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# Experimental study on friction and wear behaviour of amorphous carbon coatings for mechanical seals in cryogenic environment

Jianlei Wang<sup>a</sup>, Qian Jia<sup>a</sup>, Xiaoyang Yuan<sup>a,\*</sup>, Shaopeng Wang<sup>b</sup>

- a Key Laboratory of Education Ministry for Modern Design and Rotor-Bearing System, Department of Mechanical Engineering, Xi'an Jiaotong University, Xi'an 710049, China
- <sup>b</sup> Xi'an Aerospace Propulsion Institute, China Aerospace Science and Technology Corporation, Xi'an 710100, China

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#### ABSTRACT

The service life and the reliability of contact mechanical seal are directly affected by the wear of seal pairs (rotor vs. stator), especially under the cryogenic environment in liquid rocket engine turbopumps. Because of the lower friction and wear rate, amorphous carbon (a-C) coatings are the promising protective coatings of the seal pairs for contact mechanical seal. In this paper, a-C coatings were deposited on 9Cr18 by pulsed DC magnetron sputtering. The tribological performances of the specimen were tested under three sealed fluid conditions (air, water and liquid nitrogen). The results show that the coatings could endure the cryogenic temperature while the friction coefficients decrease with the increased contact load. Under the same contact condition, the friction coefficient of the a-C coatings in liquid nitrogen is higher than that in water and that they are in air. The friction coefficients of the a-C coatings in liquid nitrogen range from 0.10 to 0.15. In the cryogenic environment, the coatings remain their low specific wear rates  $(0.9 \times 10^{-6} \text{ to } 1.8 \times 10^{-6} \text{ mm}^3 \text{ N}^{-1} \text{ m}^{-1})$ . The results provide an important reference for designing a water lubricated bearing or a contact mechanical seal under the cryogenic environment that is both reliable and has longevity.

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#### 1. Introduction

The contact mechanical seal pairs in turbopumps are now employing the hard–soft pattern, commonly using stainless steel as the rotor ring and the graphite as the stator ring. However, it was found that the pairs showed serious wear during the running stage [1,2]. The low temperature of the cryogenic propellants (such as liquid oxygen, liquid hydrogen) in the highly efficient liquid rocket engines affected the mechanical seals. Many recent research efforts have demonstrated that the performances of the seals are greatly determined by the friction between the seal pairs and the lubricating capability of the fluid. Meanwhile, many seals operate under mixed lubrication consisting of the dry friction and fluid lubrication [3–6].

Besides the improvement in turbopump require better mechanical seals. These mechanical seals have been developed because of the increasing demands for seal pairs with less leakage, lower friction coefficient and lower wear rate. Therefore, protective coatings deposited on the pairs' surface can effectively seal the pair and diminish the friction and wear of the contact seal rings [7,8]. As one of the protective coatings, amorphous carbon (a-C) coating has

potential applications for the protection of cryogenic mechanical seal. Much research efforts have demonstrated that the a-C coatings have excellent mechanical and tribological properties [9–12]. Hence, the a-C coatings are widely used in bearings, seals and MEMS. Mechanical seals, coated with a-C, are also mentioned for their potential applications [13–16]. Much concern has been reported regarding friction and wear of a-C coatings in water and air [17–20]. However, as far as we know, there are few studies of friction and wear behaviour of mechanical seals, coated with a-C, at cryogen [21]. In this study, we deposit the a-C coatings on 9Cr18 by pulsed direct current (DC) magnetron sputtering and explore their suitability for cryogenic mechanical seal applications.

#### 2. Experimental details

#### 2.1. Coating preparation and characterization

Before being introduced into the deposition chamber, mirror polished 9Cr18 alloy  $(15\,\mathrm{mm}\times15\,\mathrm{mm}\times2\,\mathrm{mm})$  were washed by ultrasonically waves sequentially in acetone, alcohol and distilled water for 5 min, then dried in cool air. Pulsed DC magnetron sputtering system with one pair of ultra-pure graphite targets was used to deposit the amorphous carbon (a-C) coatings on the mirror polished 9Cr18. Oxide film and contamination on the surface were removed by Ar+ washing for 20 min at a bias of  $-800\,\mathrm{V}$  before

<sup>\*</sup> Corresponding author. E-mail address: jlwang@stu.xjtu.edu.cn (X. Yuan).

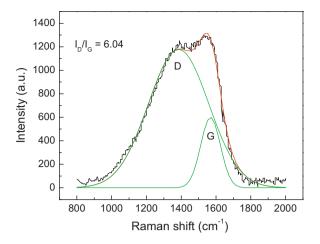
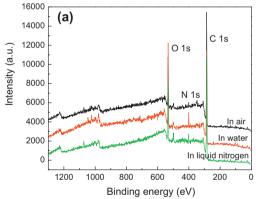


Fig. 1. Raman spectrum of a-C coatings.

sputtering. Afterwards, a Ti interlayer was deposited between the substrates and the a-C coatings to enhance the adhesion strength. And the detailed deposition parameters are as follows: Pulsed DC bias of -100 V, duty factor of 60%, chamber pressure of 0.7 Pa in a gas of Ar, Ti target power of 3kW and deposition time of 10 min. The deposition parameters of the a-C coatings were the same as that of Ti interlayer but the deposition time was

Raman spectroscopy (Almega, Nicolet) was used to characterize the microstructure of carbon coatings. Raman spectrum was excited at room temperature using 532 nm argon ion laser and the spectrum in the range of 900-1800 cm<sup>-1</sup> was fitted using a Gaussian distribution peak.



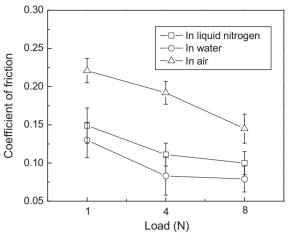


Fig. 2. Friction coefficients of a-C coatings in different lubricants and under different applied load.

#### 2.2. Tribological tests

We studied the tribological characteristics of the a-C coatings by using a ball-on-disk tribometer. The friction media were air (relative humidity 35-45%), water and liquid nitrogen, respectively. The counterpart was 9Cr18 ball with the diameter of 3 mm and the rotational frequency of the samples underneath the counterpart was 10 Hz. Three forces (1 N, 4 N and 8 N) were loaded in each friction medium for 30 min each. To evaluate the chemical composition of wear tracks, X-ray photoelectron spectroscopy (XPS, Axis Ultra, Kratos, UK) with monochromatic Al Kα radiation (150 W, 15 kV, 1486.6 eV) is used. The survey spectra are obtained at constant pass energy of 160 eV and the high-resolution spectra were recorded

(a) C	(b)	Test condition	C(at.%)	N(at.%)	O(at.%)
		In air	79.73	3.22	17.06
N 1s	In air	In water	69.30	5.36	24.49
	In water	In liquid nitrogen	75.56	6.32	18.12
Rinding energy (eV)	-	•			

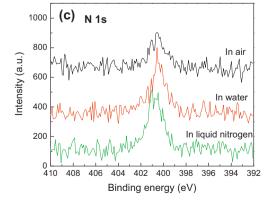


Fig. 3. XPS analysis results of wear tracks under different lubricants: (a) survey spectra; (b) chemical composition; (c) high resolution spectra of N 1s.

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