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Ultra-low friction W-S-N solid lubricant coating

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Abstract

W-S-N films were deposited by reactive magnetron sputtering from WS2 target in Ar/N2 atmosphere. Besides the standard evaluation of composition, structure, morphology, hardness and cohesion/adhesion, the core objective of this paper was to analyze coating tribological behavior. The chemical composition was 34 at.% N, 12 at.% O, 29 at.% W and 25 at.% S and the as-deposited films were completely amorphous. The film thickness was 2.3 μm including the approximately 300 nm thick adhesion improving titanium interlayer. The friction coefficient was lower than 0.003 when sliding in dry nitrogen. The coating showed remarkable wear resistance surviving more than 2 million laps on pin-on-dics. The excellent friction properties were attributed to the formation of a thin tungsten disulfide tribofilm on the top of the wear track of the coating and on the counterpart surface. Moreover, the coating showed ability to replenish damaged areas with solid lubricant. We demonstrated that a

structural transformation of the coating from an amorphous-like to a gradient quasi-ordered structure and an ordered transfer layer formation improved mechanical properties and radically decreased friction and wear.

Keywords: Tungsten disulfide; ultra-low friction; self-lubricant; coating; magnetron sputtering

1. Introduction

Molybdenum disulfide and later tungsten disulfide have been used for decades as oil additives or solid lubricants; typically in the form of thin films. It has been demonstrated that the low-friction behaviour of pure transition metal dichalcogenides (TMDs) is associated with a sliding induced formation of a phase with an optimum orientation of the (002) planes parallel to the movement [1]. In ultra-high vacuum the frictional force has been reported to virtually disappear, being lower than the sensitivity of the measuring device [2]. Further, the presence of contaminants has been shown to be detrimental for the low-friction properties. Formation of the (002) crystallographic direction in the sliding interface has been found in the wear tracks of very different alloyed TMD materials, such as amorphous-like (Pb-Mo-S [3]), nanostructured (Mo-Se-C [4]), and nanocomposite (DLC/WS_2/WC [5]) coatings. In these materials, the mechanism for the formation of the (002) orientation is still not known.

Due to a high tendency to transfer, TMD layers quickly form on the counterparts during sliding. These transfer layers have also proven to have a (002) orientation, which facilitate low-friction sliding by: i) minimizing the force required for slip and ii) making sure that the reactive prismatic edges in the planes are not exposed to the ambient atmosphere. Moreover, this orientation exhibits relatively high hardness [6], which is related to higher wear resistance. The role of doping elements still has to be understood. It seems that they are

rapidly removed from the contact area and only contribute indirectly; improving the tribological properties by increasing the coating hardness and density [7].

Several mechanisms have been proposed to explain the reorientation of the originally randomly oriented phase, including detachment and adhesion of the wear particles [8,9] or re-orientation of the grains [10]. Since such macroscopic or structural transformations increase the shear stress, tribological films manufactured to give exclusive or predominant (002) phase orientation have been explored by depositing films directly in the (002) orientation using chemical (van der Waals rheotaxy [11]) or physical methods (pulsed magnetron sputtering [12]). Due to delamination at the film-substrate interface, tribological properties of such films are presently worse than expectations.

Recent studies have focused mainly on production of well-ordered nanotubes [13,14,15] or fullerene-like particles [16,17,18,19,20]. However, such materials are impractical for most industrial applications. Magnetron sputtering is one of the most versatile methods to produce dense, well-adhered and thin TMD-based films. Pure sputtered films are typically porous due to a columnar structure with a random orientation of the TMD phase. Their mechanical properties and resistance to environmental attacks are generally very low. In general, hardness, adhesion, and scratch resistance have been found to be improved by alloying. Co-sputtering of TMD films with metallic and non-metallic elements or compounds leads to different microstructures, such as solid solutions or nanocomposite [21] materials. Summary of recent state of the art in the area of TMD-C can be found in our recent review [22]. W-S-N coatings with different nitrogen content were synthetized by magnetron sputtering for the first time by Nossa et al. and their structure [23, 24], mechanical properties and tribology [25,26] were investigated. The wear performance of W-S-N films was often limited due to insufficient adhesion to the substrates; moreover, their friction in humid air was relatively high. Nevertheless, our recent study has indicated that W-S-N coating might exhibit

extremely low friction in dry air, since the friction was much below 0.01 at elevated temperatures [27].

Here we present tribological properties of W-S-N coating deposited by magnetron sputtering sliding in dry nitrogen.

2. Experimental details

W-S-N coatings were deposited on 100Cr6 (tribological tests) and M2 (scratch tests, nanoindentation) polished steel samples with hardness close to 8 GPa and 9 GPa, respectively. The depositions were carried out in a magnetron sputtering Hartec unit, equipped with two cathodes – titanium and tungsten disulfide. Prior to the depositions the substrates were sputter cleaned during 20 min by establishing the plasma close to the substrate. Immediately after, a Ti interlayer was deposited with an approximate thickness of 300 nm in order to improve adhesion. The W-S-N film was then reactive sputtered from a WS_2 target in Ar/N_2 atmosphere.

The chemical composition of the coating was determined with Electron Probe Microanalysis (EPMA), using a Cameca SX-50 system and verified with X-ray photoelectron spectroscopy (XPS), using an ESCA PHI QUANTUM 2000 with Mg Kα radiation. The latter was also used to investigate the chemical bonding. The surfaces were mildly polished with 200 V Ar^+ ions for 10 minutes before acquisition. The crystal structure was analyzed with X-ray diffraction (XRD) in a Philips X-Pert system using Co Kα radiation ($\lambda = 0.178897$ nm) in grazing mode (2°). To further analyze the microstructure, high resolution transmission electron microscopy (HR TEM) and selected area electron diffraction (SAED) was performed in a JEOL 3010 TEM.

The hardness (H) and Young's modulus (E) of the coatings were determined using a nanoindenter (Micro Materials NanoTest) with a diamond Berkovich tip under 10 mN applied

load. The adhesion/cohesion of the coatings to the M2 steel substrate was evaluated by scratch testing. The load was increased linearly from 0 N to 50 N, using a Rockwell C indenter with a 200 µm tip radius. The loading speed was 100 N/min and the scratch speed was 10 mm/min. The thickness of the coatings was measured using a mechanical profilometer in steps that were produced on the substrate.

