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WEAR RESISTANCE OF STEEL DESIGNED FOR SURGICAL INSTRUMENTS AFTER HEAT AND SURFACE TREATMENTS

ODPORNOŚĆ NA ZUŻYCIE ŚCIERNE STALI PRZEZNACZONEJ NA NARZĘDZIA CHIRURGICZNE PO OBRÓBKACH CIEPLNYCH I POWIERZCHNIOWYCH

The paper presents results of studies on wear resistance of martensitic steel designed for surgical instruments. The steel was examined in as-delivered state, after heat treatment, after surface treatment and after sterilisation. The heat treatment consisted of quenching by compressed nitrogen from 1050°C (1323K) after austenitising during 20 min (1.2 ks), then the steel was tempered for two hours (7.2 ks) at temperatures of 300°C (573K) and 620°C (893K). The nitriding was carried out at T = 460°C (733K) and pressure p = 150 Pa during t = 20 h (72 ks). A reactive atmosphere consisting of a 25% N_2 and 75% H_2 mixture was used to carry out the plasma nitriding. The sterilisation by steam was carried out in an autoclave at T = 134°C (407K) and pressure p = 0.21 MPa (2 atm.) during 30 min (1.8 ks). The abrasive wear of X39Cr13 steel was described basing on such quantities as the mass decrement and mutual displacement of frictional pair. Most favourable results of wear resistance were obtained for high-temperature tempered and nitrided steel and for high-temperature tempered, nitrided and sterilised steel.

W pracy przedstawiono wyniki badań odporności na zużycie ścierne stali martenzytycznej przeznaczonej na narzędzia chirurgiczne. Badano stal w stanie dostawy, po obróbce cieplnej, powierzchniowej oraz po zabiegu sterylizacji. Badaną stal poddano obróbce cieplnej, która polegała na hartowaniu sprężonym azotem z temperatury 1050°C (1323K) po austenityzowaniu w czasie 20min (1,2ks), następnie stal odpuszczano przez dwie godziny (7,2ks) w temperaturach 300°C (573K) i 620°C (893K). Azotowanie przeprowadzono w temperaturze T=460°C (733K) przy ciśnieniu p=150Pa i w czasie t=20h (72ks). Do realizacji procesu azotowania jarzeniowego zastosowano atmosferę reaktywną składającą się z mieszaniny 25%N_2 i 75%H_2. Sterylizację parą wodną przeprowadzono w autoklawie w temperaturze T=134°C (407K) przy ciśnieniu p=0,21MPa (2atm) i czasie 30min (1,8ks). Na podstawie takich wartości jak ubytek masy oraz wzajemne przemieszczenie pary trącej scharakteryzowano zużycie ścierne stali X39Cr13. Najkorzystniejsze wyniki odporności na zużycie ścierne uzyskano dla stali wysoko odpuszczonej i azotowanej oraz dla stali wysoko odpuszczonej, azotowanej i sterylizowanej.

1. Introduction

The wear of materials occurs during operation and corrosive-erosive influence mainly on the surface, therefore the development of new surface engineering technologies creates broad possibilities to manufacture products of required properties based on the existing materials. The use of surface engineering techniques enables forming the microstructure, phase and chemical composition, state of internal stresses in surface layers of processed materials (metallic, ceramic, polymer), so forming their practical properties, such as: corrosion resistance, resistance to friction and erosive wear, resistance to oxidation at elevated temperatures, increase in fatigue strength as well as biocompatibility with the environment of tissues and body fluids [1-4].

Specific conditions existing in a human body create very strict criteria both for metallic materials used for implants and surgical instruments as well as for layers created on their surfaces. Good biocompatibility, high adhesion to the basic metal, and corrosion resistance are only some of requirements allowing applying specific material in medicine. Components of medical instruments must meet requirements of functionality and safety; in the case of multiple use of the instruments, their sterilisation is also important. Appropriate selection of sterilisation parameters allows replacing disposable instruments with multiuse equipment [5, 6].

