



Transfer layer evolution during friction in W-C:H coatings

The case study focuses on the development of nanocomposite W-C:H coatings with high hardness and simultaneously, with a lower coefficient of friction. We studied the W-C:H coating system with different hydrogenated carbon matrix content obtained during a hybrid PVD-PECVD process from hydrocarbons (mostly acetylene) added into sputtering atmosphere. The coatings were prepared using three different PVD techniques (DC magnetron sputtering, HiPIMS, HiTUS) and the contents of hydrogenated carbon were controlled via different additions of hydrocarbon gas.

Since the friction behavior of the coatings studied herein is controlled by the formation of a transfer layer on the ball, while the focus of our earlier works was to study the basic relationships between hardness and coefficients of friction, the focus turned to the investigation of the evolution of the transfer layer itself.

It is important to note that that the humidity is a very important factor in friction in hydrogenated carbon (based) coatings. Several W-C:H coatings with different amounts of carbon and hydrogen were tested in humid air and in flowing nitrogen with reduced humidity. Up to now, conventional optical microscopy, SEM/EDS, SEM/FIB (Figure 1) and Raman spectroscopy were used to evaluate different aspects of the transfer layer formation. However, we have introduced the information obtained with the 3D optical profiler, Plu neox, which provides further qualitative and quantitative information about the transfer layer within the whole contact area.



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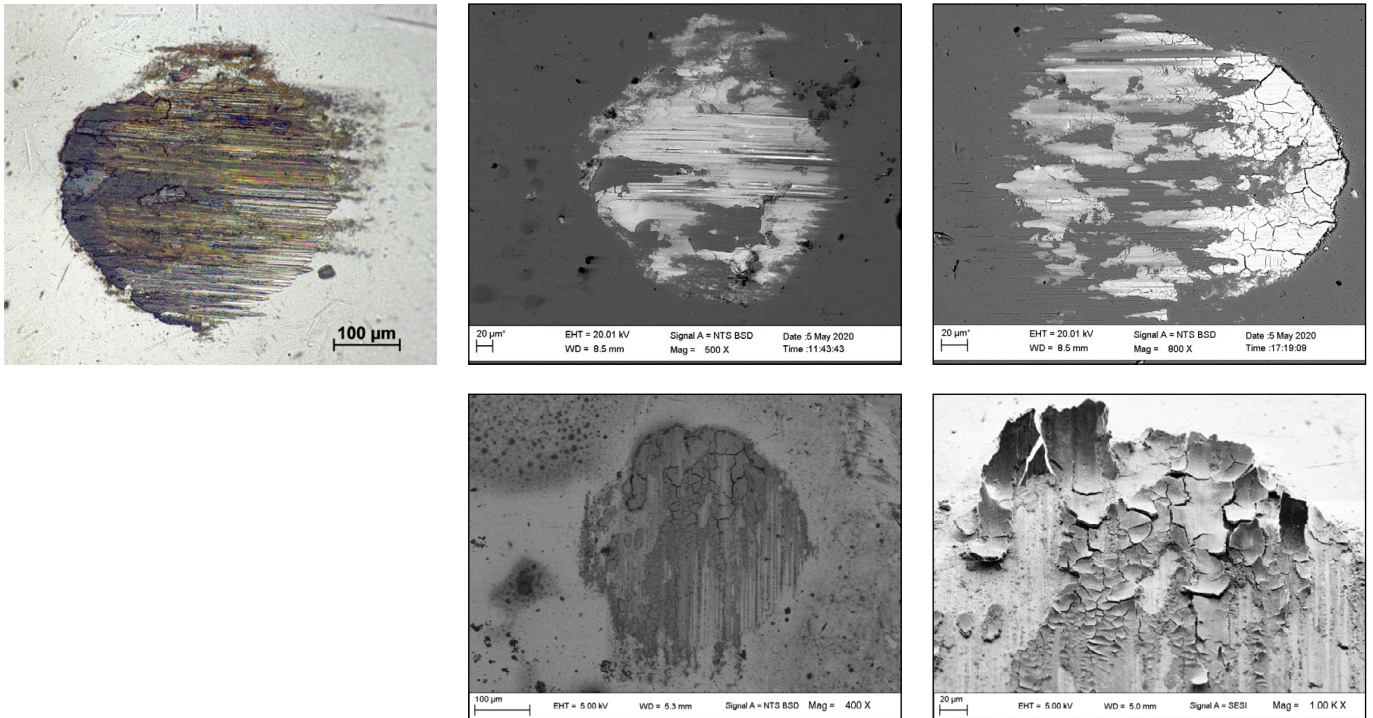


Figure 1. SEM (and light microscopy) micrographs of the transfer layers formed on the worn ball cap during friction with various W-C:H coatings and under different conditions.

