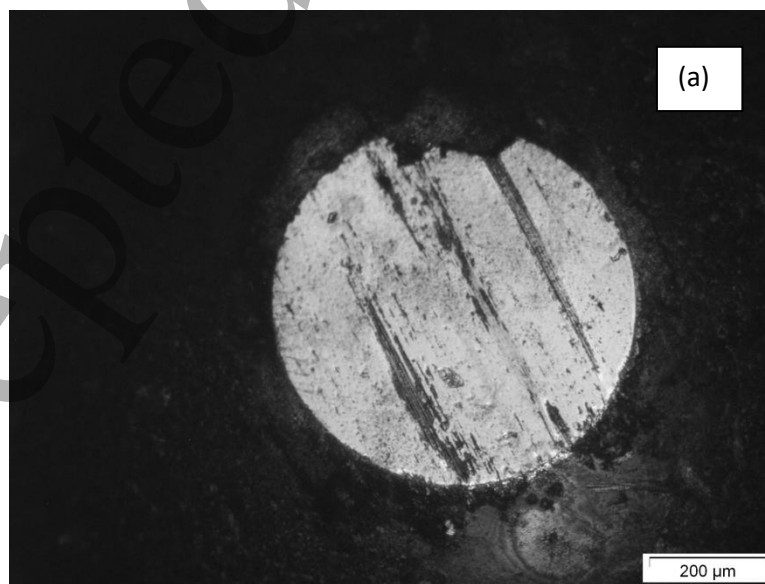
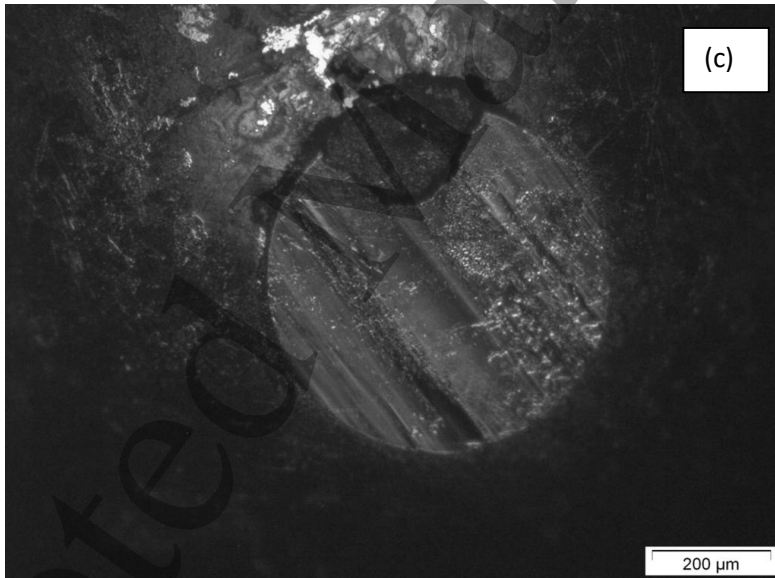
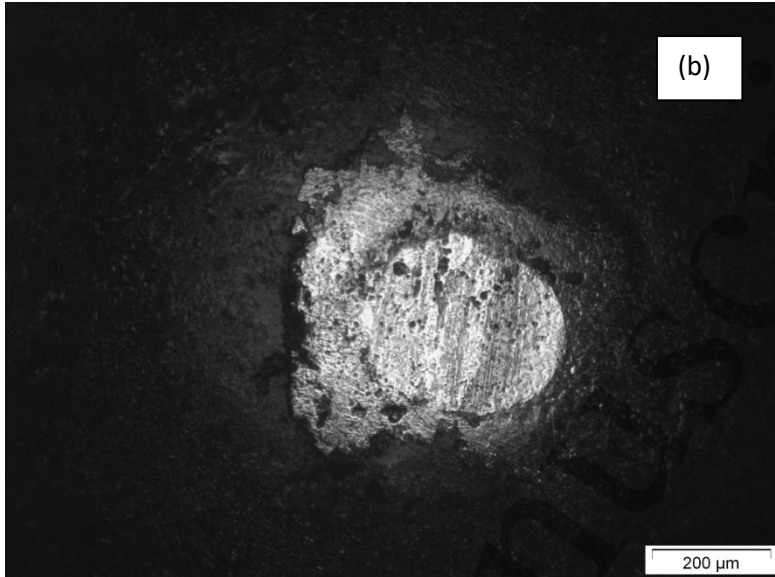
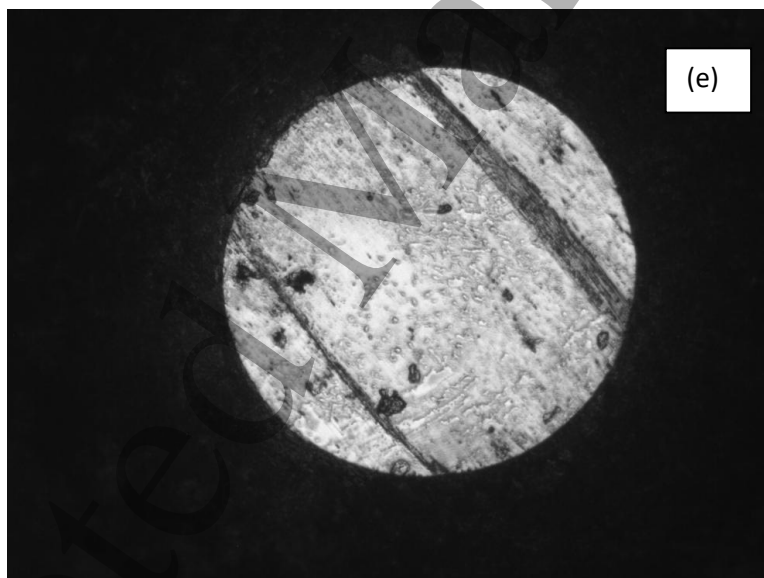
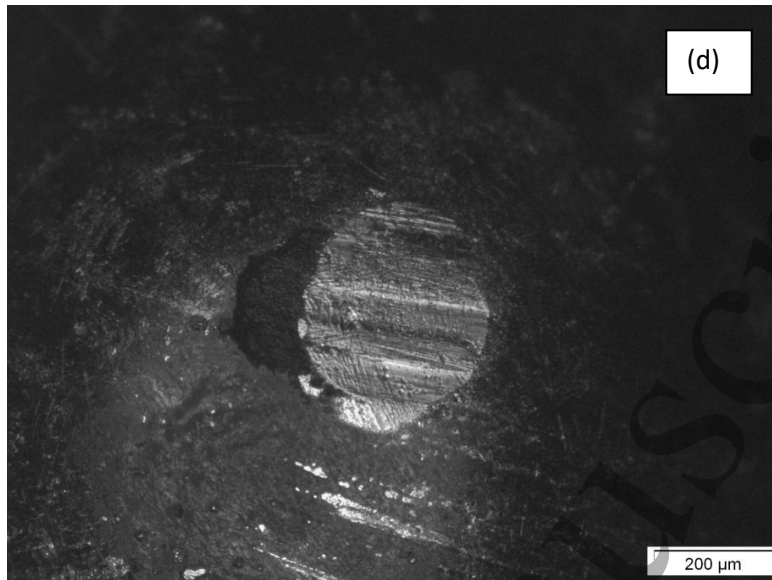


