



# Effects of surface nanocrystallization on tribological properties of 316L stainless steel under MoDTC/ZDDP lubrications

Yanyan Wang<sup>a</sup>, Wen Yue<sup>a,\*</sup>, Dingshun She<sup>a</sup>, Zhiqiang Fu<sup>a</sup>, Haipeng Huang<sup>b</sup>, Jiajun Liu<sup>c</sup>

<sup>a</sup> School of Engineering and Technology, China University of Geosciences (Beijing), Beijing 100083, PR China

<sup>b</sup> SINOPEC Lubricant Company (Beijing), Beijing 100085, PR China

<sup>c</sup> Mechanical Engineering Department, Tsinghua University, Beijing 100084, PR China

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## ABSTRACT

In this paper, 316L stainless steel (316LSS) is treated by a ultrasonic cold forging technology (UCFT) which is one of the methods of surface nanocrystallization. The tribological properties of UCFT sample are investigated by a SRV tribometer under lubrications with MoDTC and ZDDP. The chemical analysis of tribofilms is performed using X-ray photoelectron spectroscopy (XPS). The results show that the UCFT sample exhibits the lowest coefficient of friction (0.066) under MoDTC lubrication and the lowest wear rate under ZDDP lubrication, indicating that surface nanocrystallization has synergistic effects on low friction and antiwear performances of both MoDTC and ZDDP. The obviously synergistic effects between UCFT steel and MoDTC/ZDDP may be attributed to the higher hardness and surface reactivity of the UCFT surface.

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## 1. Introduction

Surface nanocrystallization is recognized as a useful method applied in the industrial field to enhance the mechanical properties of materials without changing the chemical compositions and the shape [1]. Surface nanocrystallized steel exhibits significant improvements in friction and wear properties [2–6]. It can be attributed to the surface layer with nanograins and a gradient variation in the microstructure and properties along the depth from top surface [7].

It is well known that the tribological properties of a rubbing-pair are mainly dominated by the lubrication and mechanical properties, while mechanical components usually suffer severe wear in the boundary (or mixed) lubrication regime. Many studies have focused on the tribological properties of the surface nanocrystallized steel under oil lubrication. Sun [8] reported that the nanocrystallized 304 stainless steel provided more resistance to plastic deformation and better wear resistance under lubrication due to its much higher hardness. Zhang et al. [9] reported that the nanocrystallized Cu sample exhibited an excellent load-bearing ability and a remarkably enhanced wear resistance in fretting tests under oil-lubricated condition. Amanov et al. [10] proposed that the micro-dimples promoting retention of a thin oil film

improved the tribological properties of the nanocrystallized steel. Lv et al. [11] suggested that the higher reactivity of nanocrystalline grains resulted in the improvement of friction and wear properties of the nanocrystalline iron ingot under liquid paraffin lubrication, especially when ZDDP was contained in liquid paraffin. Therefore, surface nanocrystallization may have positive effects on tribochemical reactions with lubricant additives, whereas little work has been carried out.

Molybdenum dithiocarbamate (MoDTC) and zinc dialkyldithiophosphate (ZDDP) are the most commonly used friction modifier and antiwear additive in commercial fully formulated engine oils. MoDTC can reduce friction by forming a MoS<sub>2</sub>-containing film on the tribological contact [12,13], while ZDDP can reduce wear and scuffing by forming a polyphosphate tribofilm [14,15]. The microstructures and the tribological behaviors of MoDTC and ZDDP are affected by the chemical and mechanical properties of the rubbing-pairs, such as the nitrided and the sulfur-nitrided layer on metallic materials and diamond-like carbon films [16–19]. However, the synergistic/antagonistic effects between MoDTC/ZDDP and surface nanocrystallized steel have not been reported yet.