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2. Experimental methodology

Martensitic steel, classified as stainless steel, grade X39Cr13 acc. to EN 10088-1:1995 standard [7] (4H13 acc. to PN-71/H-86020), was selected for testing. The material was delivered in the form of soft annealed sheet (1 mm thick); the chemical composition of the tested material is presented in Table 1.

TABLE 1

Chemical composition of steel selected for tests, wt%

acc. to	C	Si	Mn	P	S	Cr
analysis	0.42	0.39	0.55	0.020	0.004	13.73
EN 10088-1	0.36-0.42	max. 1.00	max. 1.00	max. 0.04	max. 0.015	12.50-14.50

Specimens were subject to heat treatment, consisting of quenching with compressed nitrogen from the austenitisation temperature of 1050°C (1323K), the time of holding at this temperature was 20 minutes (1.2 ks). After quenching the steel was tempered for two hours at 300°C (573K) – low-temperature tempering and at

TABLE 2

Parameters of treatments performed

Specimen No	Parameters of consecutive treatment stages			
	Quenching	Tempering	Nitriding	Sterilisation
a	X	X	X	X
b	• T=1050°C (1323K) • t = 20 min (1.2 ks) • cooling with compressed nitrogen	• T=300°C (573K) • t = 2 h (7.2 ks) • cooling with compressed nitrogen	X	X
c	• T = 1050°C (1323 K) • t = 20 min (1.2 ks) • cooling with compressed nitrogen	• T = 300°C (573 k) • t = 2 h (7.2 ks) • cooling with compressed nitrogen	X	• T = 134°C (407 K) • t = 30 min (1.8 ks) • p = 0.21 MPa
d	• T = 1050°C (1323 K) • t = 20 min (1.2 ks) • cooling with compressed nitrogen	• T = 300°C (573 K) • t = 2 h (7.2 ks) • cooling with compressed nitrogen	• T = 460°C (733 K) • t = 20 h (72 ks) • p = 150 Pa Reactive atmosphere: 25%N_2 + 75%H_2	X
e	• T = 1050°C (1323 K) t = 20 min (1.2 ks) • cooling with compressed nitrogen	• T = 300°C (573 K) • t = 2 h (7.2 ks) • cooling with compressed nitrogen	• T = 460°C (733 K) • t = 20 h (72 ks) • p = 150 Pa Reactive atmosphere: 25%N_2 + 75%H_2	• T = 134°C (407 K) • t = 30 min (1.8 ks) • p = 0.21 MPa
f	• T = 1050°C (1323 K) • t = 20 min (1.2 ks) • cooling with compressed nitrogen	• T = 620°C (893 K) • t = 2 h (7.2 ks) • cooling with compressed nitrogen	• T = 460°C (733 K) • t = 20 h (72 ks) • p = 150 Pa Reactive atmosphere: 25%N_2 + 75%H_2	X
g	• T = 1050°C (1323 K) • t = 20 min (1.2 ks) • cooling with compressed nitrogen	• T = 620°C (893 K) • t = 2 h (7.2 ks) • cooling with compressed nitrogen	• T = 460°C (733 K) • t = 20 h (72 ks) • p = 150 Pa Reactive atmosphere: 25%N_2 + 75%H_2	• T = 134°C (470 K) • t = 30 min (1.8 ks) • p = 0.21 MPa

620°C (873K) – high-temperature tempering followed by cooling with compressed nitrogen. Specimens after low- and high-temperature tempering were additionally subject to surface treatment (plasma nitriding). The nitriding was carried out in JON-600 type installation for ion treatment with cooled anode. Specimens of martensitic steel selected for tests were subject to nitriding; they were placed on the cathode, where specimens' surface was bombarded with ions of energies resulting from the cathode fall value [8-10]. Before nitriding the specimens were ground and then polished. The nitriding was carried out at T = 460○C (733K) and pressure p = 145 Pa during t = 20 h (72 ks). 25%N_2 + 75%H_2 was used as reactive atmosphere. Part of specimens was sterilised. The sterilisation by steam was carried out in an autoclave at T = 134°C (407K) and pressure p = 0.21 MPa (2 atm.), during time t = 30 min (1.8 ks) in four cycles. Specimens' designations and parameters of treatments carried out are presented in Table 2.