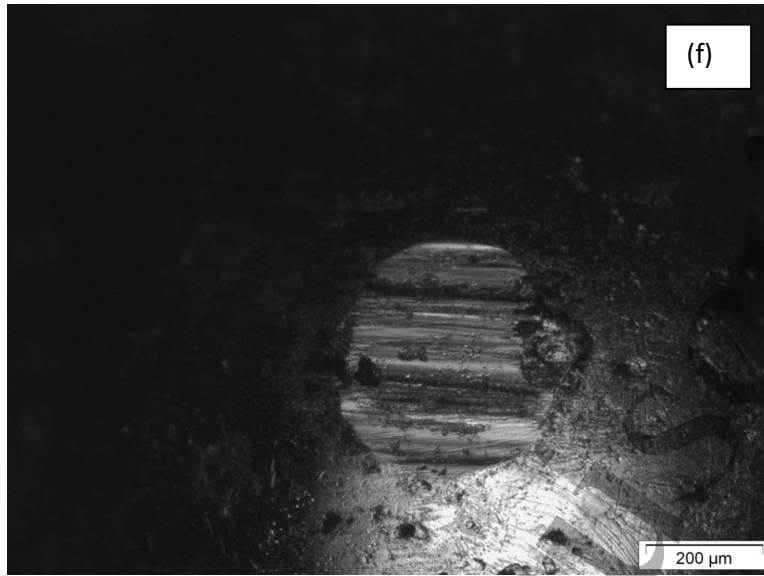
Figure No. 3.