Sliding tests were carried out in two pin-on-disc setups. Short tests were performed in humid air (relative humidity 10%) with a sliding speed of 0.1 $m.s^{-1}$ and a wear track radius of 7 mm. Long tests in dry nitrogen were carried out in a second setup with controlled environment, at a sliding speed of 0.1 $m.s^{-1}$ and a wear track of radius 2.5 mm. Steel balls (100Cr6) with a diameter of 6 mm were used as counterparts. The wear tracks on the coatings and wear scars on the balls were characterized with white light interferometry using a WYKO NT 1100, and scanning electron microscopy (SEM) in a Zeiss Leo 1550. The chemical composition and chemical bonding of the worn surfaces were investigated using Energy dispersive X-Ray spectroscopy (EDX), XPS, Raman spectroscopy, and Auger electron spectroscopy (AES). Raman spectroscopy was performed on a Horiba Yvon Xplora system using a DPSS laser with wavelength 532 nm. A low laser power of 0.2 mW was used to avoid film damage. AES analyses were performed with a PHI 660 scanning Auger microprobe with an acceleration voltage of 10 kV and a beam current of 200 nA. Depth profiles were obtained using 3.5 kV Ar^+ ion sputtering. Auger mapping was performed in the 2-point mode, i.e. the difference between the peak intensity and the background divided by the background, in order to reduce topographically induced intensity variations.

Selected areas in the wear tracks on the coatings and transfer layers on the balls were investigated with HR TEM in a FEI Tecnai F30 ST, equipped with a Gatan post column energy filter. All TEM images and energy filtered elemental maps (EFTEM) were acquired

using 300kV acceleration voltage. The samples for HR TEM were prepared using focused ion beam (FIB) in a FEI Strata DB235 with in-situ lift out of the sample.

3. Results

3.1 Characterization of the W-S-N coating

The chemical composition as determined using EPMA was 34 at.% N, 12 at.% O, 29 at.% W and 25 at.% S. The film thickness was 2.3 µm including the approximately 300 nm thick adhesion improving titanium interlayer. XPS analysis showed the presence of W–N, W–S and W–O bonds, with the latter matching tungsten trioxide. The high oxygen content probably originates from contamination of the target. Based on the observed chemical bonding and composition, a high S/W ratio is expected, which is considered optimal for the sliding [8]. The films appear to be amorphous both when studied with XRD (not shown), and with HRTEM and SAED, see Fig. 1. The hardness of the films was 7.7 GPa, which is about one order of magnitude higher than typical values for pure sputtered W-S films [23].

3.2 Sliding tests

The coatings were tested in humid air with a relative humidity (RH) of 10%, at two loads with test duration of 5000 laps. At a load of 5 N, the coefficient of friction (µ) stabilised at about 0.008, see Fig. 2. When the load was increased to 55.8 N, µ dropped below the resolution of the equipment (0.003). Tests in a second pin-on-disc setup in dry nitrogen (RH < 1%) with loads of 5 and 20 N again showed µ values below 0.003 (Fig. 2). SEM images (Fig. 3) and 3D optical profiles of the wear track showed extremely smooth surfaces, with a roughness Ra < 1 nm in the sliding direction and a maximum peak-to-valley height Rt < 40 nm. The coating showed remarkable wear resistance, not being worn out until after 2.1 million laps with a load

of 20 N. This corresponds to the removal of one WS_2 layer every 500 laps. The friction had increased to 0.015 before the coating failed. Balls examined after the tests showed round wear scars always completely covered by an adhered transfer film. Steady state wear with a stabilised friction was reached after approximately 100 laps, indicating that the steel ball was worn only at an initial stage. When the transfer film had formed, the ball was protected from further wear and the friction remained very low.

3.3 Analysis of the worn surfaces

Direct observation of the structure of the worn surfaces was performed using HRTEM. The TEM cross section samples were taken (using FIB) from the centre of the wear track on the coating (Fig. 3) and from the centre of the contact spot on the ball. The samples were cut perpendicular to the sliding direction.

The outermost surface of the tribofilm formed in the wear track consisted of a thin layer of WS_2 with (002) orientation. This layer covered the whole wear track, with a thickness varying from 2 to 10 WS_2 planes. Beneath this layer we found a distinct 'transition' layer consisting of a few WS_2 planes (predominantly (002) orientation), small nanograins identified as WO_3, and an amorphous or quasi-amorphous phase (Fig. 4). Even further down, the tribofilm displayed separated WS_2 platelets with a length not exceeding 5 nm. Most of these platelets were also oriented with the (002) basal planes parallel to surface. Obviously, sliding contact completely transformed the structure of the W-S-N coating close to the sliding interface.

WS_2 layers with their basal planes parallel to the sliding surface were observed also in the outermost parts of the transfer film on the ball (Fig 5a). As on the coating side, a mixed region dominated by amorphous tungsten oxide was found beneath the top layer (Fig 5b). The bottom layer, close to the steel surface was similar to the top layer, with an exclusively (002) oriented WS_2 phase (Fig 5c). This transfer film was fully dense without cracks or other

defects. It could be speculated that such dense transfer films are built up by attaching molecules or very small particles, not more than a few nanometre large. The extremely smooth wear track supports that transfer of material occurs on the molecular scale, since large wear particles would cause scratches and increase the friction.

Macroscopic tribological processes are inherently stochastic, so TEM observations from a very limited volume could lead to misleading conclusions. Therefore, further analyses with various depth and spatial resolutions have been carried out. Raman spectroscopy with a spot size less than 1 µm is often an ideal tool to examine worn surfaces. The as-deposited coating only exhibited a very small peak at ~326 cm^{-1} attributed to WO_3 bending vibrations and a large broad peak at 660-1100 cm^{-1} related to stretching vibrations of WO_3 (717 and 807 cm^{-1}) [28]. Raman spectra of the wear track and the transfer film showed peaks representing well-ordered tungsten disulfide (including second-order peak at ~535 cm^{-1}) [29], which are absent in the as-deposited spectrum (Fig. 6). However, the relative intensity of the WS_2 peaks decrease close to the border of the wear track. The relative intensity of the WO_3 peaks was significantly lower in the wear track than in the as-deposited film, showing the opposite tendency to that of WS_2. The top surface layer consisted exclusively of WS_2 and the thickness thus depends on the applied contact pressure, which has its maximum in the centre of the wear track. These results are in agreement with a frictional analysis of carbon-doped WS_2 films forming a similar WS_2 top layer [30].

AES was performed to identify the chemical composition at different depths of the transfer film on the ball run in dry N_2 for 100 000 laps with a load of 20 N load. Unfortunately, preferential sputtering of sulphur leads to changes in chemical composition and, thus, the results of AES (and XPS) should only be used qualitatively. Four etching times were selected to measure the chemical composition at depths of approximately 3, 20, 40, and 80 nm. The composition on the surface and at the 3 nm depth was remarkably similar in all spots analysed

(indicated by white boxes in SEM image of Fig. 7). The majority of the signal originated from tungsten and oxygen, followed by sulphur and nitrogen. However, a large iron signal was reached after different etching times in the investigated areas, suggesting that the thickness of the transfer film varies. The distribution of elements over a part of the transfer film is presented by the AES maps in Fig. 7. The relatively low amount of sulphur in the contact could be due to the presence of native oxides, indicating that the sputtering was not sufficient to remove the oxide from all parts of the contact. Iron seems to be present mainly in a few deeper scratches. XPS spectroscopy on a different ball also after a depth of approximately 3 nm (not shown), showed only W-O and W-S bonding. Nitrogen was hardly distinguishable from the noise, indicating that the amounts are very small.

