



Tribology and wear resistance of the stainless steel. The sol–gel coating impact on the friction and damage



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ABSTRACT

The sol–gel coating method is considered to be simple, easy to adopt, requires low processing temperature and leads to high degree of purity oxide coatings. Moreover, the fabrication of metal oxide thin films by sol–gel technic is cost effective, reproducible and applicable to large substrates without any shape restriction. This work focuses on optimizing conditions for depositing alumina coatings by sol–gel method on stainless steel substrates with a low annealing temperature, in order not to degrade the metal substrate. Moreover, to obtain coatings of several micron thick, the sol–gel was adapted by adding ceramic powder or by successive layers deposition which reduces the stresses occurring in the coatings during the heat treatment. The various as-obtained layers are characterized in terms of morphological features, microstructure, hardness and the tribological behavior and wear resistance.

The results showed that the deposited alumina coating at low temperature crystallized in δ - Al_2O_3 . Adding to that, the increase of the temperature favors the presence of α - Al_2O_3 structure. Moreover, the measurements demonstrate that the mechanical properties of sol–gel deposited alumina coatings in terms of hardness, wear resistance are improved in comparison with uncoated stainless steel or the classic nitrited steel.

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1. Introduction

Degradation by wear surfaces is one of the most current industrial phenomena, apart from fatigue and corrosion, which cause replacement of parts in the engineering industry. This phenomenon is often disastrous, but it results in an increase in losses by degradation of the surface of opposing pieces. The choice of material for the production of parts is often a complex issue because it has to meet many requirements that are sometimes contradictory. Among the various properties that the designer must pay attention, there are (but not limited to): the mechanical properties, it is mass properties (hardness, resilience, yield strength, fatigue resistance, etc.) that will condition the dimensioning pieces; the cost of raw material is obviously a criterion of prime importance; ease of implementation (shaping, machining, etc.) is also an important criterion in economic terms; the

availability of the material (or its components) determines the supply and can have a significant impact on cost; durability (corrosion resistance, wear etc.) is also an important economic argument; the appearance is probably the least important criterion except in some special cases (jewelry etc.). In the great majority of cases, notably in mechanical engineering, it is the mass mechanical properties which are essential for the selection argument that is performed. However, the same material can meet all required specifications. As regards the appearance and durability, it is then necessary to use special surface treatment. To meet the reliability requirements in these areas that have been developed treatments or coatings that increase the longevity of parts. The thin films methodology exhibit a different technics and process like: physico-chemical and electrochemical which aim to adapt the surface to the intended use. In this study we will focus on the surface treatment process developed by the sol–gel (SG) technology in a stainless steel [1–3]. In fact, the sol–gel technology that has enjoyed success in the last 20 years, for deposition of hard thin films, good corrosion resistant ceramics (e.g. SiO_2 , ZrO_2 , Al_2O_3) or functional ceramics for sensors, membranes, thermal barrier etc. [3,4]. The SG technology often fails if the film is thicker than about

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1 μm , because of the damaging shrinkage strains during drying and densification [4]. Moreover, the sol–gel technic seems to be advanced to other process since being simple, cheap, low-temperature and not requiring vacuum [5]. This SG method enables preparation of good purity and highly homogeneous materials that form the coating one or more constituents in the form of mono-or multilayers. It was also found that thin coated film prepared by the sol–gel approach exhibit better adhesion to substrate compared to the CVD and PVD techniques [6]. Dry sliding wear and some abrasion experiments on sol–gel coated were reported in other researches. Hardness alumina layers, the major problem which limits their use in mechanical applications is its low resistance for cracking under stress. In spite of its hardness, alumina coating is very brittle [7–10]. It is known that the mechanical properties of the materials may be enhanced if the grain size reaches the nano-metrics range. The mechanical properties of the layer are at the center of interest of numerous studies including alumina layers [11–16]. Moreover, alumina is also doped with nanoparticles creating nano-composites in order to improve its mechanical properties. The result show that materials having the presence of a mono-disperse phase also improves the tribological properties of the material [5]. On another side, stainless steel is used as a structural material in various industries thanks to its good corrosion resistance and high mechanical properties [16]. However, it is quite prone to pitting attack in chloride acid environments. Considering the above reasons, the life of this stainless steel is limited while performing in such environments. Therefore, a surface treatment seems to be necessary to protect it [17–19].

