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Highlights

1. Friction force microscopy measurements as a function of load for WSC/Cr coatings.
2. Non-linear contact area dependence on load in solid-solid nanocontacts.
3. Friction coefficient decreases with increasing applied load.
4. Low shear strength - formation of an easy-shear layer at the sliding interface.
Frictional properties of self-adaptive chromium doped tungsten-sulfur-carbon coatings at nanoscale

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Abstract

Transition metal dichalcogenides (TMD) are excellent dry lubricants forming thin (~ 10 nm) tribolayer that simultaneously protects the coating from environmental attack and provides low friction. In this paper, we focus on nanoscale frictional properties of chromium doped tungsten-sulfur-carbon (WSC-Cr) coatings with various Cr content. Friction force microscopy was used to investigate friction force as a function of load. A non-linear contact area dependence on the normal force was observed. The calculated interfacial shear strength was relatively low in the region of 70 – 99 MPa. Friction coefficient decreased with increased applied load independently of chromium content in the coatings.

Keywords

Atomic force microscopy, friction force microscopy, nanoscale friction, magnetron sputtering, WSC/Cr.

1. Introduction

Macroscopic tribology often focuses on determining the friction coefficient and wear rate for materials. Neither friction coefficient nor the wear rate are intrinsic physical property of the material in question, and both depend on the specific structure, chemistry, elastic/plastic properties of surfaces, as well as the environment in which measurements are performed, mechanics of instrument, etc. Nanometer-scale measurements of friction and intermolecular forces can disentangle the complex factors contributing to friction. Atomic force microscopy
(AFM) can be used to measure normal and lateral forces at the nanometer scale and, thus, represents a powerful tool for probing the microscopic mechanisms of friction [1-3]. Studies of friction between probe tips and different surfaces have yielded a number of interesting observations, including atomic scale stick-slip motion [2, 4], friction force dependence on contact area [3, 5-12], or the dependence of functional groups such as COOH, CH₃ on friction [8, 13].

Friction (also known as lateral) force microscopy (FFM, LFM) is often used to study nanoscale tribological properties of lamellar solid lubricants such as niobium diselenide (NbSe₂), molybdenum oxide (MoO₃), molybdenum disulphide (MoS₂), graphite and graphene, as well as non-layered carbon-based solids [9-11, 14-18]. Most of those layered materials are used as solid lubricant films in engineering applications to reduce friction.

Transition metal dichalcogenides (TMD), such as MoS₂ or WS₂, are excellent lubricants in vacuum and dry atmospheres. However, their tribological behaviour deteriorates in the presence of water vapour due to rapid oxidation. Moreover, TMD coatings deposited by sputtering, which is one of the most used and convenient methods, are soft and porous with limited load-bearing capacity. Among many attempts to improve TMD mechanical and tribological properties (e.g. co-sputtering with metals or compounds), the design of TMD coatings co-deposited with carbon (MoSeC [19, 20], WSC [19, 21, 22]) emerges thanks to their unique self-adaptive structure. Such coatings produce during sliding a thin TMD tribolayer with basal planes parallel to the sliding direction; the tribolayer simultaneously protects the coating from environmental attack and provides low friction. Thickness of the tribolayer is typically lower than 10 nm, which makes its analysis very challenging.

In this paper we will focus on nanoscale frictional properties of as-deposited WSC-Cr coatings with different Cr content.
2. Experiment

2.1 Coating characterisation

The W-S-C-Cr films were deposited using an r.f. magnetron sputtering chamber (Edwards, UK); the deposition conditions are in detail given in Ref. [22]. Prior to the coating deposition, the substrates were cleaned by establishing the plasma close to the substrates electrode for 20 minutes. Two targets were used: chromium (purity 99.9\%) and carbon (graphite, purity 99.6\%) with WS$_2$ pellets (purity 99\%) placed on its erosion zone. The number of WS$_2$ pellets was determined empirically to obtain approximately 40 at.\% of C in a deposition without Cr. The Cr content was controlled via the power applied to each target. A pure Cr interlayer was deposited on the substrates before every coating deposition, to improve adhesion.