3.4 Coating damage recovery during sliding of W-S-N coating

The ultra-low friction described above was not observed in all tests. In a few cases (2 from 10), in dry nitrogen, the friction curves were noisier with an average μ around 0.01. Both optical observation and SEM of the wear tracks showed large delaminated parts in the wear tracks showing higher friction, see Fig. 8.

In most of cases the delaminated part covered the full width of the wear track. EDX mapping of the damaged area showed mostly titanium with some oxygen and nitrogen; tungsten and sulphur did not appear to be present, see Fig. 9. The nitrogen originated from the top part of the titanium layer, since there is an overlap between the Ti deposition and the introduction of nitrogen flow during the coating process. However, this observation contradicts the friction results – the measured friction coefficient of 0.01 is far below 0.5-0.8 typical of steel to Ti/TiN contact [31]. The size of the delaminated areas rules out the possibility that the non-delaminated parts of the wear track would be carrying the load. Thus, there must be some mechanism keeping the friction low after local coating failure. It is also worth noting that the

parts of the wear track surrounding the delaminated areas were not more worn than wear tracks where no failure had occurred.

Detailed observation of delaminated areas in SEM (Fig. 8b) revealed two distinct features: i) thin layers adhering to the surface and ii) a surface resembling the titanium interlayer (hereinafter Ti surface), which represented the majority of the total investigated area. Raman spectroscopy clearly identified the adhered layers as tungsten disulphide. However, small WS_2-related peaks appeared together with WO_3-related peaks in the Raman spectra acquired from the Ti surface (not shown). This Raman result indicates that the entire damaged area was covered by a very thin layer of tungsten disulfide. It was confirmed by HR TEM that it indeed was covered by thin a tribofilm (thickness 10-50 nm) consisting mostly of well oriented (i.e. basal planes parallel to the surface) WS_2 layers together with crystalline and amorphous tungsten oxide (Fig. 10).

EFTEM elemental maps further corroborated these results, see Fig. 11. It is worth noting that the Ti interlayer contains an approximately 30 nm thick nitrogen-rich surface layer. Thus, the W-S-N coating was deposited on a TiN surface.

4. Discussion

The observed transfer film formation and structural transformation of the coatings provide interesting information about the wear mechanisms. In the initial stage, the highest asperities are worn down and the surfaces flatten. The wear debris from the steel ball must become removed from the contact area, since no traces of iron or iron oxides were observed on either part. The formation of a WS_2 tribofilm further smoothen the surfaces. The tribofilm is easy to shear and is readily smeared out, which helps covering initial scratches and surface defects. The surface roughness drops from $Ra > 30$ nm to $Ra < 1$ nm in the sliding direction, thereby reducing friction related processes such as ploughing and other plastic deformation to a

minimum. The presence of the low shear strength tribofilm on top of a hard coating on both surfaces results in the ideal combination of a small contact area and a very low shear stress. This tribo-induced or self-generated system can therefore provide a low-friction condition similar to the inter-crystalline slip known for the lowest observed friction [2].

Due to the nature of the analysed surfaces and limitations of the experimental techniques, we cannot rule out the presence of oxygen in the top part of the tribofilm. Even if oxygen would be present, Fleischauer and Lince [32] have demonstrated that its presence in sputtered MoS_2 films does not increase the friction substantially, provided that it substitutes sulphur atoms in the basal plane. Since TEM observations strongly suggest the exclusive layered structure typical of WS_2 on the outermost surfaces of both parts, the oxygen could only be present as a substitution (i.e. a $WS_{2-x}O_x$ phase would be formed) and thus would not strongly interfere in the sliding process. The absence of nitrogen in the contact zone of either part indicates it was released in gaseous form.

It has been observed that sliding in dry nitrogen improved the wear life significantly for MoS_2 coatings containing relatively large amounts of residual water [33]. The nitrogen was suggested to bond with the water, preventing it from adsorbing to the surface and forming hydrogen bonds between the easy-sheared planes, increasing the shear strength and thus the friction and wear. Previous investigations of W-N coatings have also shown that nitrogen bonded to tungsten can be released due to the reaction of tungsten with oxygen [4,5]. The exact mechanism for the depletion of nitrogen in this study requires further studies.

The presence of tungsten oxide below the thin tungsten disulfide is another interesting feature. Typically, a mixture of WO_3 nanograins (size up to 5 nm) and amorphous tungsten oxide was observed in the tribofilms. We should point out that amorphous tungsten oxide could easily be crystallized under the electron beam. Thus, we cannot rule out the possibility that the tungsten oxide layer formed was amorphous. The origin of such an oxide layer just below the WS_2

layer is not known. It is not clear whether the oxygen originated from the test atmosphere or from the coating, although the latter possibility is more likely (the nitrogen gas used in the tribological testing was pure, the test chamber was purged with nitrogen before the test and the chamber was slightly over-pressured with continuous flooding of nitrogen during the whole tests). In our recent study [34] we showed that a W-S-C-Cr coating exhibited a similar tribofilm structure when sliding in dry nitrogen, *i.e.* a thin top layer consisting exclusively of well-ordered WS_2 layers with an underlying oxide layer. On the other hand, such subsurface oxide layers were not observed in the related W-S-C [30] and Mo-Se-C [4] systems. Interestingly, the four low-friction coatings referred to above can be grouped into two different microstructures: the W-S-N and W-S-C-Cr coatings are amorphous, whereas the structure of the W-S-C and Mo-Se-C coatings could be described as randomly oriented single WS_2/$MoSe_2$ platelets embedded into an amorphous carbon matrix. Possibly, the possible formation of an oxide sub layer during the sliding process could be related to the initial nanostructure of the coating.

