Vacuum 156 (2018) 135–139

Contents lists available at ScienceDirect

Vacuum

journal homepage: www.elsevier.com/locate/vacuum

Short communication

Influence of low temperature plasma oxynitriding on the mechanical behavior of NiTi shape memory alloys

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ARTICLE INFO

Keywords:
NiTi alloy
Glow discharge treatments
Structure
Mechanical properties
Industrial applications

ABSTRACT

NiTi shape memory alloy was modified at low temperature plasma under glow discharge conditions in order to produce a $TiO_2 + TiN$ type diffusive surface layer. The paper presents the structure, chemical composition, surface topography and mechanical properties, such as hardness, reduced modulus and friction coefficient of NiTi alloy after proposed surface treatment in comparison to initial state. The oxynitriding process slightly increased the surface roughness and both hardness and elastic modulus. The friction coefficient was lowered independently of the friction path and load. Such results are promising in the context of increasing the wear resistance of NiTi alloy and designing its new industrial applications.

1. Short communication

NiTi shape memory alloys belong to the group of intelligent materials, which are finding increasingly more applications in various industries such as medicine, as bone or cardiac implants [1–3] and in microelectromechanical systems (MEMS) [1–5] as micro-grippers [1], fluid flow valves [4], actuators and sensors [6]. In terms of such applications the mechanical properties and wear resistance of these materials, as well as the relationships existing between them, are of key importance. The literature indicates that the wear resistance of various materials increases mostly with hardness [7]. To achieve the best results, various methods of surface engineering, including plasma treatment, CVD and PVD methods (deposition of hard coatings), electrochemical and electroless chemical processes are used [8–11]. In the present work, the results of nanoindentation and friction coefficient investigations are reported for NiTi shape memory alloy subjected to low-temperature oxynitriding under glow-discharge conditions carried out in two stages in one technological cycle. Glow-discharge nitriding can be carried out at low temperatures, i.e. up to 300 °C, thanks to cathodic sputtering phenomenon and the high chemical affinity of titanium to atomic nitrogen and oxygen present in low-temperature plasma. What is more, low temperature make it possible to maintain the unique properties of NiTi alloys, since at higher temperatures, precipitation of the Ni_4Ti_3 phase is observed, which can affect shape memory and superelasticity [12]. The aim of this work is to present the structure, phase composition of the produced oxynitrided surface layer and its impact on surface topography, hardness, reduced modulus and the friction coefficient.

The tested material was NiTi shape memory alloy (50,8%at. Ni). The samples measuring ϕ8x1mm were mechanically ground with SiC papers to 2400 grit and degreased with acetone. The layers were formed using glow-discharge oxynitriding processes at low temperature plasma in two steps: nitriding in an atmosphere of pure nitrogen (99.999%) for 30 min and oxidizing in an atmosphere of air for 15 min. Both stages of the process were conducted at a temperature of 290 °C and a working chamber pressure of 1.6 hPa. During heat-up, immediately before nitriding, the samples were subjected to cathodic sputtering in a gaseous mixture of argon and nitrogen (at a volume ratio of 3:1) for 10 min at a pressure of 0.3 hPa. The temperature was controlled by a thermocouple.

The microstructural characterization of the layer was carried out on the cross sections of the specimens using an FEI TECNAI Super TWIN (200 kV) FEG transmission electron microscope operated at 300 kV and equipped with an EDS system. The samples used in the HRSTEM tests were prepared using the Focused Ion Beam (FIB) Lift-out technique and an Ion Scanning Microscope [13]. The linear distribution of the