The coatings were deposited on polished W.Nr. 1.2379 (X153CrMoV12; AISI D2) steel substrates (Ra < 30 nm, diameters of 50 and 22 mm, hardness 9 GPa) used for macroscopical tribological measurements and on Si wafer used in presented study.

The chemical composition of the coatings was evaluated by electron probe microanalysis (EPMA). Hardness ($H$) and reduced modulus ($E_r$) values were determined by depth-sensing indentation and adhesion was evaluated by progressive load scratch tests. The chemical bonding of the films was analyzed by Raman spectroscopy (DPSS laser, wavelength 532 nm), Fourier-transform infrared spectroscopy (FTIR) and X-ray photoelectron spectroscopy (XPS; Mg K\textalpha\ radiation). The structure was analyzed by X-ray diffraction (Cu K\textalpha\ radiation) and by transmission electron microscopy (TEM); the morphology was observed by scanning electron microscopy (SEM).
2.2 Friction measurements using AFM

Measurements of surface topography and lateral force were performed in air at room temperature using atomic force microscope (MAC Mode III, 5500 Scanning Probe Microscopy, Agilent Technologies, US). PicoView 1.12 and PicoImage Basics 6.0 (Agilent Technologies, US) software were used for data acquisition and image analysis, respectively. Standard lateral force mode silicon probes (NanoWorld, distributed by Windsor Scientific, UK) with nominal spring constant of 0.2 N/m and tip radii of 8 - 10 nm were used. Actual spring constant values for every cantilever were obtained using built-in thermal noise method [23]. The measured constant varied in the range 0.2 – 0.45 N/m. Normal forces were calibrated by measuring the deflection sensitivity (nm/V) from the slope of the linear part of a force-displacement curve obtained on a flat silicon surface. The normal force, $F_N$, was set to be zero at the point where the cantilever left the surface. Calibration of lateral forces using commercially available gratings was achieved using the “wedge calibration method” according to Ogletree et al. [24]. The probe geometry was obtained from the reconstruction of the tip shape from imaging of a calibration sample consisting of sharp Si spikes, TGT1 (NT-MDT, cone angle 50°, Moscow, Russia) [25]. The spikes have very small radii and a cone half angle similar to an angle of AFM probe. Therefore, the image of the spike is virtually the image of the AFM tip representing more precise probe geometry. A reconstruction algorithm presented in Refs. [25, 26] was built-in the PicoImage analytical software and was applied to recover the shape of the cantilever tip. The geometric mean radius of the tip was calculated by fit of a circle on the top of deconvoluted image profile. All calibration procedures were done before and after friction experiments. Tip radii were measured to be $8 \pm 2$ nm and $20 \pm 3$ nm before and after the experiments, respectively.

For the friction measurements the instrument was operating in contact mode with the long cantilever axis perpendicular to scanning direction. The lateral deflection was adjusted so that
it was zero with the tip out of contact with the surface. The normal applied load varied from 0 to 80 nN. For each load, topography and friction maps over areas of 1 x 1 µm² consisting of 512 lines were recorded at scanning speed of 3.99 µm/s. Friction forces were determined from trace-retrace loops acquired along single lines by subtracting and halving mean signals as described in Ref [1]. Surface area roughness of each sample was determined from topographical images of the size of 10 x 10 µm² using PicoImage software. Mean surface roughness, $S_a$ (arithmetic mean height deviation), related to the analysis of 3D areal surface texture, was calculated according to ISO 25178 standard using Gaussian filter 0.008 mm.