Wear resistance tests were carried out in conditions of dry friction on a T-05 tester with an arrangement of "roller – block" rubbing pairs. Because of too small thickness of the sheet, the specimens subject to abrasive wear were placed on a specially prepared plate (block), which corresponded to dimensions of specimens used in this method (Fig. 1). T-05 tester operates in tandem with Spider 8 control module and is controlled by Catman Expres 3.0 software, which enables recording of the contact load (N), the amount of wear – depth of the wear track (μm) and temperature changes (°C) versus time. Counter-specimens (rollers, 35 mm in diameter) were made of bearing steel, with hardness of 62 HRC. Wear resistance tests were carried out under constant load of 52.95 N during 9 ks for the as-delivered and heat treated material. However, for nitrided specimens this time was elongated to 18 ks. The rotational speed of the counter-specimen was 560 rpm (9.33 rps). The mass decrements of specimens weighed with accuracy of 0.00001 g after specific period of wear, was the basic magnitude adopted in the tests. The rotational speed used was 560 rpm (9.33 rps).

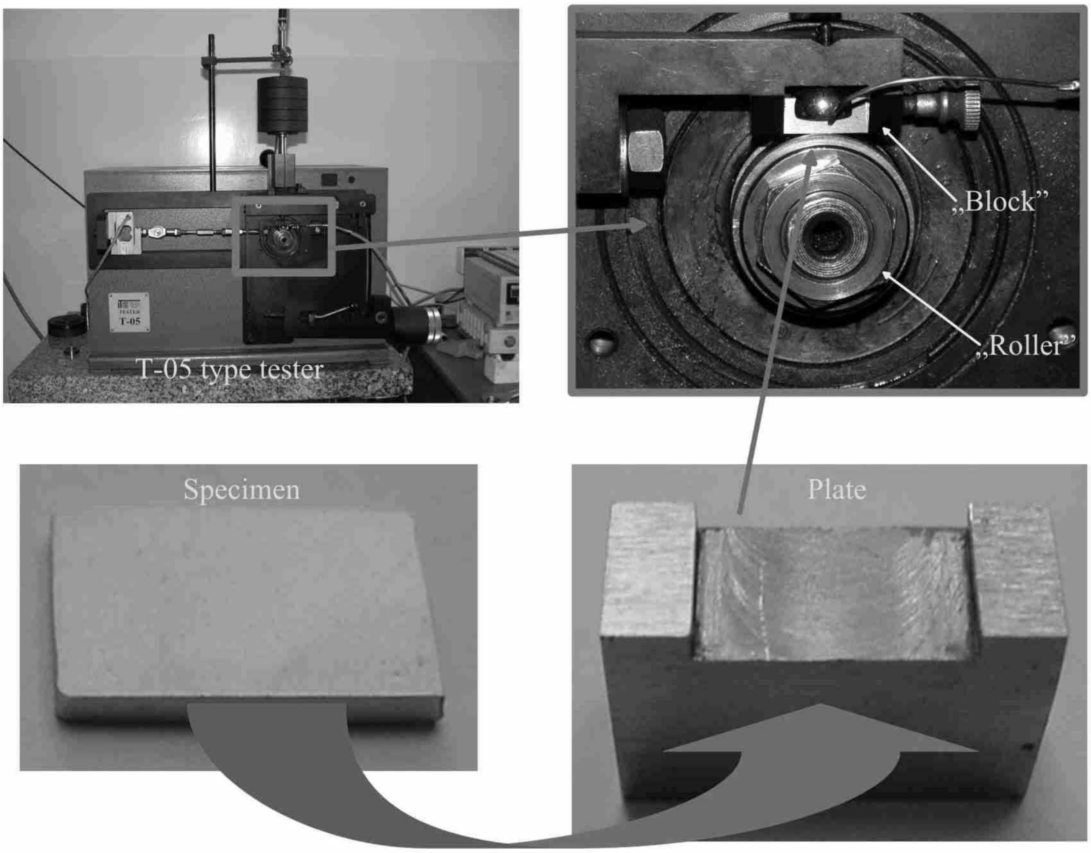

Fig. 1. Stand for wear resistance tests

3. Results and discussion

Specimens' mass decrements, which were the indicator of wear resistance, are specified in Fig. 2.

