

Corrosion–erosion of TiN-PVD coatings in collagen and cellulose meat casing

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Abstract

The meat casing industry works with highly fibrous materials and low pH conditions. The pumps propelling the gel must therefore be highly corrosion–erosion resistant. In fact, the most common failure in this medium is corrosion, erosion or, most commonly, a combination of both. Coatings are thus required to protect the pump surface and maintain the operational conditions. TiN-PVD coatings seemed to be an attractive alternative amongst the range of options to extend pump lifetime. However, despite the greater chemical inertia expected of TiN, the combined effect of corrosion and erosion can induce a fast attack on the material. The present paper analyses failure of a TiN coating with a single-layer configuration in contact with an edible slurry of collagen used in the meat casing industry. The degradation mechanism comprises two different steps. Firstly, the acidic components reach the base steel through the pores, promoting severe dissolution of the steel substrate and leaving the TiN coating without support. Secondly, the coating detaches under working conditions and merges with the casing solution. Finally, the fibres together with the hard particles erode the pump walls, leading to fast deterioration.

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1. Introduction

Traditionally, natural casings are extracted from the digestive tract of edible animals, primarily from the submucosa, a largely collagen layer of the intestine [1]. However, natural casing is more expensive than the artificial alternative, and hence is primarily used for the most expensive sausages and cold meats. Artificial meat casings can be manufactured from various raw materials [2], most of them rich in collagen and cellulose. Their main advantages are high resistance (natural casings are prone to break) and easy manufacturing (producing uniform size and shapes). Basically, the casing consists of collagen and cellulose fibres that form an acid slurry. Subsequently, this gel is extruded through a mixer and acquires integrity after chemical treatment.

During the process, the casing is propelled by different lobe pumps exposed to a highly aggressive solution. The collagen

and cellulose slurry is usually kept in gel form at pH between 1 and 3 prior to handling.

The lobe pumps commonly used in the food industry are generally made from stainless steel (mainly AISI 316) because of its high corrosion resistance. However, the materials used in the meat casing industry are both highly fibrous and low pH, requiring high corrosion–erosion resistance in these pumps.

In fact, the most important causes of damage to these pumps are corrosion, erosion in the form of abrasion or, most commonly, a combination of both.

Physical vapour deposition (PVD) has aroused considerable great interest in recent years since it permits the deposition of dense, compact coatings, leading to improved chemical and mechanical properties. The technique permits the production of a wide range of hard coatings that are extensively used in manufacturing because of their high wear and chemical resistance, relatively low cost and easy coating procedure.

Since the commercial success of TiC-coated hard metal tools in the late 1960s, the interest in hard coatings has steadily increased. In the class of hard coatings, which range from

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diamond-like carbon films to transition metal carbide and nitride coatings, titanium nitride films are at present the most widely studied and used. Nowadays, they are grown either by chemical or physical vapour deposition methods (CVD and PVD) [3].

Early TiN coatings were typically 5–10 μm thick and were obtained by CVD at temperatures of about 1273 K, atmospheric pressure and titanium tetrachloride (TiCl_4) and nitrogen (N_2) as the precursors to Ti and N respectively. Later, PVD methods permitted the growth of TiN films at temperatures below 550 $^\circ\text{C}$, enabling their use in High Speed Steel (HSS) tooling to avoid softening. Coatings with thicknesses of about 2–8 μm are generally found to increase the tool lifetime by several hundred per cent. Recently, Iwai et al. [4] showed that TiN deposited at 500 $^\circ\text{C}$ had high wear resistance (solid particle impact test or so-called slurry jet) although it had a relatively low hardness.

Many other applications have arisen including machining (stainless steels, cast irons, and aluminium alloys) and protection dies, moulds, punches and a range of metal stamping and forming tools [5–7]. Thus, PVD-TiN coatings are an interesting alternative to stainless steel to be used in the pumps.

The present paper analyses the failure of mechanical components due to the combined effect of erosion and corrosion of a TiN coating in contact with an edible slurry of collagen used in the meat casing industry.

2. Experimental procedure

In the casing, composed of collagen with up to 6% solids (collagen plus 30% cellulose), average fibre length was 300 μm . These fibres are highly abrasive. The slurry had pH 2 and 7 cP viscosity. The operating conditions of the pumping were in the range of 400–1500 ml/min, at 20 and 78 rpm rotation speed.