This approach is not absolutely new, already in 2003 Scharf and Singer [Tribol. Letters, vol. 14, No1 and No2] applied Raman combined with optical profilometry in 2D to evaluate thickness of the transfer layer in a-C:H nanocomposite coatings. To the best of our knowledge, we are not aware of any study where Confocal 3D optical profilometry is applied in nanocomposite W-C:H coatings and in such an extensive range.

■ Measurements

On a steel bearing ball with a diameter of 6 mm, a worn ball cap with the transfer layer is formed during friction and strongly affects the resulting coefficient of friction. The diameter of the worn cap is around 200-400 µm and the transfer layer covers only some parts of this contact area: a thick and dense but heavily cracked layer develops along the leading edge while a much thinner transfer layer occurs in the central part of the contact area.

The 3D optical profiler Plu neox in Confocal mode and using a 20X objective magnification will be used to compute:

1. The amount of material of the ball lost due to wear.
2. The thickness of the transfer layer on the worn ball cap and its distribution over the contact area. Based on this information, the correlations between the deposition conditions, composition and coating structure related to friction/wear resistance and transfer layer can be established.

To obtain this data, we took 3D topographies of the initial ball, the worn ball cap after testing (some examples are shown in figures 2, 3 and 4) with and without the transfer layer and subtracted them from each other. The 3D distribution of the thickness of transfer layer can be obtained as the difference between the last two images whereas the worn volume of the ball as the difference between the initial ball and of the ball without the transfer layer.