The purpose of this work is to enhance hard sol–gel coatings onto stainless substrates to improve their behavior as wear. The chosen method is the hydrolysis and poly-condensation of organometallic precursor (to form a gel) and the dip-coating process (to deposit the matured gel on the stainless steel). All the processing parameters are controlled: the gel viscosity, the dipping speed, the grain size of alumina powder added to the gel and the conditions to disperse it in order to amplify the thickness of the coating. After the deposition, the drying and thermal treatment conditions will be optimized to ensure the layer adhesion to the stainless steel and the presence of $\alpha\text{-Al}_2\text{O}_3$ (and/or) $\delta\text{-Al}_2\text{O}_3$ phase, which exhibits the best behavior in terms of friction and wear properties.

2. Experimental techniques

2.1. Specimens

Stainless steel is rarely used for shaping mechanical parts. Its performances (good workpiece surface quality after the formatting process, a long life of tools) are requested for special shapes too [20]. The layer developed by the sol–gel process has a good compromise between performance and cost. Before the sol–gel process, all substrate surfaces are prepared by soft polishing, so they have the same surface quality. The so polished surface (approx. 0.3 μm , Ra) is cleaned by ethanol and acetone meaning ultrasounds. The hardness of the AISI304 stainless steel is about 420 HV_{25 g}, and the chemical composition is illustrated in the following Table 1. Moreover, AISI H11 nitrated steel (classic steel

Table 1

Nominal composition of stainless steel AISI304 mass examined in wt%.

Material	Mn	Si	Cr	Ni	Fe
AISI304	2	1	18–20	8–10.5	Balance

Table 2

Nominal composition of steel AISI4820.

Material	C	Si	Mn	Cr
AISI4820	0.2162	0.25	1.2	1.2

used for formatting) is also tested for a comparative study with coated and uncoated stainless steel AISI304. Thereby, to test this steel in wear, a circular samples steel is cut into pieces of 30 mm of diameter, and 5 mm of thickness. The chemical composition of this material is mentioned in the following Table 2.

2.2. Coatings synthesis

The sol–gel coating has proven important results concerning the corrosion resistance by modification of the chemical nature of the interface and the preservation of the coated tool to a temperature of 1000 °C [21]. In the present work, a sol–gel coating alumina is developed by gelling. Alumina is selected because of its high hardness and was successfully used as powder charge sol–gel alumina coating.

A standard experimental protocol is held for the elaboration of the alumina gel [21]. The alumina isopropoxide precursor Al (OiPr)₃, 98% (Aldrich) is dissolved in water with 0.01 M at watery solution in the presence of the acid HNO₃. This solution is heated at 85 °C under magnetic agitation in a confined environment during 24 h. The viscosity of the gel is controlled after maturation: $\eta = 10 \text{ mPa s}$. For the mixed $\alpha\text{-Al}_2\text{O}_3$ powder/ $\gamma\text{-Al}_2\text{O}_3$ gels, the powder of alumina (CT 3000SG-Alcan) is introduced into the gel with a way ratio of 20%. The gel loaded with the powder is maintained under agitation during 24 h at room temperature (25 °C). To make sure of the good dispersion of the powder within the gel, the mixture is treated by ultrasounds during 120 seconds. Laser particle size analyzer follows the impact of the powder dispersion. The gels are deposited on the substrate by using a dip-coater. Each substrate is dipped into the solution at a constant speed of 60 mm/mn, and then withdrawn at approximately the same speed. The coated substrates are then annealed at 500 °C or 700 °C for 1 h at a heating rate of °/mn. Composite sol–gel coatings were prepared by spraying this mixture onto the stainless steel substrates, followed by drying and heating at different degrees of temperature to convert the sol to a hydrated nanostructured γ or/and α -alumina gel phases. The process of deposition, drying and heat-treatment were repeated twice to prepare a multilayer coating.

All the coated samples, The hardness value measured at load increases from 425 Hv_{25g} for AISI304 stainless steel surface up to 780 Hv_{25 g} for γ -alumina sol–gel coated surface (corresponding to approximately +85%).

2.3. Samples characterization

In this analysis, a microstructural scanning electron microscope (SEM) equipped with an energy spectroscopy X (EDX) light dispersion is employed. The identification of the film phase formed by the sol–gel on the stainless steel was studied using XRD technique (simens D-500 system) with the CuK α radiation. SEM is also used to identify and analyze the waste and debris trails formed during wear tests.

2.4. Wear tests

The study of the wear and tribology behavior is developed by a pin-on-disc tribometer (MT/10/SCM from Microtest) without lubrication in room temperature, at a humidity of about 31%. The counter-body was a 6 mm diameter of 100Cr steel ball. The tests

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