The composition of the pump, made of AISI M2 steel, was determined by Glow Discharge Optical Emission Spectroscopy exhibiting a surface composition of C 0.97%, Si 0.2%, Mn 0.2%, Cr 4.11%, W 6.3%, V 1.8%, Mo 4.9% and Fe (balance), with TiN coating. Titanium nitride coatings were grown by cathodic arc PVD technique on mirror polished and hardened AISI M2 steel. The substrate temperature was kept below 450 $^\circ\text{C}$, with 160 min deposition time and 0.5 nm/s evaporation rate. These conditions yielded uniform, homogeneous TiN layers, apparently defect free with a thickness of up to 5 μm .

After 600 h of operation, the pump evidenced severe damage as shown in Fig. 1. In order to determine the causes of the degradation, specimens from the flat surfaces of the pump were used to study the corrosion and wear behaviour.

The coating microstructures were examined with a field emission gun scanning electron microscopy microscope (FEG-SEM) JEOL 6500F and analysed with a Link-Inca Energy Dispersive Spectrometer (EDS). Secondary electron images were taken.

X-ray diffraction (XRD) analysis of the coatings was carried out in order to identify their phase compositions. Cu K_α radiation with a goniometer in a Bragg–Brentano configuration was used in the XRD analysis.

Electrochemical tests were carried out using a conventional three-electrode cell consisting of a saturated calomel reference electrode (SCE), a platinum counter electrode and the studied specimens as working electrode. The electrolyte was HCl 0.1 N, simulating the casing pH. Prior to testing, specimens were cleaned with acetone in an ultrasonic bath for 5 min followed by a further 5 min in isopropanol.

A scan rate of 0.16 mV s^{-1} was used for the polarization curves. The potential scan began after a stabilization period of 15 min. For the potentiodynamic curves, a potential step in the cathodic direction was applied up to -0.3 V with respect to the open circuit corrosion potential. The scan started beyond this value in the anodic direction until it reached breakdown potential or a threshold intensity level of 0.2 mA/cm^2 . Once this value was reached, the reverse cycle was started.

Sliding wear tests were performed in two different environments, ambient air of $\text{RH} < 40\%$ and 0.1 N HCl to simulate operational conditions. The contact geometry used was a ball-on-disk configuration. A 3 mm diameter corundum ball was used as counterbody material because of its high wear resistance, chemical inertness and electrical insulating properties. Specimens and corundum counterbodies were cleaned with ethanol prior to testing. A normal load of 1 N was applied, with 3 Hz rotation speed (track radius = 2 mm), corresponding to a linear speed of 0.04 m/s, and sliding distance varied between 250 and 3000 m. All experiments were performed at room temperature.

Electrochemical noise (EN) measurements were conducted during corrosion–wear tests in the acidic solution. The experimental setup is shown schematically in Fig. 2. A zero resistance ammeter (ACM Instruments)-ZRA-, was used to record voltage and current variations with a resolution of 10 μV and 100 pA, respectively. TiN specimens used as working

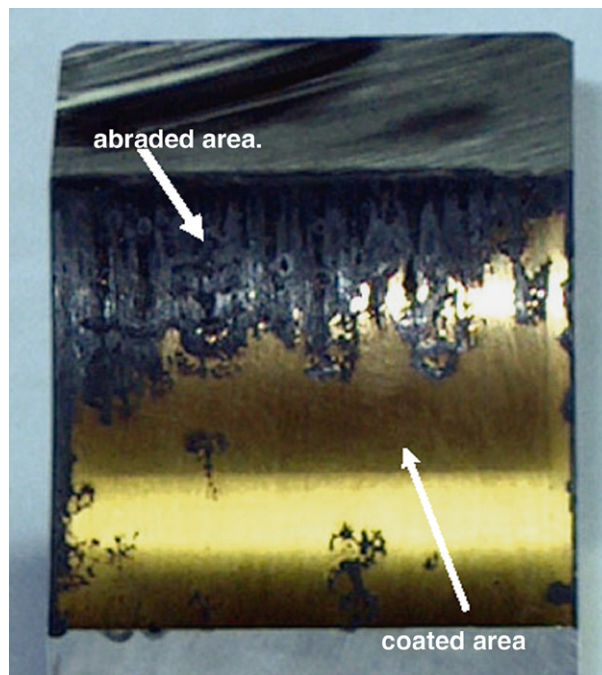


Fig. 1. Severe pump damage after 600 h of operation.

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