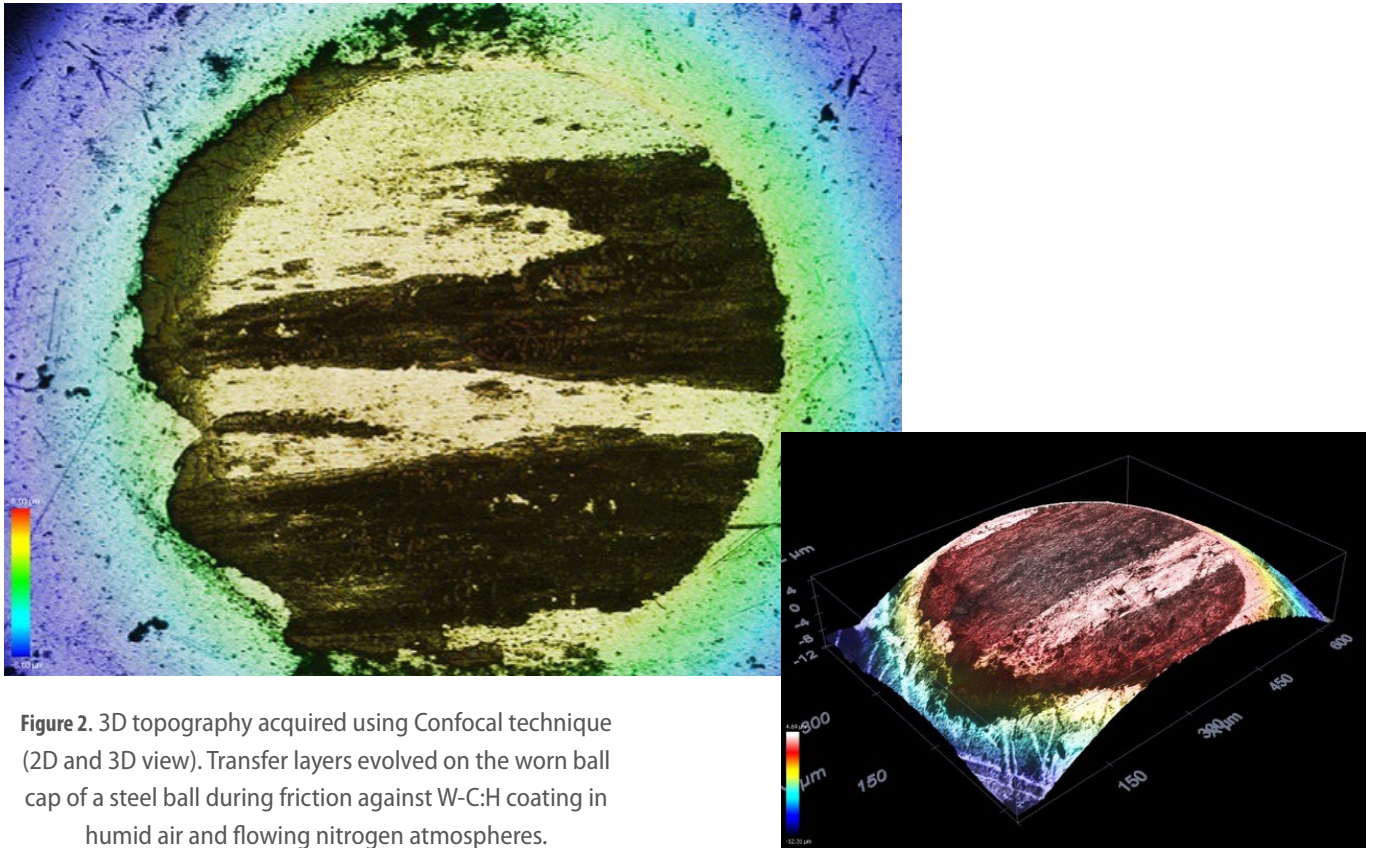


Figure 2. 3D topography acquired using Confocal technique (2D and 3D view). Transfer layers evolved on the worn ball cap of a steel ball during friction against W-C:H coating in humid air and flowing nitrogen atmospheres.

The Plu neox is intended to measure the volumetric thickness of the transfer layer on the worn ball cap and its distribution over the whole contact area. This provides important information about the role of the transfer layer in the improvement of the friction performance of W-C:H coatings and subsequent optimization of the coatings structure and composition to obtain the best tribological performance in different environments.

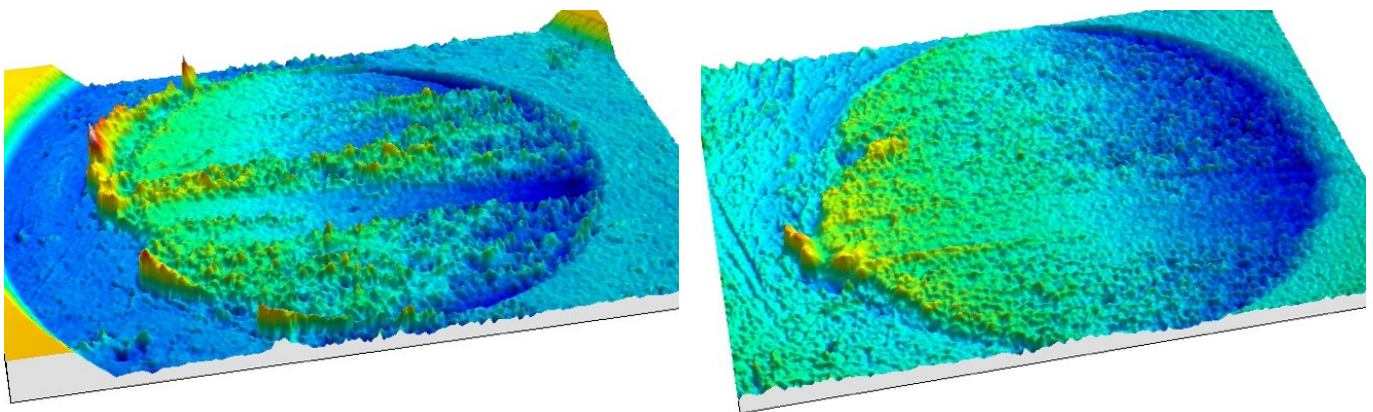


Figure 3. 3D topographies of the same tribological test applied in Humid air (a) and flowing nitrogen (b) maintaining the same W-C:H coating conditions.