316L austenitic stainless steel is a typical material for the piston ring in engine due to its excellent properties in corrosion and oxidation resistance. However, the low hardness and poor tribological properties of the steel can shorten the life of the component subject to wear. In this paper, the effects of UCFT treatment on the tribological properties of 316L stainless steel under MoDTC/ZDDP lubrications are investigated. It will be helpful

\* Corresponding author. Tel.: +86 10 82320255; fax: +86 10 82322624.

E-mail addresses: [cugbyw@163.com](mailto:cugbyw@163.com), [yw@cugb.edu.cn](mailto:yw@cugb.edu.cn) (W. Yue).

**Table 1**  
UCFT treatment parameters.

Vibration frequency (kHz)	Amplitude (μm)	Load (N)	Spindle speed (rpm)	Feed rate (mm rev <sup>-1</sup> )	Tip (tungsten carbide) diameter (mm)	Number of shots per (mm <sup>2</sup> )
20	20	300	200	0.025	10	96,000

to further recognize the tribochemical reactions between nanocrystallized steel and additives, promoting a wide application of surface nanocrystallization in engineering fields.

## 2. Experimental details

### 2.1. Materials

The material used in this work was 316L stainless steel plate of 3 mm thick, its chemical compositions (wt%) are 0.019 C, 17.07 Cr, 11.95 Ni, 2.04 Mo, 1.68 Mn, 0.35 Si, 1.14 Cu, 0.007 S. The surface roughness of the steel was 4 nm and the initial structure was an fcc austenite with grain sizes in the range of 10–60 μm. The plate was machined into samples of 60 × 60 mm<sup>2</sup> for UCFT treatment.

The main concept and mechanism of UCFT is as follows: A tungsten carbide ball is attached to an ultrasonic device and it strikes the surface of a workpiece at a frequency of 20 kHz. These strikes cause severe plastic deformation on the surface layer and induce a nanocrystal structure. The system was demonstrated in the literature [20] and the parameters used in this work are shown in Table 1. The samples were ultrasonically cleaned for 20 min by immersing in acetone.

### 2.2. Tribotests

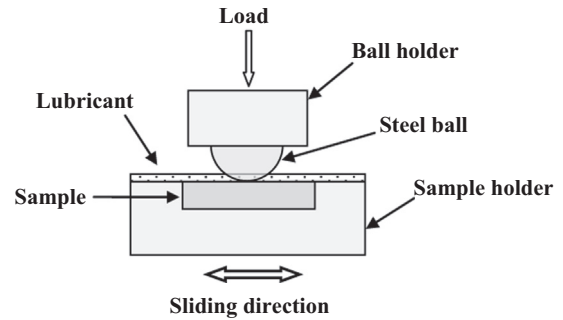
The base oil was synthetic oil Polyalphaolefin (PAO4) of viscosity 16.68 mm<sup>2</sup>/s at 40 °C and 3.84 mm<sup>2</sup>/s at 100 °C. MoDTC was added in the base oil as the friction modifier and the alkyl chains are C<sub>8</sub> (2-ethylhexyl) and C<sub>13</sub>. The main chemical compositions (mass fraction) of MoDTC are Mo 10.0% and S 11.0%. The antiwear additive was a primary ZDDP with the alkyl chain C<sub>16</sub>, and its main chemical compositions (mass fraction) are Zn 10.0%, P 8.0% and S 16.0%. The concentrations of MoDTC and ZDDP added in the base oil were both 1.0 wt%. The density of all lubricants was almost the same (0.85 g/ml).

The sliding wear tests were conducted on an Optimol SRV reciprocating tribometer. During the test, the untreated or UCFT sample (12 × 20 mm<sup>2</sup>) was stationary, while an AISI 52100 steel ball reciprocated on the sample. Fig. 1 shows the schematic diagram of the contact obtained in SRV reciprocating apparatus and the test condition is given in Table 2. The lubricants were dropped on the samples before testing, and then the samples were covered by the oil. The test began when the temperature in the chamber has reached 100 °C and it was repeated for three times. The wear samples were degreased by immersing several times in acetone with ultrasonic cleaning before other tests.