3. Results and Discussion

3.1 Coating composition, structure and mechanical properties

Three series of coatings were deposited, two with different Cr content and WSC as a reference. The chemical composition measured by EPMA is shown in Table 1. To facilitate reading, we denominate coatings as WSC-Cr-X, where X is the chromium content. The WSC-Cr coatings thickness increased with chromium content from 1.7 to 2.4 µm including the 300 nm thick Cr interlayer improving adhesion. Coating structure and mechanical properties were described in detail in our previous paper [22] and, thus, we will just summarize the results here. XRD analysis of WSC coating showed very broad peak at $2\theta \approx 40^\circ$ with an extended shoulder corresponding to a turbostrating stacking of (10L) planes ($L = 0, 1, 2, 3$), and peak at $2\theta \approx 70^\circ$ indexed as (110) planes. Such spectrum is typical for structure with the lateral order of the basal planes not exceeding a couple of lattice parameters [21]. TEM observation supports XRD results showing randomly oriented separated WS$_2$ platelets embedded into a carbon matrix (Fig. 1). Co-sputtering of Cr with WSC led to completely amorphous structure, see Fig. 1. Surface topography of coatings
measured with AFM is shown in Fig. 2 and corresponding areal surface roughness is given in Table 2. Addition of Cr resulted in the formation of larger columnar structures and correspondingly rougher surface.

XPS was carried out after sputter cleaning of approx. 5 nm of coating material. We identify W-S and W-C bonds together with small fraction of W-O bond. Chromium showed two peaks close to metallic Cr and Cr-O bond; however, binding energy of chromium carbide is very close to that of metal, so we cannot rule out the existence of Cr-C bond. Sharp WS$_2$ peaks observed on Raman spectrum of WSC-Cr0 coating almost disappeared in spectra of chromium-containing coatings, which corroborates TEM observation.

Hardness and reduced elastic modulus of the coatings is given in Table 1. Doping of WSC with chromium led to higher hardness values, since easy shear of WS$_2$ platelets existing in W-S-C film was eliminated due to amorphous nature of WSC-Cr coatings.

3.2 Friction measurements

A large number of published works have indicated that AFM tip can form single asperity contact with the sample surface [2, 3, 6, 7, 9, 10]. The measurements are usually performed in low load regime where tip-sample interaction during frictional sliding is believed to be completely elastic [2, 4]. According to single-asperity theories based on continuum mechanics, friction force in solid-solid nanocontacts is proportional to the true contact area [2-4, 6, 7, 27]. Therefore, the friction force, $F_f$, for single asperity contact is given by:

$$F_f = \tau A$$

where $A$ is the contact area and $\tau$ is the interfacial shear strength, which represents the frictional force per interfacial atom. Typically, single-asperity contact area does not vary
linearly with load. A model for non-adhesive contact developed by Hertz [28], showed that

\[ A \propto F_N^{2/3}. \]

It is in a contrast to the dynamic contact of surfaces at macroscale, where friction

force depends linearly on the normal load, \( F_N, F_f = \mu F_N \), where \( \mu \) is the friction coefficient.

The contact area, \( A \), in the idealized case for non-adhesive junction of a spherical tip on a

perfectly flat surface is given by:

\[ A_{Hertz} = \pi \left( \frac{R}{K} \right)^{2/3} F_N^{2/3} \tag{2} \]

where \( R \) is the radius of the tip and

\[ K = \frac{4}{3} \left( \frac{1-v_1^2}{E_1} + \frac{1-v_2^2}{E_2} \right)^{-1} \tag{3} \]

the effective elastic modulus of the contact, \( v_i \) is Poisson’s ratio, \( E_1 \) and \( E_2 \) are the Young’s

moduli of the sphere and flat surface, respectively.

The simple Hertzian model, which assumes a hemispherical tip shape and neglects adhesive

forces, was successfully applied to systems with very low surface energy and small applied

static loads [3]. In reality, attractive forces between the tip and the sample are present and,

therefore, the effect of adhesion had to be included in subsequent models. If attractive forces

are only present inside the contact area, elastic deformation of the tip and sample are

described by Johnson-Kendell-Roberts (JKR) theory [29]. Derjaguin-Muller-Toporov (DMT)

theory [30] includes attractive forces that act predominantly outside the contact area. DMT

model was successfully applied to the interfaces that involved small tip radii, low adhesion,

and high elastic moduli as demonstrated for diamond-like carbon (DLC) and highly oriented

pyrolitic graphite (HOPG) films [3, 9] or carboxylic acid terminated SAM in polar solvents