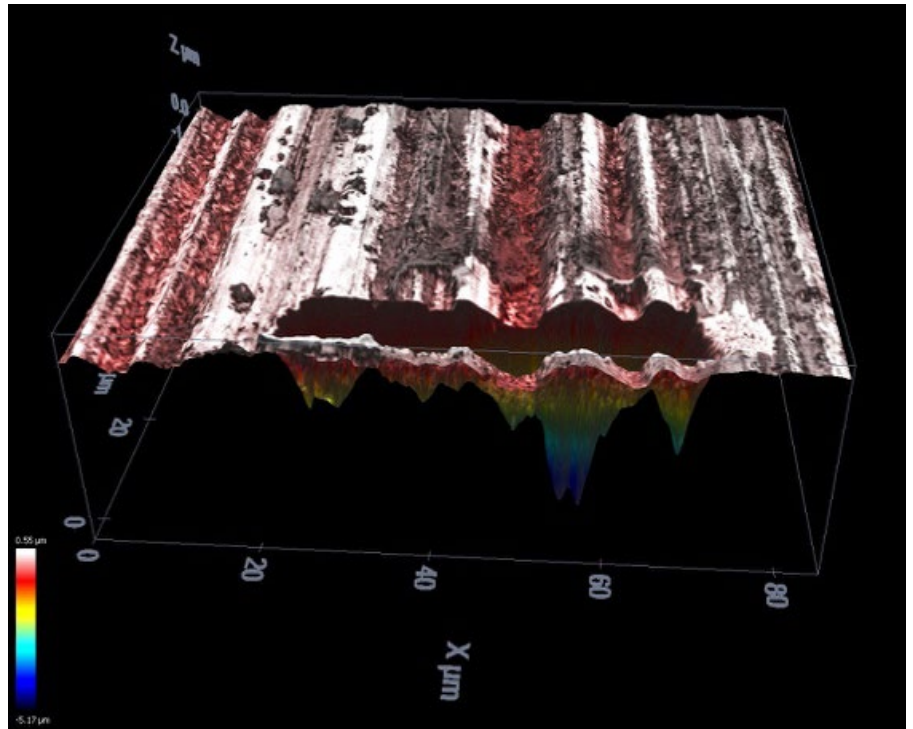


Figure 4. Topography of the scratched area in the contact zone with the transfer layer filling the grooves; the groove profiles were also revealed by a FIB cross section.

■ Results

It has been measured the evolution of the transfer layer and steel ball wear in the studied W-C coating/steel ball system during friction test up to a predetermined length. Results are shown in the next table:

Sliding distance/COF (m/-)	Transfer layer volume (μm^3)	Avg. transfer layer formation rate ($\mu\text{m}^3/\text{N.m}$)	Worn volume of the ball (μm^3)	Average wear rate of the ball ($\mu\text{m}^3/\text{N.m}$)
30 / 0.68	11646	1 553	24034	3205
150 / 0.79	10580	288	89281	2381
500 / 0.58	9238	74	52582	421
2000 / 0.75	15100	30	283975	568

■ Conclusions

The main goal, the 3D visualization of the transfer layer formed during friction of W-C:H coatings, was achieved. The treatment of the surface topographies of the contact areas with and without the transfer layer allowed us to subtract the contribution of the volume of the ball lost due to its wear and to quantify only the volume of a transfer layer. Thus, the key data for the understanding of the correlations between the formation of transfer layer and friction performance of W-C:H coatings can be obtained.

We used the Plu neox in Confocal mode using a 20X objective to investigate the topography of the contact surfaces after friction tests between a steel ball and W-C:H coatings. This combination provided sufficient resolution within one shot. In limited cases, a 150X objective and stitching of the images applied to obtain higher details may be required. However, for general purposes and consistent conditions, a 20X objective was sufficient.