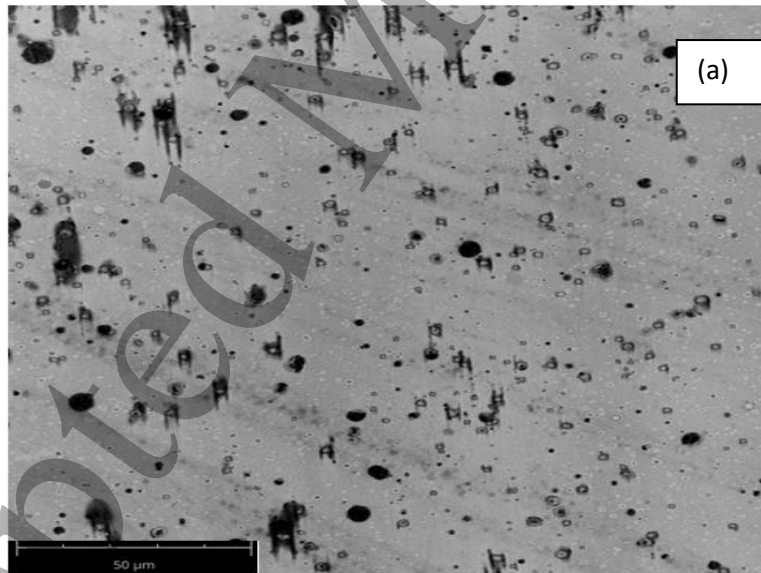
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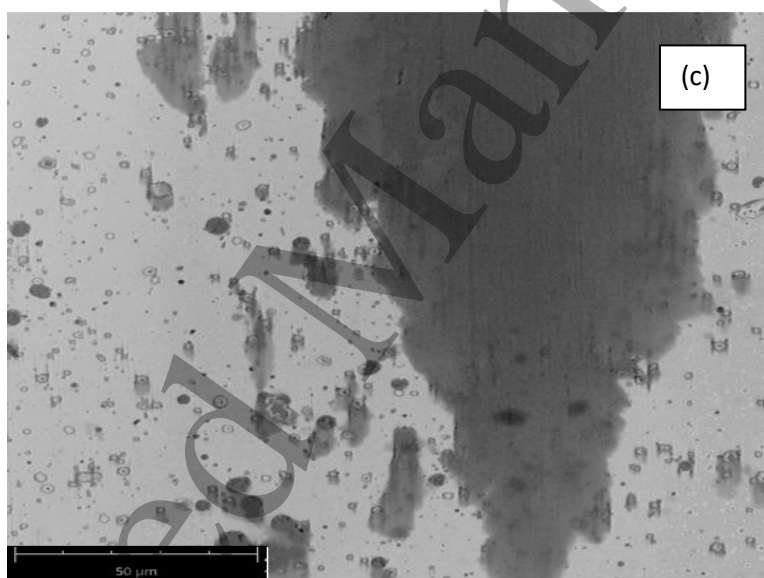
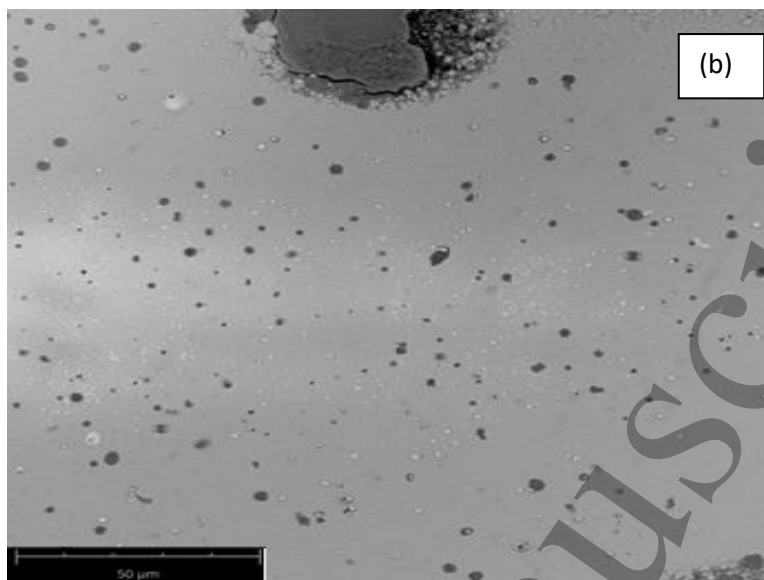