[13]. The friction measured between a platinum coated AFM tip and mica surface in UHV [5]

showed a load dependence on the contact area predicted by JKR theory for interfaces
involving large tip radii (> 100 nm), softer materials and high adhesion. However, many interfaces fall somewhere between JKR and DMT limits. Simple JKR-DMT transition theories based on Maugis-Dugale [31] model were proposed by Carpick-Olegtree-Salmeron (COS) [6] and later justified by Schwarz [32] and, eventually, proved experimentally on single crystal diamond [15].

Friction force, $F_F$, as a function of normal load, $F_N$, for the three coatings obtained by LFM is given in Fig. 3. As expected from single asperity theories, a non-linear dependence between $F_F$ and $F_N$ was observed. Additionally, adhesive forces in the range of 13 – 16 nN were recorded from force-displacement curves. Load-dependent results were fitted to DMT-Maugis theory, also known as the Hertz-plus-offset model [3, 32]. In this model, a spherical tip elastically deforms a flat surface, while the additional adhesive forces are indirectly introduced via the increasing value of $A$:

$$A = \pi \left( \frac{R}{K} \right)^{2/3} \left( F_n - F_{off} \right)^{2/3}$$  \hspace{1cm} (4)

where $F_n$ is an effective force acting between the surface and the sphere and is the sum of the applied load and the adhesive forces ($F_n = F_N + F_{ad}$), $F_{off}$ is the constant offset caused by adhesion. Thus the friction force is

$$F_F = \tau A = \tau \pi \left( \frac{R}{K} \right)^{2/3} \left( F_n - F_{off} \right)^{2/3}$$  \hspace{1cm} (5)

It is assumed that $\tau$ is constant, and $F_{off}$ should be lower than the adhesion force, $F_{ad}$ [3]. It was also shown that the actual contact area would be proportional to the load and would depend on the shape of the tip: in Hertzian case the contact area is proportional to $F_n^{2/3}$, while for pyramidal or conical tips to $F_n^{1/2}$ [3, 7]. The dependence contact area versus load can be approximated as $A \sim F_n^m$ with $0 < m < 1$ at low loads (consequence of undefined tip/sample contact). With the additional fitting parameter $m$, the Eq. 5 has been modified to
\[ F_f = C'(F_n - F_{off})^m \]  \hspace{1cm} (6)

where \( C' = \tau \pi (R/K)^{2/3} \) is the constant in units adequate to \( m \). Our data were fitted to Eq. 6, and results are summarised in Table 2. In all cases, the condition \( F_{off} \ll F_{ad} \) was fulfilled. The \( m \) value for coatings without Cr is close to that of Hertzian contact (0.666), whereas the best fit for Cr containing coatings was achieved with lower \( m \) values. The deviation from 2/3 power law is usually observed if the tip apex shows deviation from the spherical shape that might occur due to wear or material transfer from sample to the tip. As referred to experimental section, the increase in tip radii from initial 8 nm up to ~20 nm was observed for all AFM tips that were used in this study. Tip reconstruction using TGT1 calibration standard did not indicate formation of WSC-Cr film on the tip apex; neither tip fracture nor variation in the shape of the tips were observed. Therefore, we expect the increase in tip radii should be attributed to the tip wear. It has been shown by several researchers [33, 34] that standard cone-type Si tip with initial radius of ~10 nm fractured upon first engagement with the hard surface, ultrananocrystaline diamond. The tip became blunter obtaining “semi-constant” radius of about 20 nm during the first 10 mm of scanned distance and remained constant for another 30 - 40 mm, when scanned at zero applied load. Gotsmann and Lantz [35] investigated the atomistic wear of AFM tips on cross-linked polyaryletherketone film within the load region from 5 to 100 nN, for a sliding distance up to 750 m. The authors showed that the wear rate of the tip increased with load and decreased with sliding distance. Overall, the tip wear rate is rather slow and occurs through atom-by-atom loss process. We have checked height images acquired during FFM measurements for any significant bluntness in topographical features. After careful analysis, it was noticed that topography did not vary significantly after scans obtained at loads 4 – 6 nN corresponding to sliding distance 7 – 8 mm. Considering previously published studies [33-35], we can assume that AFM tips reached 20 nm radii during the first 5 – 6 scans, and its shape remained almost constant till the end of
the experiment. As the shape and the radii of the three tips before and after experiments remains similar, the deviation of $m$ parameter from 2/3 power law should be related to different area-load dependence for the tip in contact with Cr-doped coatings as a consequence of their different surface microstructure. Recently it has been shown, that surface geometry, and not the roughness amplitude, influenced the measured friction with AFM [36, 37]. Moreover, structural and chemical changes at the surface of the tip might occur under high pressure [5]. Schwarz et al. [7] obtained different $m$ values for $C_{60}$ and GeS surfaces using the same tip, which was attributed to the difference in the surface layer corrugation.

