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### Phase transformation studies on the a-C coating under repetitive impacts

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#### ABSTRACT

The phase transformation of hydrogen-free amorphous carbon (a-C) coating on tungsten high speed steel (SKH2) substrates under repetitive impact testing has been studied. The a-C coated disc was impacted by the chromium molybdenum steel (SCM420) pin at several different impact loads and impact cycles (up to 100,000) under lubricated conditions. The results show that the  $sp^3$  fractions of impacted a-C coating obtained from the surface of impact craters are significantly increased with impact cycles due to decreasing ID/IG ratio. This means that the amorphization of a-C coating also increased after several impact cycles. As for the full-width at half maximum (FWHM) of G peak characterization, it is shown that the hardness of impacted a-C coating is higher than the as-received. From the observation of surface roughness using atomic force microscopy (AFM), it is supposed that increasing sp<sup>3</sup> fractions and the hardness of the impacted a-C coating during impact correlate to the reduction of surface roughness. In addition, the tribochemical reaction to the environment during impact occurred at the mating material, where the transfer layer adhered, as well as in the wear debris. This is due to the oxidation of ferrum (Fe) to magnetite (Fe<sub>3</sub>O<sub>4</sub>) and hematite ( $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>) phases with predominant peak at about 680 cm<sup>-1</sup> and 1317 cm<sup>-1</sup>, respectively. The formation of Fe<sub>3</sub>O<sub>4</sub> and  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> phases was revealed from Raman spectroscopy and the existence of oxide elements was verified by energy dispersive X-ray spectroscopic (EDS) analysis. Increasing the G peak position, together with a concomitant decrease of their width, it is believed that the structural transformation from sp<sup>3</sup> to sp<sup>2</sup> is taking place within the wear debris and leads to the graphitization process at a higher contact pressure. It was suggested that the high contact pressure is not just only corresponding to the applied normal impact load, but it is also exerted by an oil lubricant during impact. A high contact pressure can significantly reduce the graphitization temperature and substantially accelerate the graphitization process. However, a significant phase transformation of the transfer layer on the SCM420 pin does not intensely occur because it is mainly coming from the surface layer of the impacted a-C coating, where the sp<sup>3</sup> content increases and no wear debris is observed inside it.

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#### 1. Introduction

Hydrogen-free amorphous carbon (a-C), commonly known as diamond-like carbon (DLC) has attracted great attention for many applications due to its tremendous properties, such as high hardness, thermal stability, low friction coefficient and good chemical inertness. Furthermore, the a-C film showed an excellent wear resistance in dry, water- and oil-lubricated conditions [1]. The a-C is a disordered mixture of carbon atoms with sp<sup>2</sup> and sp<sup>3</sup> hybridizations [2]. The sp<sup>3</sup> hybrids confer diamond-like properties like high hardness, high density, chemical inertness, etc., while sp<sup>2</sup> hybrids control the

electronic and optical properties because the  $\pi$  states lie closest to the Fermi level [3].

The phase transformation of DLC due to sliding has been performed extensively for a decade [4-10]. However, there is still a lack of information about how the structure will change by repetitive impacts.

J.X. Liao et al. [6] reported that as the number of the sliding cycles or the load is increasing, the tribological properties decrease due to the graphitization of DLC films within a wear track. Y. Liu et al. [7] showed with evidence that the transfer layer contained a fine distribution of graphite nano-particles in a distorted diamond-like structure. Besides, a graphitization process took place within the wear track region of the coatings probably due to thermal and strain effects from the repeated friction. A year later, Y. Liu and E.I. Meletis [8] said that the transformation of DLC to graphite-like may also be facilitated by shear stresses existing in the surface layer. However, Z.F. Zhou et al.

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Fig. 1. Schematic illustration of a repeated impact tester.

[9] suggested that the structural transformation of DLC coatings within the wear tracks was mainly due to the formation of a compact wear debris layer rather than a frictional heating effect. In addition, according to J.C. Sanchez-Lopez et al. [10], evidence of extended graphite layer formation was not observed in the transfer film of DLC after friction test.

In this study, some other degradation also can exist on the a-C coating during repetitive impacts if the normal impact load is high enough, such as the propagation of cracks inside the impact craters and may result in the occurrence of cohesive failure. Moreover, no adhesive failures of the a-C coating are observed. However, the discussion about this degradation is beyond the scope of this paper. The objective of this paper is to discuss the changes in the structure of the a-C coating by applying different normal impact loads under repetitive impacts. The structure of the a-C coating is studied by means of Raman spectroscopy, EDS, AFM and field emission scanning electron microscopy (FE-SEM).

#### 2. Experimental

The disc specimens were coated with a-C by a physical vapour deposition (PVD) method. The measured hardness and thickness of the coating are approximately 25.13 GPa and 2.97 µm, respectively. The SKH2 disc was used as a substrate. The impact test was performed using a self-developed impact tester.

The impact test rig, as shown in Fig. 1, consisted of a load detector together with a cam that was designed to impact a-C coated disc with a SCM420 pin, with a radius of 1 mm under numerous impacts. The disc was repetitively impacted with a 90° inclination at room temperature. Kerosene was used as a lubricant. Prior to the impact test, both disc and pin were cleaned using acetone in an ultrasonic bath. The load was applied to the disc specimen via a spring system and was observed by the load detector. In this experiment, the frequency of the impacts was selected at 10 Hz. The dimensions of both disc and pin are shown in Fig. 2.

The bonding structures of the wear debris and impact crater surfaces of the a-C coated disc, as well as the transfer layer on the pin, were studied using Raman spectroscopy in addition to EDS, AFM and FE-SEM observations. The Raman spectra were measured at room



Dimension in mm

Fig. 2. Dimensions of the a-C coated disc and SCM420 pin.



Fig. 3. Raman spectra of the as-received a-C coated disc and after impacted at 100,000 impact cycles under different impact loads.

temperature and were acquired over the range of 500–2000  $\rm cm^{-1}$  at 1.1  $\rm cm^{-1}$  resolution.

#### 3. Results and discussion

Raman spectroscopy is the best way to obtain the detailed bonding structures of the DLC films [10]. The Raman spectra shown in Fig. 3, which were taken from the surface layer of the impact craters, exhibit



Fig. 4. Variation of (a) Raman intensity ratio ID/IG and (b) FWHM with impact cycles.

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