







# Onset of nanoscale wear of metallic implant materials: Influence of surface residual stresses and contact loads

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#### **Abstract**

Nanoscale wear response of cobalt—chromium alloy (ASTM F-75) was investigated as a function of contact load and surface residual stress in order to identify mechanism governing onset of surface damage in modular implants. A unique loading configuration was utilized to apply range of known in-plane stress states to the specimen surface in order to simulate different residual stress levels. Using the tip of a scanning force microscope as a well characterized "asperity," controlled contact forces were applied on the specimen to mechanically stimulate the loaded surface. Volume of material removed was measured to characterize the wear rate as a function of the contact loads and surface stress state. Experimental measurements of material removal indicate that a critical level of contact pressure is required to initiate wear of the cobalt-chromium surface and as expected higher contact pressures accelerate the wear process. At a constant contact pressure, wear rate is accelerated by compressive in-plane stress while tensile in-plane stresses tend to suppress the surface wear. A surface damage mechanism based on successive damage/delamination of native oxide covered surface due to single asperity contact and repassivation of exposed surface is proposed to elucidate the experimental observations.

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#### 1. Introduction

Fretting contact between surfaces of artificial joints results in formation of particulate and soluble metallic debris that can migrate locally or systemically and may induce a cascade of inflammatory events that may ultimately result in bone loss by osteolysis and subsequent implant failure. While a large number of studies have been undertaken to experimentally characterize wear rates of joint materials, the influence of surface parameters like residual stress, surface finish etc on the mechanisms governing damage nucleation is still not clearly understood. In the current study, the influence of residual stress and contact stresses on nucleation of nanoscale surface damage during fretting contact of an implant material is investigated.

In a total hip replacement there are two primary bearing interfaces: interface between the acetabular cup and femoral head; and interface between the femoral stem and femoral head. Fretting contact at the interface between the femoral stem and head leads to generation of soluble and particulate metallic debris [1–4]. Fretting is simply defined as small scale slipping between two contacting surface under normal and tangential loads. It decreases the fatigue life of components and in the case of biomedical implants generates soluble and particulate debris. Metallic implant materials commonly thought to have high biocompatibility (Ti alloys, Co alloys, stainless steels) have come under scrutiny primarily due to the release of debris that embed themselves into surrounding tissue. Independent work by Catelas et al. [2], Doorn et al. [3], and Fisher et al. [4] have revealed that generated debris particles are typically tens of nanometers in diameter; and in the case of cobalt-chromium alloy, chromium oxide debris were the dominant type found in surrounding tissue [2,3]. It is these small particles that Hallab and Jacob [1] identify as the cause of three body wear, osteolysis, and toxicities. In order to understand the debris generation it is necessary to understand the mechanism that governs damage nucleation and subsequent wear at the material interface.

Previous work on understanding the wear mechanisms of biomaterials has run the gamut from scratch testing to commercially available hip simulators. Catelas et al. [2] used a commercially available hip simulator to perform long term testing on different

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cobalt-chromium alloy combinations. Wear particles were collected periodically and characterized to determine the wear rate. It was determined that the initial run in period occurred under 500,000 cycles and at that point all the alloys begin to undergo steady state wear. Most interestingly, wear debris were primarily chromium oxide with complete absence of cobalt. The experimental observation supports an earlier work by Doorn [3] that discovered primarily chromium traces in surrounding tissue from retrieved hip implants. Miloslev and Strebhlow [5] conducted experiments using X-ray photoelectron spectroscopy (XPS) and determined that native oxide on cobalt-chromium alloy is primarily chromium oxide and is approximately 1.8 nm thick. Furthermore, Goldberg and Gilbert [6] determined that when the passive layer is fractured, exposed metallic surface are highly reactive and repassivate through reformation of native oxide layer with in milliseconds. Using a modified equation for repassivation kinetics the researchers determined thickness of reformed native oxide to be approximately 1.8 nm, which is in excellent agreement with experimental observations of Milosev and Strehblow [5]. In addition, it was noted that approximately 6.4 GPa of nominal contact stress was required to elicit repassivation response during scratch experiment suggesting that native oxide layer will not fracture at lower loading levels during a single scratch loading. In summary, previous studies suggests that under fretting loads chromium oxide passive layer is fractured and then rapidly repassivates in milliseconds with initial film failures occurring early in the life of the implant. Work by Catelas et al. [2] suggests that rapassivated film is primarily of the same composition since only chromium oxide debris were found throughout the fretting test duration (two million cycles). Contradictorily, Hallab et al. [7] measured a larger ratio of cobalt-chromium from a serum solution surrounding a custom built hip simulator. This discrepancy is probably due to the method of extracting the oxide debris. A 0.2 μm filter was used to extract the wear particles, which may be too large to capture a significant number of chromium oxide debris (approximately 30–50 nm in size [2–4]).