Contact area was calculated using $A = \pi (RF_N/K)^m$, where $R \approx 20$ nm and $K$ was calculated using Eq.3. As an example, the area and corresponding contact pressure results for loads of 10 nN are given in Table 2. As expected, contact area for WSC-Cr coatings was lower and, correspondingly, contact pressure was larger when compared to WSC-Cr-0 film. Interfacial shear strength, $\tau = C'K'^m/\pi R'^m$, yielded $\approx 71.0$ MPa for WSC–Cr-0, 92.1 MPa for WSC-Cr-7 and 99.3 MPa for WSC-Cr-13 for tip radius $R = 20$ nm.

In our previous studies, we showed that the wear track of WSC films produced by macroscopic sliding (pin-on-disc) was covered with a thin, well-ordered WS$_2$ layer with basal planes oriented parallel to the surface [19, 22]. On the other hand, Cr containing WSC films showed mixture of oxides and WS$_2$ at the sliding interface [22]. Consequently, WSC-Cr coatings exhibited slightly higher friction coefficient. In present study, the wear, although very small, is sufficient to remove surface contamination and oxidation. As an example, coating wear for WSC-Cr-0 and WSC-Cr-7 during LFM measurements is given in Figure 4. Here, overlaid topography images before and after experiments and corresponding line profiles clearly show pile-up formation, and difference in height within 1 x 1 $\mu$m scan. Calculated volume of pile-up matched the removed volume within the scanned region which
was approximately 1.5 \times 10^7 \text{ nm}^3 for WSC-Cr-0, and 1 \times 10^7 \text{ nm}^3 for WSC-Cr-7. The estimated tip loss-volume is \sim 5 \times 10^5 \text{ nm}^3, similar to the reported in Refs. [33-35] and, thus, comparatively small to contribute to the volume of pile-up. As the scope of this paper was to investigate the nanoscale friction properties of WSC-Cr coatings, we did not investigate nanoscale wear of those surfaces in detail, leaving it for later studies. The shear strength for pure WS$_2$ is not known; however, shear strength of MoS$_2$, which should be slightly lower, was measured in the range of 23 – 33 MPa [38] using macroscopic measurement techniques. Wang et al. [18] measured shear stress by AFM-LFM between MoO$_3$ particles and MoS$_2$ surface and reported values 40-940 MPa; the shear stress decreased with increasing particle size. Shear strength reported in present study is close to that of MoS$_2$; considering similar crystal structure and frictional properties of MoS$_2$ and WS$_2$, we can assume that easy-shearing WS$_2$ tribolayer was formed at the coating surface.