Laser surface texturing, which is often used for adaptive solid lubrication, has shown similar features to behaviour of the W-S-N coating presented in this study. Dimples produced by the laser become filled by solid lubricant MoS_2, deposited either by burnishing [35,36] or hot pressing [37]. The solid lubricant adhered to the ball during its passage over the dimple and then transferred to other parts of the wear track. Thus, the dimples acted as solid lubricant reservoirs. In our case, the formation of a tribofilm on the ball during the sliding process fulfils the same function, *i.e.* supplying a lubricant during the sliding process. It should be pointed out that the transfer of the tribofilm from the ball to the damaged surface must be smooth and continuous, since we have not observed any significant fluctuations in the friction coefficient. In fact, it is possible that the slightly higher friction measured on the wear tracks with partial coating failures was caused primarily by the topography of the damaged parts (i.e.

increased roughness and steps), since the contact interface was almost identical with that of non-damaged surfaces (*i.e.* dominated be well-aligned WS_2).

5 Conclusions

We have demonstrated that the tribo-induced formation of a WS_2 tribofilm, where the easy-slip planes become aligned along the sliding direction, in combination with a significant reduction of the surface roughness, leads to extremely low friction in nitrogen and humid air. In fact the level is comparable to that reported for incommensurate sliding of ideal MoS_2 crystals in vacuum. The material below the thin aligned easy-shear layer transforms from amorphous to self-ordered WS_2 platelets. This layer is supported by the remaining underlying hard amorphous W-S-N matrix, which keeps the area of real contact low and provides the firm support that allows the outermost basal planes to become ideally aligned during sliding.

This formation of a well-oriented self-lubricating phase further improves the low-friction behaviour, by being capable of immediately replenishing the worn contact layer. Moreover, the coating exhibits a remarkable damage recovery providing low friction despite coating delamination over large areas.

This demonstrates that structures with self-adaptability, such as this amorphous W-S-N film, may offer friction levels almost one order magnitude lower than that of TMD-based sputtered films, TMD nanotubes or fullerene-like nanoparticles. Our results show that coatings with friction coefficients close to the intrinsic value for a WS_2 crystal could be prepared by a simple and robust process, which can be directly used in many engineering applications. Alloying tungsten disulphide with nitrogen is thus shown to be a promising way to achieve amorphous, extremely wear resistant coatings capable of providing ultralow friction.

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Figure 2: *The evolution of the friction coefficient of the W-S-N coating tested in humid air (RH 10%) and dry nitrogen (inset).*

Figure 3: *SEM micrograph of the wear track formed in dry nitrogen at 20 N load, with the position of the FIB cut indicated.*

Figure 4: *HR TEM images from different positions on the wear track after 100 000 ball passages in dry nitrogen under a load of 20 N. The sliding direction is inwards in the paper. The tribofilm here consists exclusively of the easy-shear (002) WS_2 planes aligned parallel to the sliding direction, on top of a thin W-O layer (amorphous oxide plus WO_3 nanograins). Further down the film contains semi-ordered WS_2 platelets.*

Figure 5: *Cross section TEM images of the tribofilm formed on the ball in dry nitrogen, after 100.000 laps with a load of 20 N. (To prevent further oxidation and contamination, a thin gold layer was deposited on top of the tribofilm immediately after the sliding test.)*
a) Overview through the dense and defect-free tribofilm. Tungsten oxide is predominant except at the outermost surface. b) The outermost surface on the tribofilm consisting of well-aligned WS_2 layers. c) The bottommost part of the film, closest to the steel ball surface, also consisting of well-oriented WS_2 layers.

Figure 6: *Raman spectra from different parts of the wear track formed in dry nitrogen after 100 000 laps with a load of 20 N; 1 – as-deposited film, 2 – side of the wear track, 3 – slightly off centre and 4 – in the centre of the wear track.*

Figure 7: *SEM image and AES elemental maps of a transfer film formed on the ball in dry nitrogen after 100 000 laps with a load of 20 N. The black box in the SEM image indicate the area from where the maps were acquired and the white smaller boxes the areas of the compositional analysis. To remove contaminants, the surfaces were sputter cleaned for 10 s to a depth of approximately 3 nm before analysis. The maps should not be compared quantitatiely.*

Figure 8: *Friction coefficient between the W-S-N coating and steel balls in 100 000 lap tests in dry nitrogen with 20 N load. a) The red curve, displaying the lowest friction, shows the trend in an undamaged track. The blue and green curves show trends for 2 damaged wear tracks. The initial unstable appearance of the friction curves from the damaged parts*

indicates that the majority of the adhesive failure occurred relatively early. b) The adhesive damage of the wear track after 100 000 laps with a load of 20 N in dry nitrogen.

Figure 9: *SEM picture of the partially damaged wear track (upper left) and corresponding EDX elemental maps, acquired with 10 kV acceleration voltage. No iron was detected (map not shown). The maps should not be compared quantitatively.*

Figure 10: *HR TEM observations of the surface of the damaged parts of the wear track. Notice the different thicknesses of the tribofilm (upper left and right). At higher magnification the microstructure of the tribofilm with well oriented WS_2 layers mixed with WO_3 nanograins becomes clear (below).*

Figure 11: *TEM cross sectional view and EFTEM elemental maps of the tribofilm formed on top of the Ti interlayer from a part where the coating had suffered adhesive failure. It is clearly seen that the delamination occurred at the interface between the Titanium interlayer and the coating. Note the thin nitrogen layer on the Ti interlayer – probably TiN formed during the deposition.*