The minimum film thickness was calculated using Dowson equation [21]:

$$h_{min} = 3.63R(U^*)^{0.68} \cdot (W^*)^{-0.073} \cdot (G^*)^{0.49} \cdot (1 - e^{-0.68k}) \quad (1)$$

where  $U^* = \eta_0 U / E^* R$ ,  $G^* = \alpha / E^*$ ,  $W^* = P / E^* R^2$ , and  $\eta_0$  is the dynamic viscosity ( $3.26 \times 10^{-3}$  Pa·s at 100 °C),  $\alpha$  is the viscosity–pressure coefficient ( $3.53 \times 10^{-8}$  Pa<sup>-1</sup> at 100 °C),  $R$  is the radius of the ball (5 mm),  $U$  is the average linear speed (0.02 m/s),  $P$  is the normal load (20 N),  $E^*$  is the effective modulus of elasticity (222.5 GPa)



**Fig. 1.** Schematic of SRV reciprocating apparatus.

calculated according to the Young's modulus of the ball (193 GPa) and the sample (213 GPa), and  $k$  is an elliptical parameter, which is equal to 1.03 for point contact mode. The calculated  $h_{min}$  value was about 1.86 nm.

The lambda ratio ( $\lambda$ ) was calculated using the equation:

$$\lambda = h_{min} / (R_{a1}^2 + R_{a2}^2)^{1/2} \quad (2)$$

where  $R_{a1}$  and  $R_{a2}$  are the roughnesses of the ball and the sample, respectively. The calculated  $\lambda$  values were about 0.07 and 0.06 for the untreated sample and the UCFT sample, respectively. The result suggested that the tests were operated in the boundary lubrication regime.

A NanoMap-D 3D surface profilometer was used to measure wear tracks on the samples. The wear rates were used to evaluate the wear performance of various oils. It was calculated using the Archard wear equation:

$$V = k \cdot F \cdot s \quad (3)$$

where  $V$  is the wear volume,  $F$  is the normal load,  $s$  is the sliding distance, and  $k$  is the wear rate per unit load and per unit distance.

The wear rate of the counterpart ball can also be calculated using the Archard wear equation, while the wear volume of the ball  $V_{ball}$  was calculated by measuring the radius of the wear scar in the optical microscope and using the equation:

$$V_{ball} = \pi h [3r^2 + h^2] / 6 \quad (4)$$

where  $h = R - (R^2 - r^2)^{1/2}$ ,  $R$  is the radius of the ball (10 mm),  $r$  is the radius of the wear scar measured (m),  $V_{ball}$  is the wear volume of the counterpart ball (m<sup>3</sup>), and  $h$  is the height of the sphere of the ball worn after the wear test.

### 2.3. Microanalysis

X-ray diffraction (XRD) analysis was carried out on a D/max-2500 X-ray diffractometer with Cu K $\alpha$  radiation to obtain the average grain size and phase identification in the modified layer.  $2\theta$  ranges from 40° to 120° and the scanning speed was 5 °/min. The hardness variation along the depth was measured on a cross-sectional sample by a MH-6 microhardness tester, and the load (50 g) lasted for 5 s. Surface topographies and roughnesses were investigated by a NanoMap-D 3D surface profilometer. After etching in the 50 vol% HCl + 25 vol% HNO<sub>3</sub> + 25 vol% H<sub>2</sub>O etchant, the cross-sectional morphologies of the samples were investigated by a BX51M Olympus optical microscope (OM) and a NTEGRA atomic force microscopy (AFM). A NSG10 silicon probe with 125 μm long cantilever was used and the typical resonance frequency was 240 kHz. The surface morphologies of the test samples were investigated by JSM-7001F scanning electron microscopy (SEM).

Chemical analysis of tribofilms was performed by PHI Quantera X-ray photoelectron spectroscopy (XPS). The instrument utilized a high-power rotating anode and monochromatised X-ray of Al K $\alpha$

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