Investigations of fretting wear are not unique to the biomaterials community. Fretting has been identified as a damage mechanism in turbine blades, ball bearings, as well as biomedical joint replacements. A popular method to reduce fretting wear is to apply a surface treatment to the material but studies incorporating surface treatments have shown mixed results on remediation of fretting damage. Hendry and Pilliar [8] examined fretting response of Ti-alloy flats coated with TiN against a cobalt-chromium alloy counterface. The researchers concluded that hardened TiN coating reduced fretting damage of specimens but chromium debris were noted on the TiN coating after the test. Indicating that hardened TiN coating accelerated fretting wear of the cobalt-chromium counterface compared to tests conducted against uncoated specimens. Another study conducted to determine the ideal coating parameters found that compressive residual stress generally mitigated fretting fatigue damage [9]. In contrast, Morbacher et al. [10] indicated that TiN coatings on tool steel did not affect the fretting rate during the life of the coating. After delamination of coating, the wear rate of exposed substrate was higher than that of uncoated surface. Shot peening is another surface treatment method that has been explored to remediate the fretting fatigue damage through development of compressive surface residual stresses. But one troubling issue with shot peening is that tensile stresses are developed within the material to compensate for the compressive stress layer at the surface. To further complicate matters, research has demonstrated that the surface residual stresses relax upon removal of plastically deformed surface layers [11,12].

While several studies have explored the influence of surface treatments and associated residual stress on fretting wear, a complete understanding of underlying damage mechanisms is still elusive. Residual stress and real area of contact are two parameters that are very difficult to control in traditional fretting test apparatus. The goal of this study is to overcome the limitations imposed by traditional long term testing fixtures with a novel experimental setup. In the setup a four-point bending frame and an atomic force microscope are used to apply well characterized in-plane stresses and contact stresses to the specimen surface. The experiments are designed to investigate the onset of fretting damage due to a single asperity (the AFM probe) oscillating over a surface subjected to a controlled stress state. The materials, four-point bending frame, and experimental setup are discussed in following section. Nanoscale wear response of cobalt-chromium alloy (CoCrMo) under varying contact loads and in-plane stresses will be presented and the implications of experimental results for macro scale fretting damage will be discussed.

### 2. Materials and experimental set-up

#### 2.1. Material and microstructure

The material used in this study is a cast cobalt-chromium (CoCrMo) alloy as specified by ASTM F75. Mechanical properties of cast CoCrMo alloy are presented in Table 1 [13]. CoCrMo samples were cut from canine femoral stem prosthesis by electric discharge machining to form rectangular bars  $(1 \text{ mm} \times 2 \text{ mm} \times 25 \text{ mm})$ . Electric discharge machining was chosen to minimize processing induced residual stress on the machined surfaces. The 1 mm thickness of the beam was then mechanically polished by 1200 grit, 3 µm diamond suspension, 1 μm diamond suspension, and finally with a 0.05 μm colloidal silica suspension to a mirror finish. AFM measurements on the polished side indicate that the initial surface follows a Gaussian distribution with skewness approximately 0 (-0.1 to 0.08) and kurtosis of approximately 3 (2.8–3.2). The root mean square (RMS) roughness ranges from 0.5 to 2 nm on 10 µm scans, indicating a smooth initial surface.

Table 1 Mechanical properties of CoCrMo specimen and  $Si_3N_4$  AFM tip

Property	CoCrMo (F-75)	Si <sub>3</sub> N <sub>4</sub> AFM probe
Young's modulus	230 GPa	310 GPa
Poisson's ratio	0.33	0.33
Yield strength 0.2% offset	450 MPa	_

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