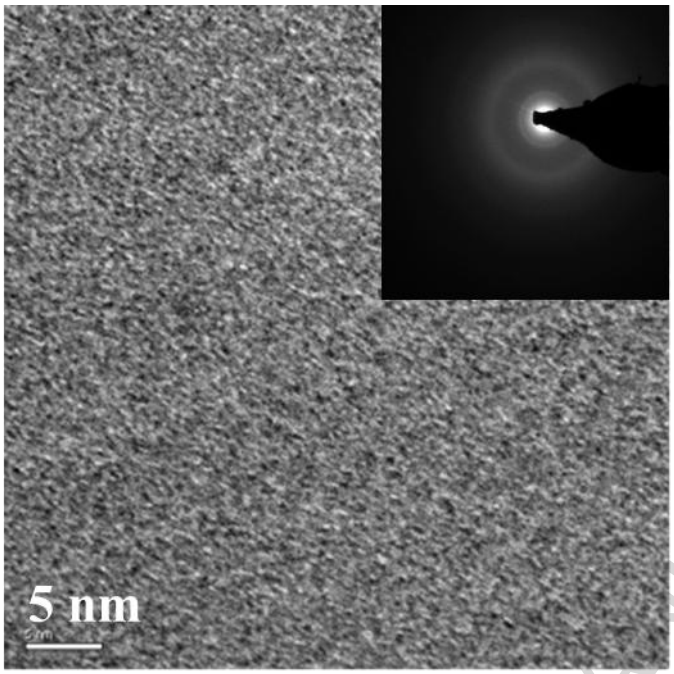

Figure 1

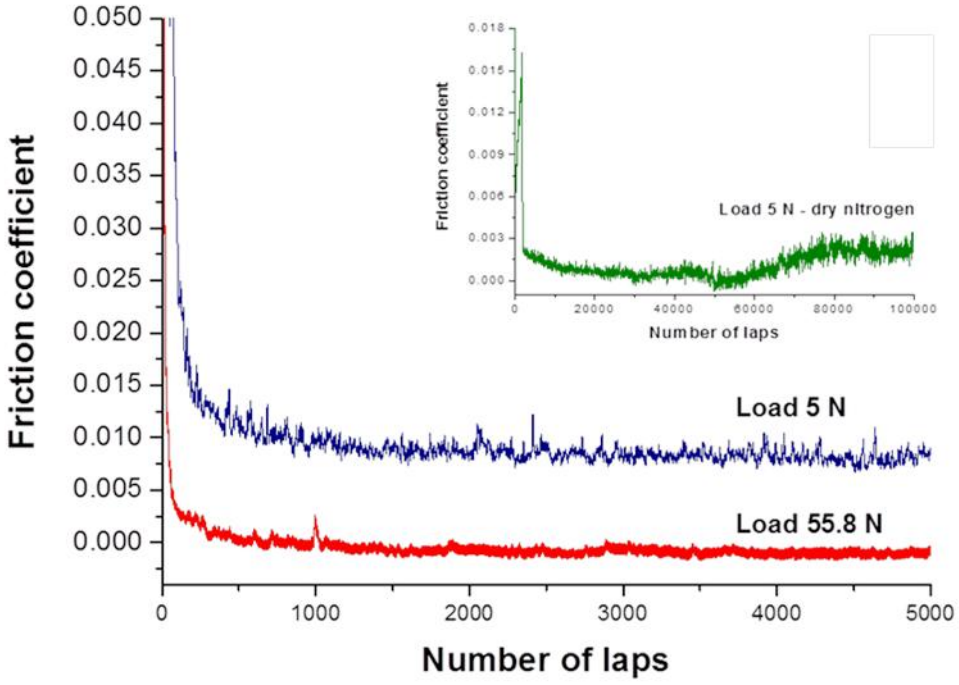

Figure 2

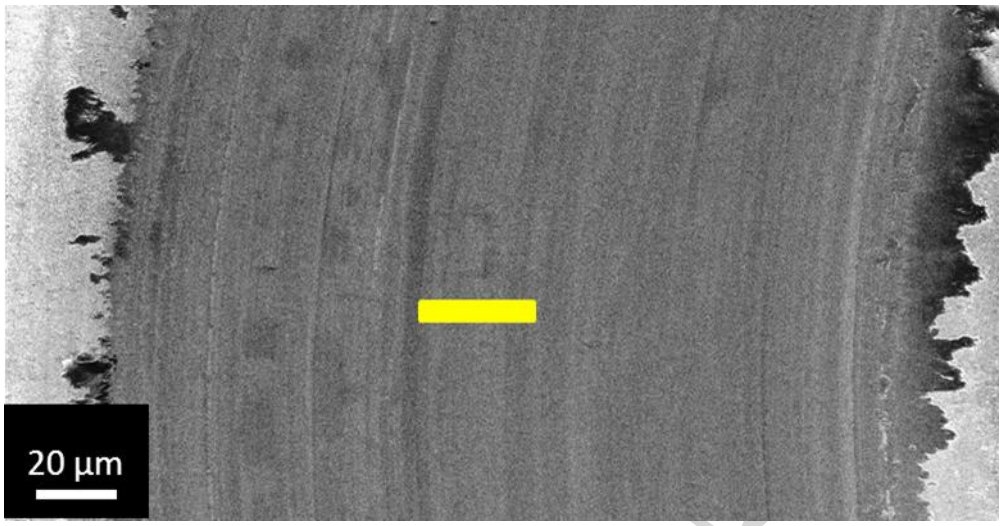

Figure 3

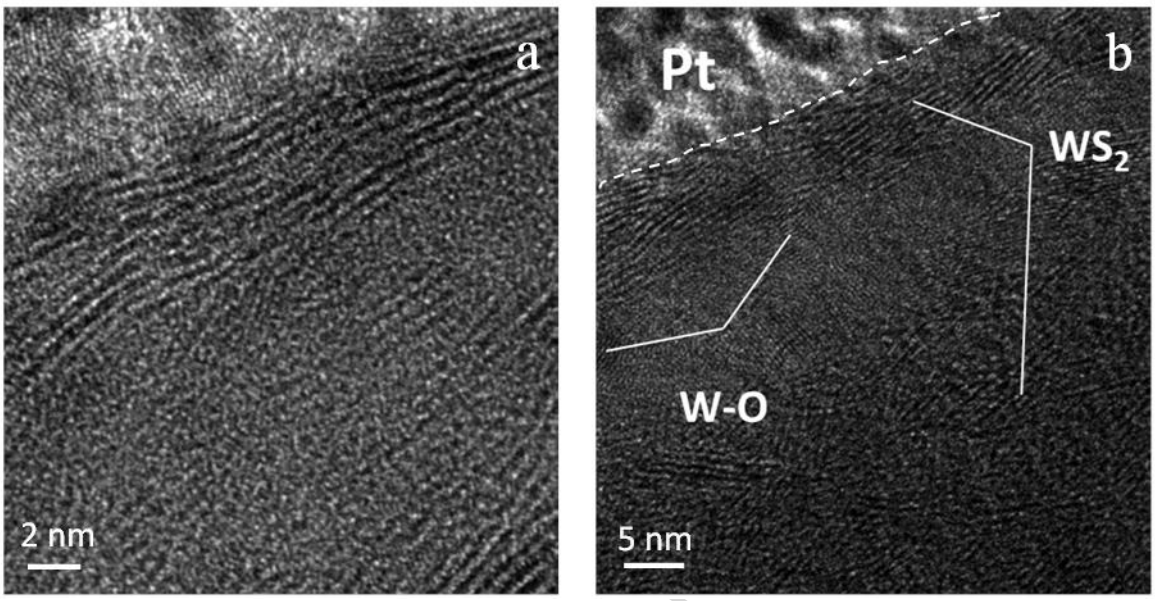

Figure 4