Friction coefficient as a function of load is shown in Figure 5. The decrease in friction with increased load is typical for TMD and TMD-based coatings, such as MoS$_x$, WSC, MoSeC [19, 22, 38-40]. The load-dependent behaviour is often approximated by the formula integrating the shear stress of solids at high pressures and the Hertzian model for contact pressure:

\[ \mu = \tau_0 \pi \left( \frac{F}{R} \right)^{2/3} F_N^{-1/3} + \alpha \]  

(7)

where \( \tau_0 \) is the interfacial shear strength and \( \alpha \) is the material constant representing the adhesive forces at zero load. Taking into account that our results of friction force dependence on load deviates from perfect Herzian contact, we have modified Eq. 7 to:

\[ \mu = C'' (F_n - F_{off})^n + \alpha \]  

(8)
By fitting Eq. 8 into our friction coefficient results presented in Fig. 5, it was found that $\alpha$ was approximately zero for all coatings, $n \equiv m-1$, and $C''$ and $F_{off}$ were close to results obtained by fitting data into Eq. 6 (Table 2).

We should point out here that the friction coefficient is typically independent of load or increasing with load. Labuda et al. [41] showed that the friction of Au (111) measured by AFM is almost constant in the load range 0-5 nN, whereas the friction of gold oxide sharply increased. Graphene tested as single sheet or in bilayer form exhibited small increase in friction with increasing load [42]. In fact, decrease in friction coefficient with load was observed mostly for TMD materials and some polymers [43]. The combination results shown in Figure 5 and the shear strength close to MoS$_2$ strongly suggests that AFM sliding experiments forms similar WS$_2$ tribolayer to those observed in the wear tracks produced by macroscopic pin-on-disc testing. Our AFM-LFM study does not show any significant difference between WSC and WSC-Cr frictional behaviour at nanoscale. Although WSC-Cr coatings fail to form pure WS$_2$ tribolayer when sliding against steel counterpart in humid air, they provide such low-friction layer during nanoscale sliding with silicon tip.

Interestingly, macroscopic friction coefficient of WSC coating [19] is remarkable similar to that presented in this work (see inset in Fig. 5). The contact pressure was similar in both cases, and the formation of WS$_2$ tribolayer was demonstrated for pin-on-disc tests by Raman spectroscopy.

4. Summary

To summarize, we presented load-dependent measurements of frictional force for pure and chromium-containing WSC coatings. The data were fitted to “Hertz-plus-offset” model and
the $A \sim F_N^m$ dependence of the contact area on the normal force was observed. The deviation of $m$ parameter from 2/3 power law was related to different area-load dependence for the tip in contact with Cr-doped coatings. The shear strength of all coatings was relatively low, suggesting the formation of an easy-shear layer at the sliding interface. Friction coefficient decreased with increased applied load independently of the chromium content in the coatings. Such behaviour is typical of pure TMD material and could be considered as an indirect proof of formation of an ultrathin low-friction WS$_2$ tribolayer.

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References


Figure captions

Figure 1  TEM observations of coating microstructure. From left WSC-Cr-0, WSC-Cr-7 and WSC-Cr-13. The insets show XRD spectrum (for WSC-Cr-0) and selected area diffraction.

Figure 2  Fig. 2 AFM topography images for three coatings. Image size is 3 x 3 µm, scale bar is 500 nm.

Figure 3  Friction force, $F_F$, as a function of load, $F_N$, for three coatings measured with LFM.

Figure 4  Overlaid 3D topography images of (a) WSC-Cr-0 and (b) WSC-Cr-7 before and after LFM measurements and corresponding line profiles recorded in the middle of the sample.

Figure 5  Friction coefficient as a function of load, for three coatings measured with LFM. Inset, macroscopic friction coefficient of WSC coating, as reported previously in [19].
Fig. 1 TEM observations of coating microstructure. From left WSC-Cr-0, WSC-Cr-7 and WSC-Cr-13. The insets show XRD spectrum (for WSC-Cr-0) and selected area diffraction.