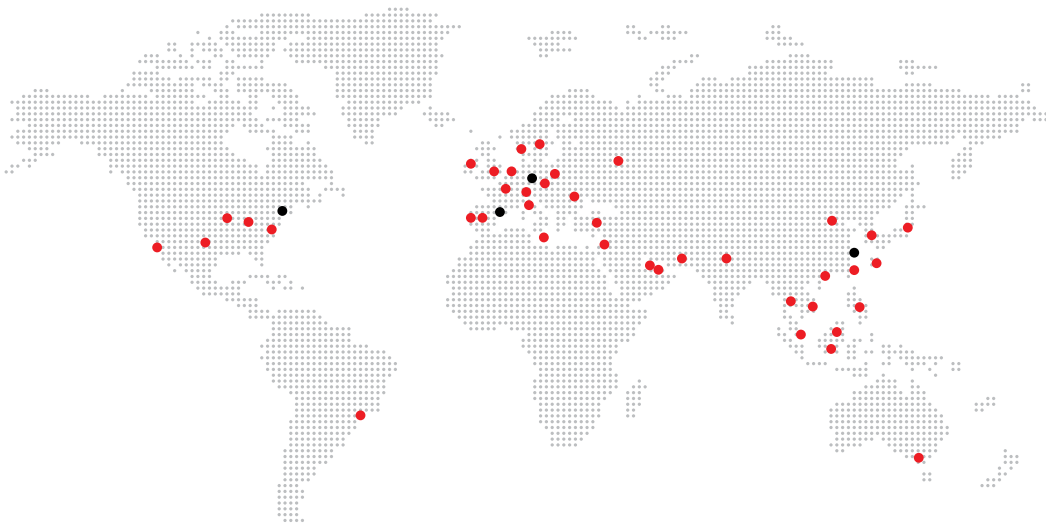
The contact surfaces were observed by conventional light microscopy in bright field and in DIC modes as well as in SEM, both in SE and BSE modes. Both techniques provided valuable information, but the quantitative information concerning volume of the studied layers was not available. The 3D optical profiler was able to provide additional information about topography, including not only 3D images with extensive possibilities to manipulate their graphics but also to obtain line profiles, volumetric and surface data (besides data from linear measurements, available also from light and scanning electron microscopy). Thus, confocal microscopy became a complimentary technique with the importance comparable to that of well-established ones such SEM and light microscopy. Moreover, the additional quantitative information obtained at the same time makes 3D optical profilometry more advantageous than the conventional light microscopy, especially when uneven surfaces must be imagined. Thus, confocal microscopy seems to be the best technique for the purpose of our study.

With Sensofar Plu neox, the required information was obtained easily. We use it often in our laboratory for the observation of contact surfaces after tribological tests because of its ease and speed, good graphic user interface and its quantitative outputs. It also became the preferred choice over conventional contact profilometers that were often applied for the same purpose in the past.

■ References

There are no direct publications describing the above mentioned work on transfer layers (the work is still ongoing) but it has been part of longer and more comprehensive studies with various aspects of structure vs. mechanical vs. tribological properties of W-C:H coatings which were already reported in the following studies:

1. F. Lofaj, P. Hviščová, P. Zubko, D. Németh, M. Kabátová, **Mechanical and tribological properties of the High Target Utilization Sputtering W-C coatings on different substrates**, Int.J. Refractory Metals and Hard Materials, 80 (2019) 305-314.
2. F. Lofaj, M. Kabátová, M. Klich, D. Vaňa, J. Dobrovodský, **The comparison of structure and properties in DC magnetron sputtered and HiPIMS W-C:H coatings with different hydrogen content**, Ceram. Int., 45 (2019) 9502-9514
3. F. Lofaj, M. Kabátová, M. Klich, D. Medved', V. Girman, **Tribological behavior of hydrogenated W-C/a-C:H coatings deposited by three different sputtering techniques**, Cerâmica, 65 (2019) 58-69.
4. F. Lofaj, M. Kabátová, L. Kvetková, J. Dobrovodský, V. Girman, **Hybrid PVD-PECVD W-C:H coatings prepared by different sputtering techniques: The comparison of deposition processes, composition and properties**, Surf. Coat. Technol. 375 (2019) 839-853.
5. F. Lofaj, M. Kabátová, L. Kvetková, J. Dobrovodský, **The effects of deposition conditions on hydrogenation, hardness and elastic modulus of W-C:H coatings**, J. Eur. Ceram. Soc., 40 (2020) 2721-2730.
6. F. Lofaj, M. Kabátová, J. Dobrovodský, G. Cempura, **Hydrogenation and hybridization in hard W-C:H coatings prepared by hybrid PVD-PECVD method with methane and acetylene**, Int. J. Refractory Met. Hard Mat., 88 (2020) 105211.



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