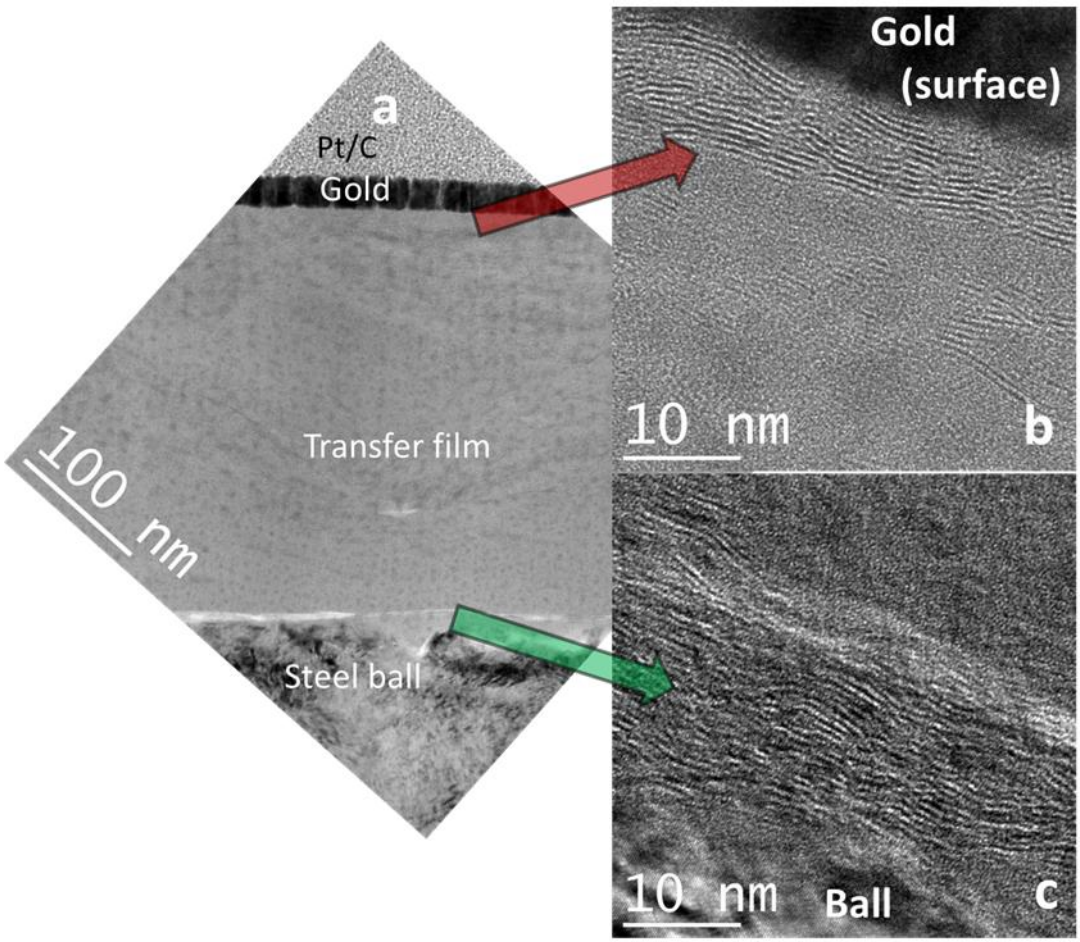

Figure 5

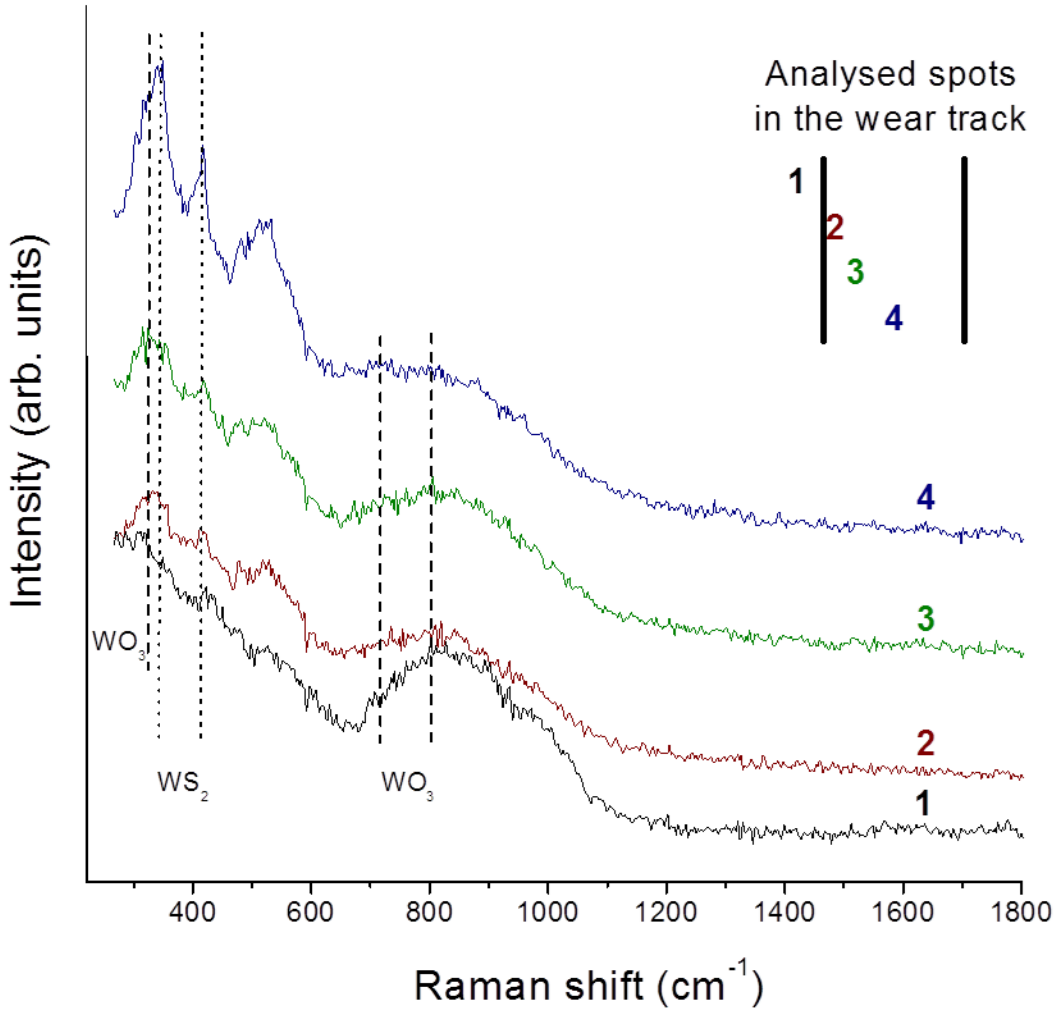

Figure 6

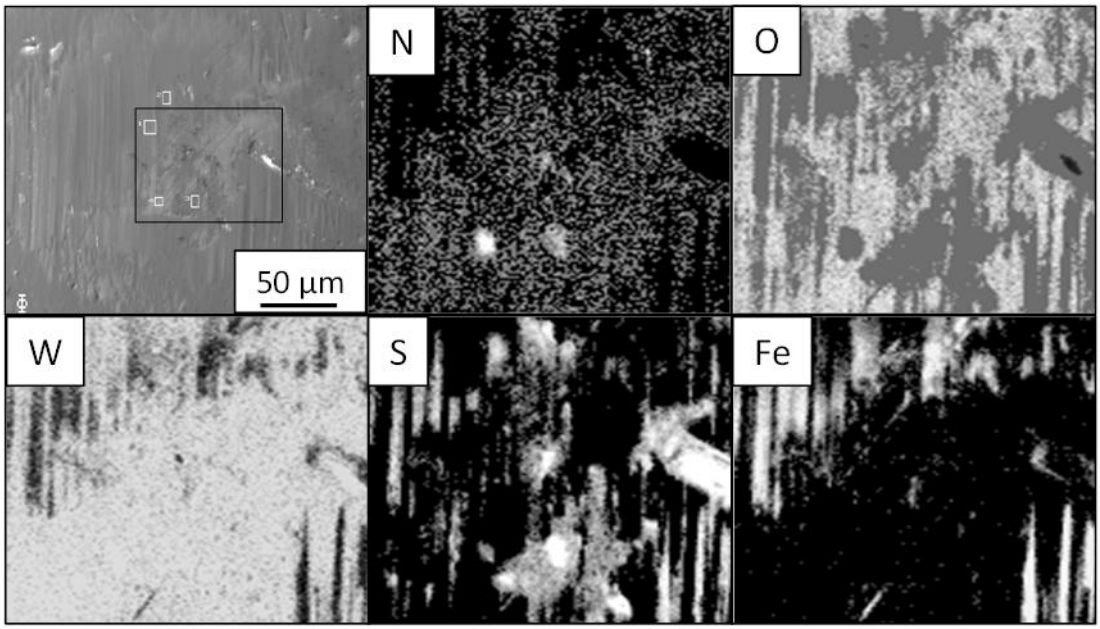

Figure 7

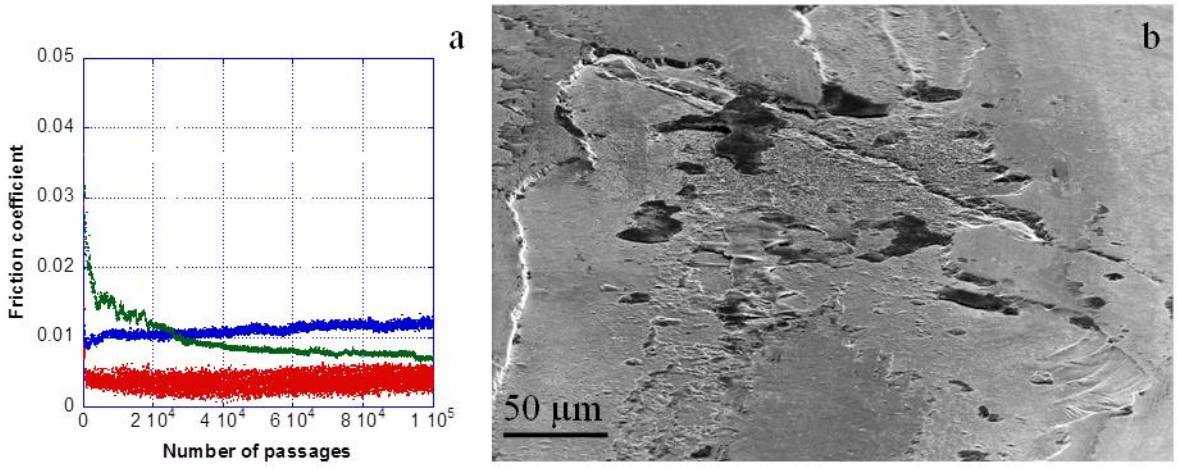

Figure 8