Fig. 2 AFM topography images for three coatings. Image size is 3 x 3 µm, scale bar is 500 nm.
Fig. 3 Friction force, $F_F$, as a function of load, $F_N$, for three coatings measured with LFM.
Fig. 4 Overlaid 3D topography images of (a-b) WSC-Cr-0 and (c-d) WSC-Cr-7 before and after LFM measurements and corresponding line profiles recorded in the middle of the sample.
Fig. 5 Friction coefficient as a function of load, for three coatings measured with LFM. Inset, macroscopic friction coefficient of WSC coating, as reported previously in [19].
Table 1. Properties of the coatings: chemical composition measured by EPMA, hardness and reduced modulus obtained by nanoindentation, and surface roughness from AFM analysis.

<table>
<thead>
<tr>
<th></th>
<th>Chemical composition (at. %)</th>
<th>Hardness, $H$ [GPa]</th>
<th>Reduced modulus, $E_r$ [GPa]</th>
<th>Areal surface roughness, $S_a$ [nm]</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>W</td>
<td>S</td>
<td>C</td>
<td>Cr</td>
</tr>
<tr>
<td>WSC–Cr-0</td>
<td>23.6</td>
<td>29.8</td>
<td>42.00</td>
<td>0.6</td>
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<tr>
<td>WSC–Cr-7</td>
<td>20.3</td>
<td>25.5</td>
<td>40.4</td>
<td>7.0</td>
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<tr>
<td>WSC–Cr-13</td>
<td>18.5</td>
<td>24.1</td>
<td>35.8</td>
<td>13.5</td>
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</tbody>
</table>
Table 2. Relevant results for measurements done on WSC/Cr coatings. Contact area, contact pressure and shear strength are calculated for tip radius of 20 nm.

<table>
<thead>
<tr>
<th></th>
<th>WSC – Cr - 0</th>
<th>WSC – Cr - 7</th>
<th>WSC – Cr - 15</th>
</tr>
</thead>
<tbody>
<tr>
<td>$C'$ [nN$^{(1-m)}$] (Eq.6)</td>
<td>0.095 ± 0.009</td>
<td>0.114 ± 0.004</td>
<td>0.136 ± 0.007</td>
</tr>
<tr>
<td>$C''$ [nN$^{(1-n)}$] (Eq.8)</td>
<td>0.093 ± 0.031</td>
<td>0.115 ± 0.003</td>
<td>0.128 ± 0.178</td>
</tr>
<tr>
<td>Offset force $F_{off}$ [nN] (Eq. 6)</td>
<td>1.481 ± 0.255</td>
<td>0.804 ± 0.037</td>
<td>1.210 ± 0.30</td>
</tr>
<tr>
<td>Offset force $F_{off}$ [nN] (Eq. 8)</td>
<td>1.394 ± 0.55</td>
<td>0.608 ± 0.165</td>
<td>1.040 ± 2.52</td>
</tr>
<tr>
<td>Fitting parameter $m$ (Eq.6)</td>
<td>0.654 ± 0.022</td>
<td>0.604 ± 0.008</td>
<td>0.504 ± 0.012</td>
</tr>
<tr>
<td>Fitting parameter $n$ (Eq.8)</td>
<td>-0.33 ± 0.001</td>
<td>-0.391 ± 0.01</td>
<td>-0.489 ± 0.012</td>
</tr>
<tr>
<td>$\tau$ [MPa] (Eq.6)</td>
<td>71.0 ± 3</td>
<td>92.1 ± 2</td>
<td>99.3 ± 2</td>
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<tr>
<td>$\tau_0$ [MPa] (Eq.8)</td>
<td>70.6 ± 3</td>
<td>93.4 ± 2</td>
<td>94.4 ± 2</td>
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<tr>
<td>$K$ [GPa]</td>
<td>73 ± 4.4</td>
<td>93 ± 5.0</td>
<td>98 ± 6.0</td>
</tr>
<tr>
<td>$A$ [nm$^2$] at $F_N = 10$ nN</td>
<td>≈ 6.06</td>
<td>≈ 4.98</td>
<td>≈ 4.38</td>
</tr>
<tr>
<td>Contact pressure [GPa] at $F_N = 10$ nN</td>
<td>≈ 1.65</td>
<td>≈ 2.01</td>
<td>≈ 2.29</td>
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