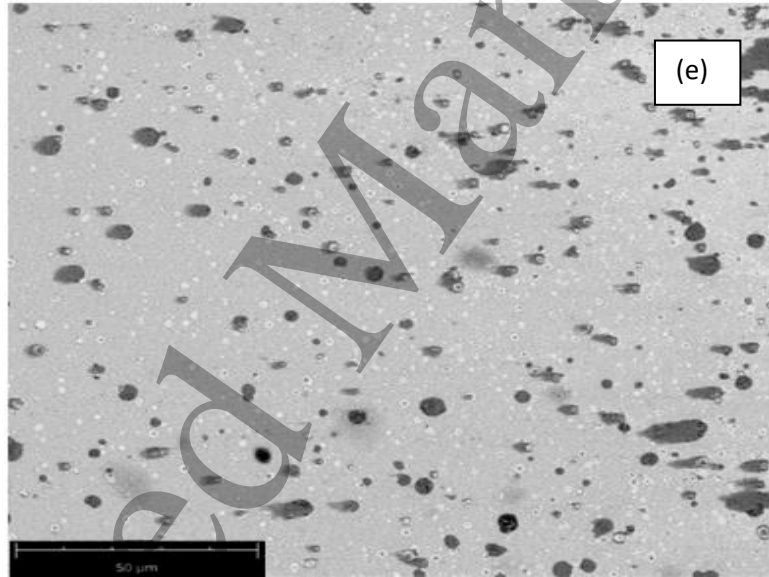
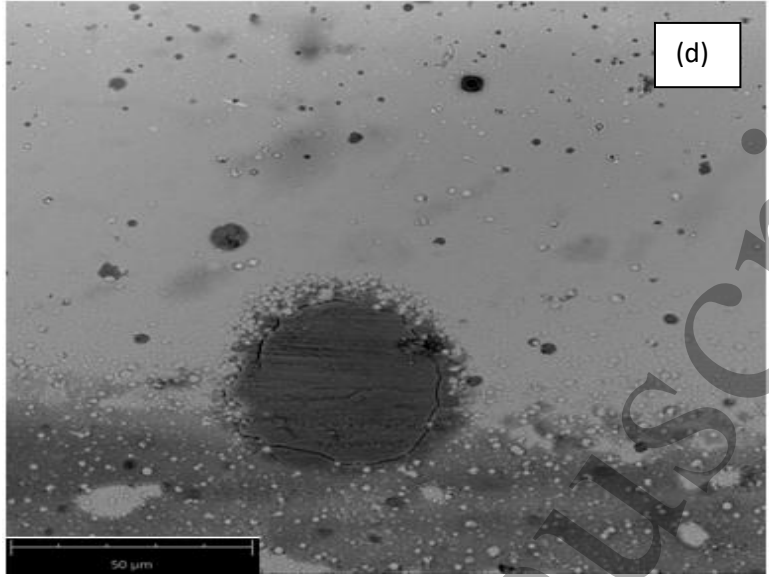