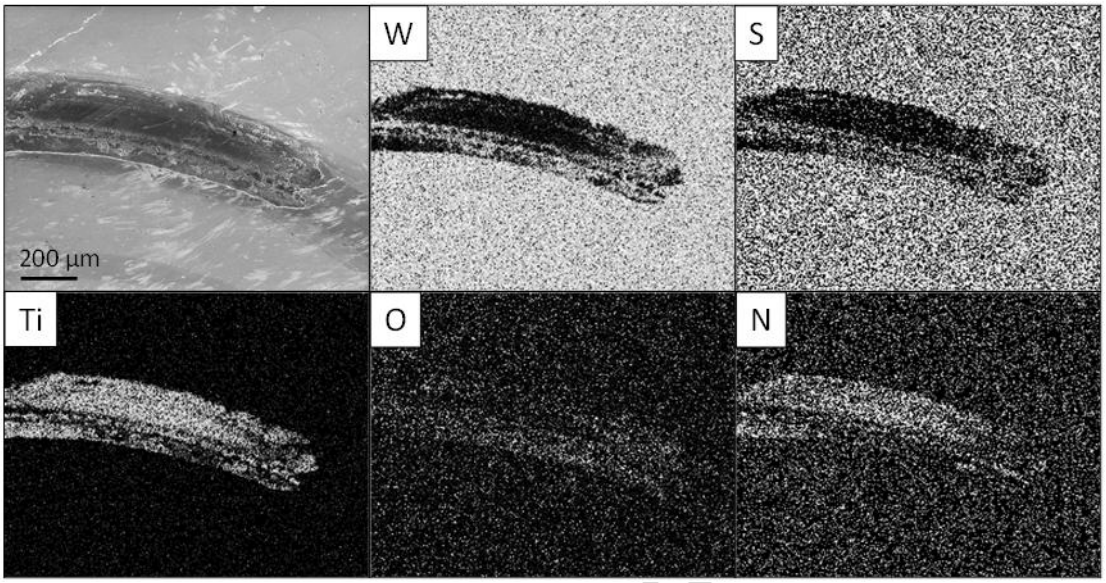

Figure 9

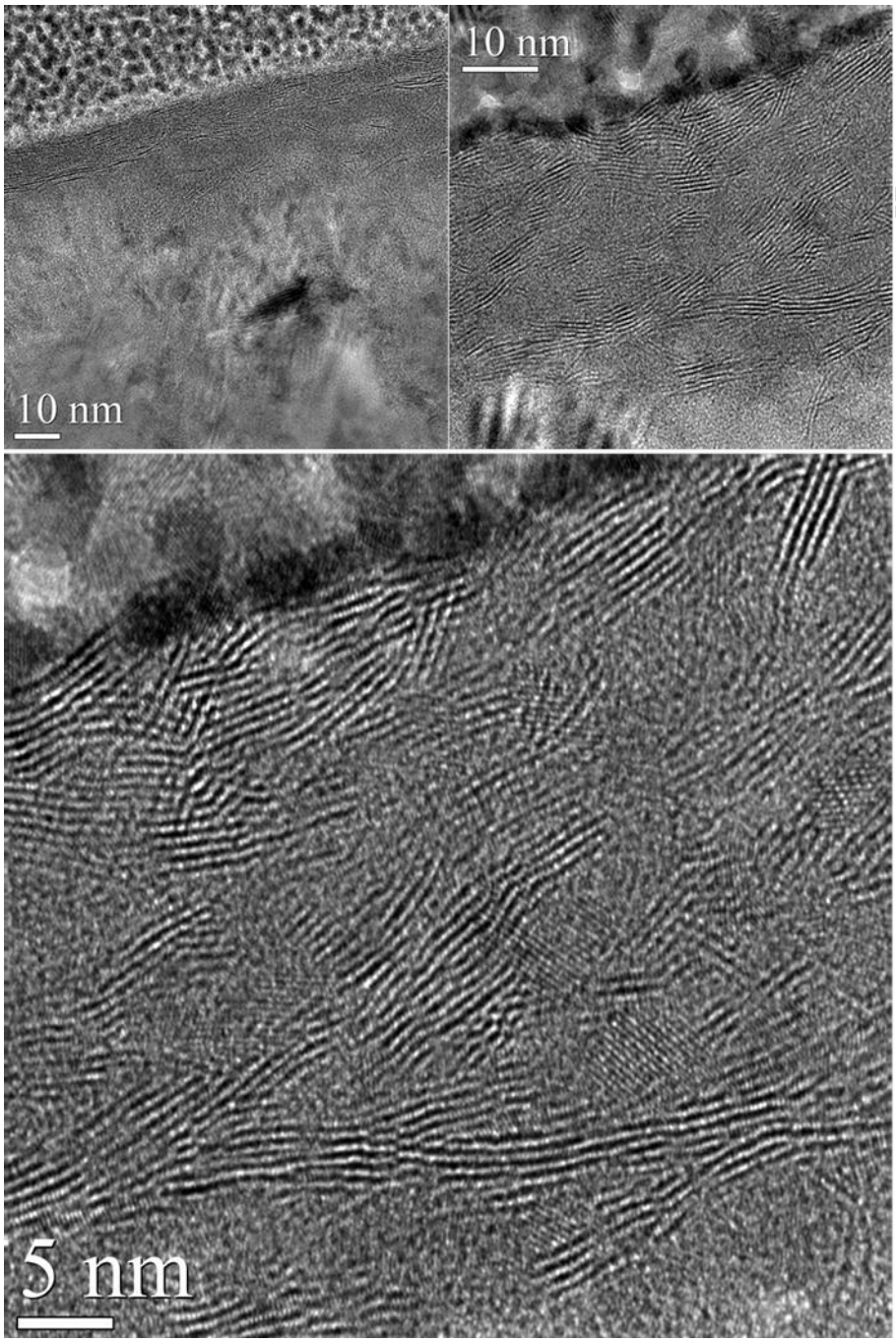

Figure 10

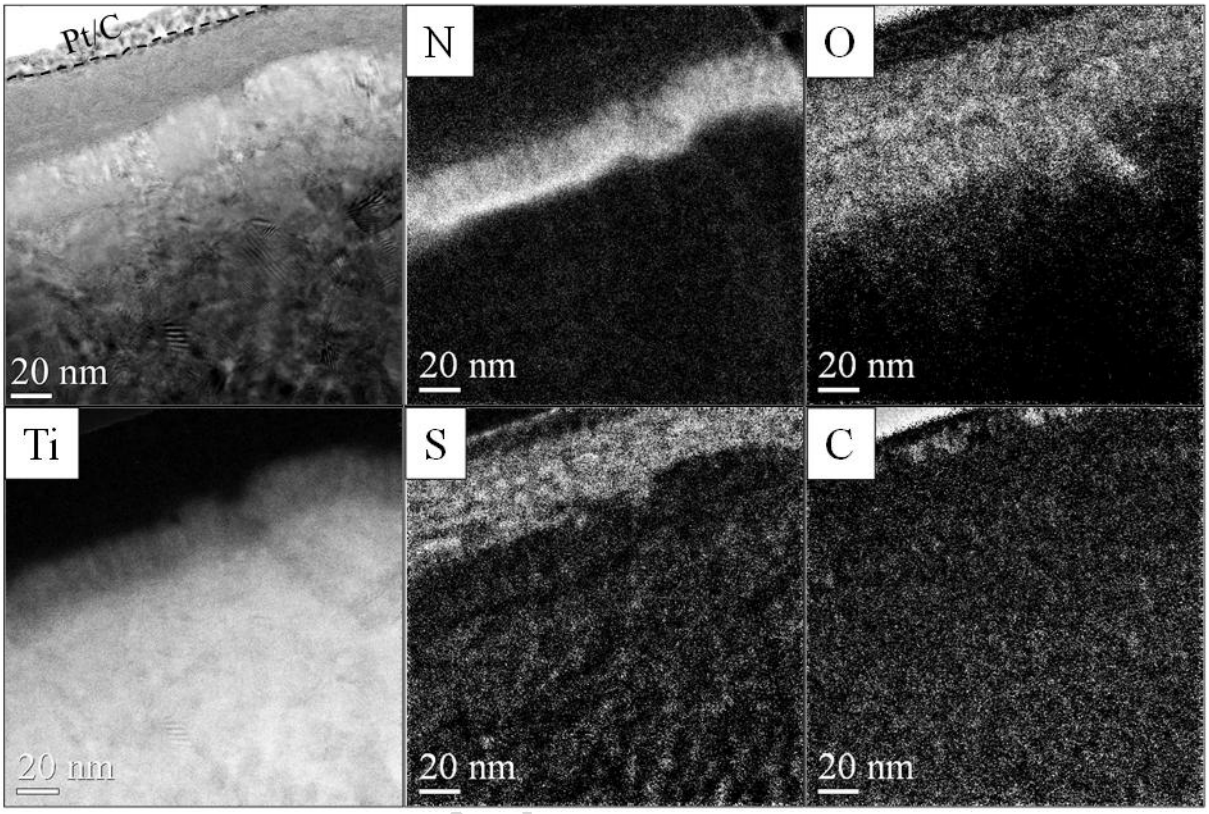

Figure 11

Highlights

- W-S-N coatings deposited by magnetron sputtering were amorphous
- Friction coefficient in nitrogen was as low as 0.003
- Formation of well-ordered WS2 tribolayer was observed on the worn surfaces
- W-S-N coating exhibited a remarkable recovery after damage