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https://doi.org/10.1016/j.vacuum.2018.07.027
Received 21 May 2018; Received in revised form 4 July 2018; Accepted 19 July 2018
Available online 20 July 2018
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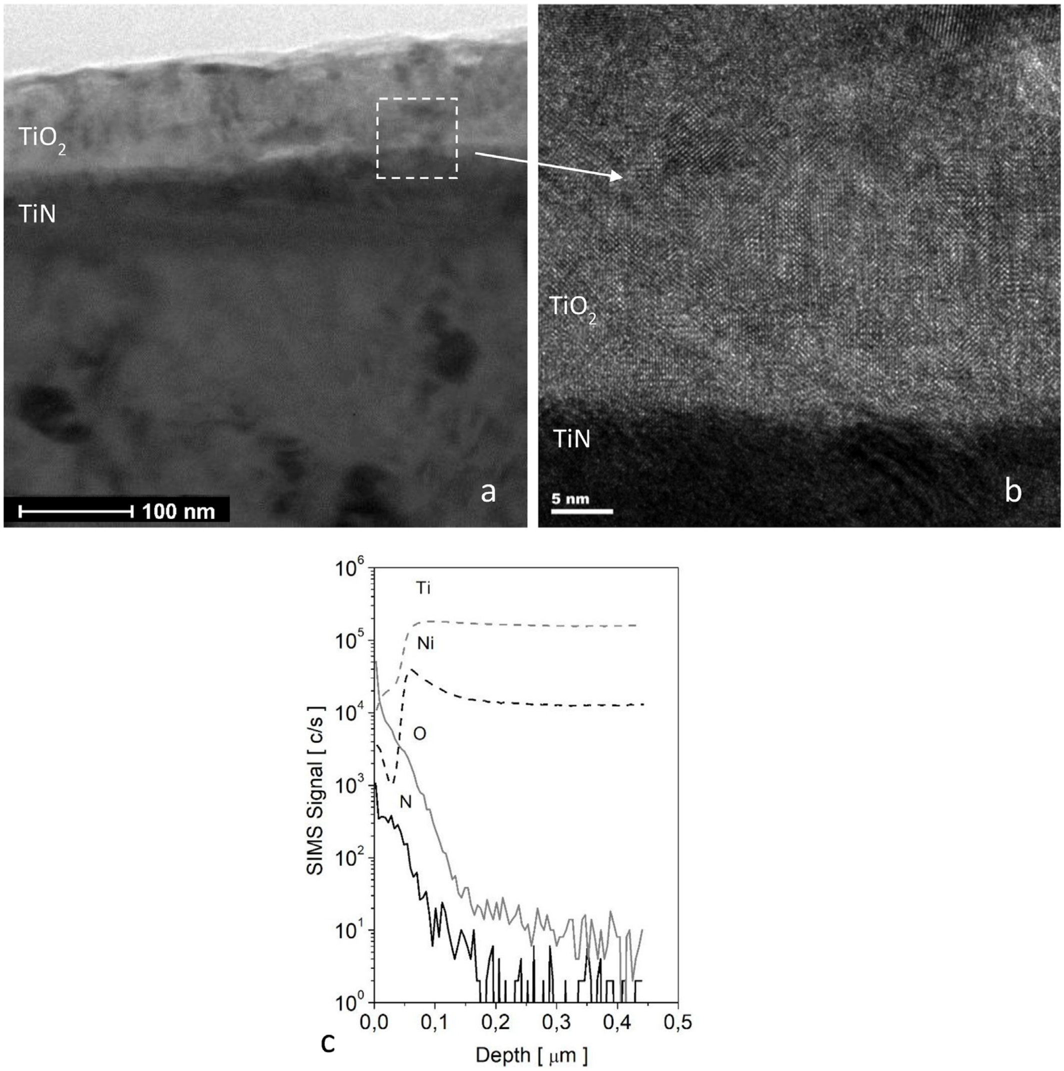

Fig. 1. Structure of a surface oxynitrided layer produced on NiTi substrate in low temperature plasma: cross section of the layer (STEM image) (a), HRSTEM image of the surface layer (b) and linear distribution (c) of oxygen, nitrogen, titanium and nickel in the surface layer.

Table 1
Surface roughness measurement of NiTi alloy in initial state and after glow-discharge oxynitriding carried out using a scanning optical profilometer.

	R_a [μm]	R_q [μm]	R_t [μm]
NiTi alloy initial state	0,012	0021	1977
NiTi alloy after oxynitriding	0,041	0054	2283

R_a - roughness average; R_q - root mean square roughness; R_z - vertical distance between the maximum profile peak height and the maximum profile valley depth along the evaluation length.

elements in the surface layers was measured by secondary ion mass spectrometry (SIMS) analysis (Cameca IMS6F). Ionic beam etching with an 800 eV Ar + ion beam was carried out.

The surface topography of the NiTi in initial state and with an oxynitrided layer was measured by means of a WYKO NT 9300 scanning optical profilometer and was also observed using a scanning electron microscope (SEM) (Hitachi, S-3500 N) with an acceleration voltage of 15 kV in secondary electron (SE) mode.

In order to determine hardness and the elastic modulus as a function of depth, nanoindentation studies were carried out using a system consisting of NanoTest Vantage (Micro materials) with a Berkovich indenter. For data analysis, the method of Oliver & Pharr was used [14]. All measurements were performed with a controlled load in the range from 0.03 mN to 1 mN. For the depth profile, the data from the experimental ranges was divided into intervals for averaging and presented as a graph with error bars. The testing technique used combines conventional Oliver and Pharr unloading curve analysis with rapid acquisition of test data.

Nanotribological property tests were carried out on the CSM nanotribometer on polished samples in the initial state and with the oxynitrided layer. The studies were done with loads of 20 mN, 50 mN, 100 mN and a sliding distance of 20 m or 100 m, with a ball made of Al_2O_3 as a counterbody (with a diameter of 2 mm and a hardness of 75 HRC).

TEM cross-section observations show that a composite surface layer TiO_2 + TiN type layer is produced on NiTi alloy (Fig. 1a). The outer zone was composed of titanium oxide – TiO_2- and below titanium nitride – TiN - with a thickness of about 50 nm and 40 nm, respectively. According to HRSTEM observations (Fig. 1b) the layers have a nanocrystalline structure and as we demonstrated in the earlier studies produced titanium oxide is rutile [15,16]. There is almost no nickel in the outer zone what is confirmed by the distribution of elements (Fig. 1c).

The glow-discharge oxynitriding process slightly increased the surface roughness of the polished NiTi alloy (Table 1, Fig. 2), which

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