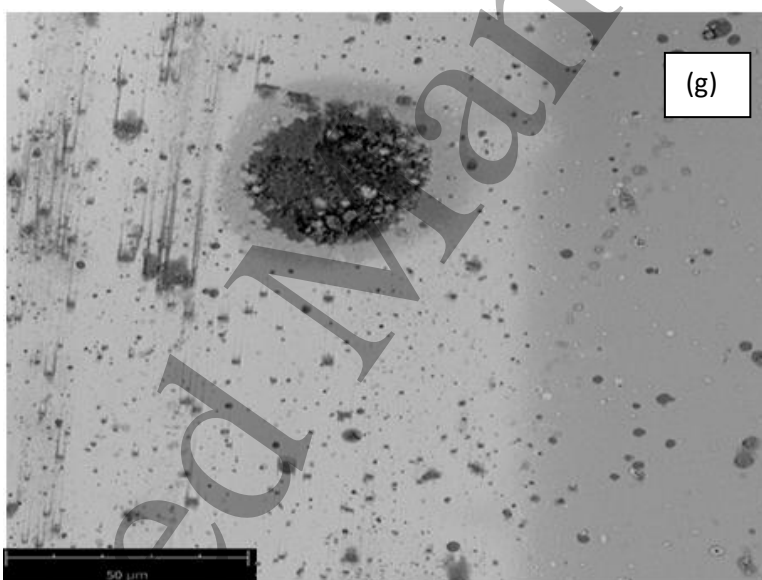
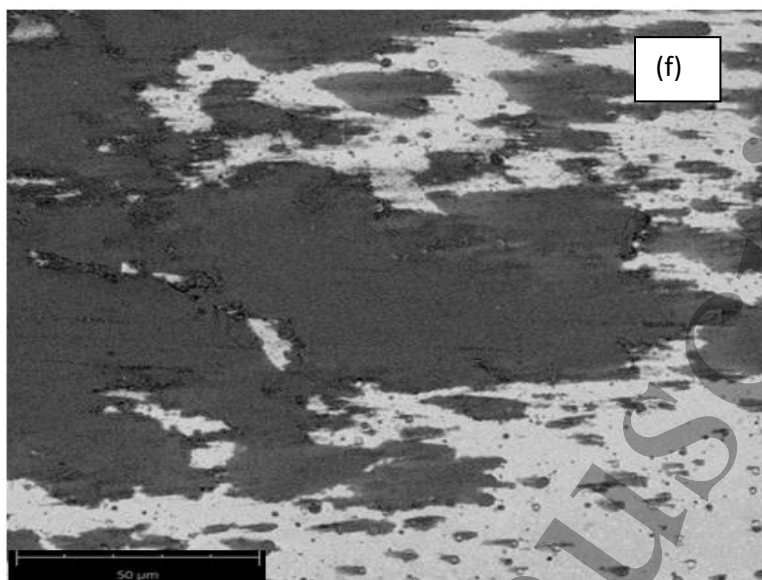
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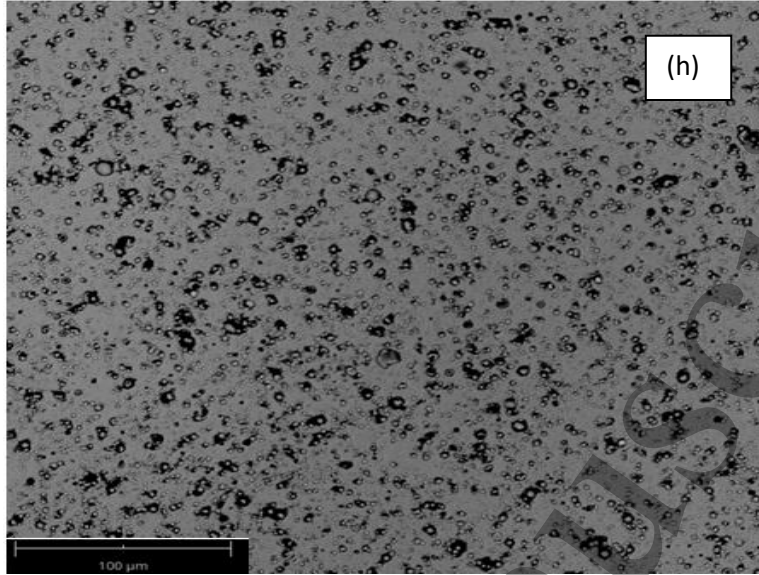




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26 **Figure No. 5.**

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31 **Table No. 1**

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Physical Properties	Steel Ball	DLC coating
Material	AISI-52100	W-DLC
Hardness	~62 HRC	12–15 GPa
Composition	Fe 96.5 %, C 0.98 %, Mn 0.250 %, Si 0.150%, Cr 1.30%, P ≤ 0.0250, S ≤ 0.0250	Hydrogen 15 % (atomic weight)
Roughness (average)	40 nm	-

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Thickness	-	2-3 μm
Interlayer	-	CrN

Table No. 2

Sample	Carbon	Tungsten	Oxygen	Nickle	Chromium	Iron
W	74.56	18.53	3.9	-	1.4	1.61
W-15	53.95	32.43	8.18	2.40	1.84	1.20
W-15t	49.95	36.34	9.15	2.42	1.32	0.82
W-25	20.83	24.02	43.6	2.19	2.64	6.72
W-25t	38.74	32.70	21.05	4.15	-	3.36
W-35	14.93	18.20	50.07	1.02	2.71	13.07
W-35t	33.45	43.63	12.84	2.63	5.46	1.99

Table No. 3

Sample Name	Id/Ig ratio
W-DLC (Before Test)	1.49
W-DLC (After Test)	4.39
W-DLC Textured (After Test)	2.92

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