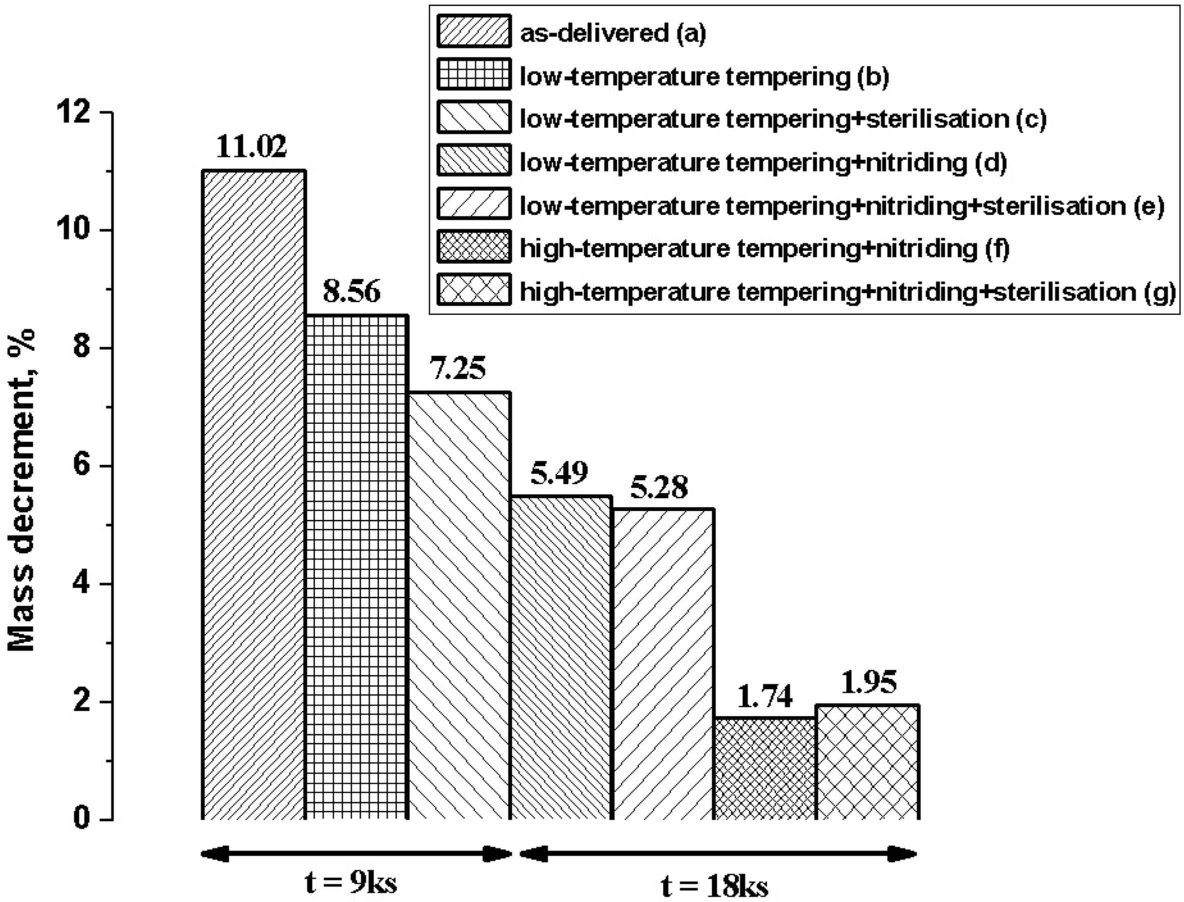

Fig. 2. Mass decrements of X39Cr13 steel after various heat and surface treatments

The record of wear track depth enables analysing the way of material's wear, decrements violence and growth rate, what enables identification of phenomena occurring during the process of wear. The results of laboratory wear tests are shown in Fig. 3.

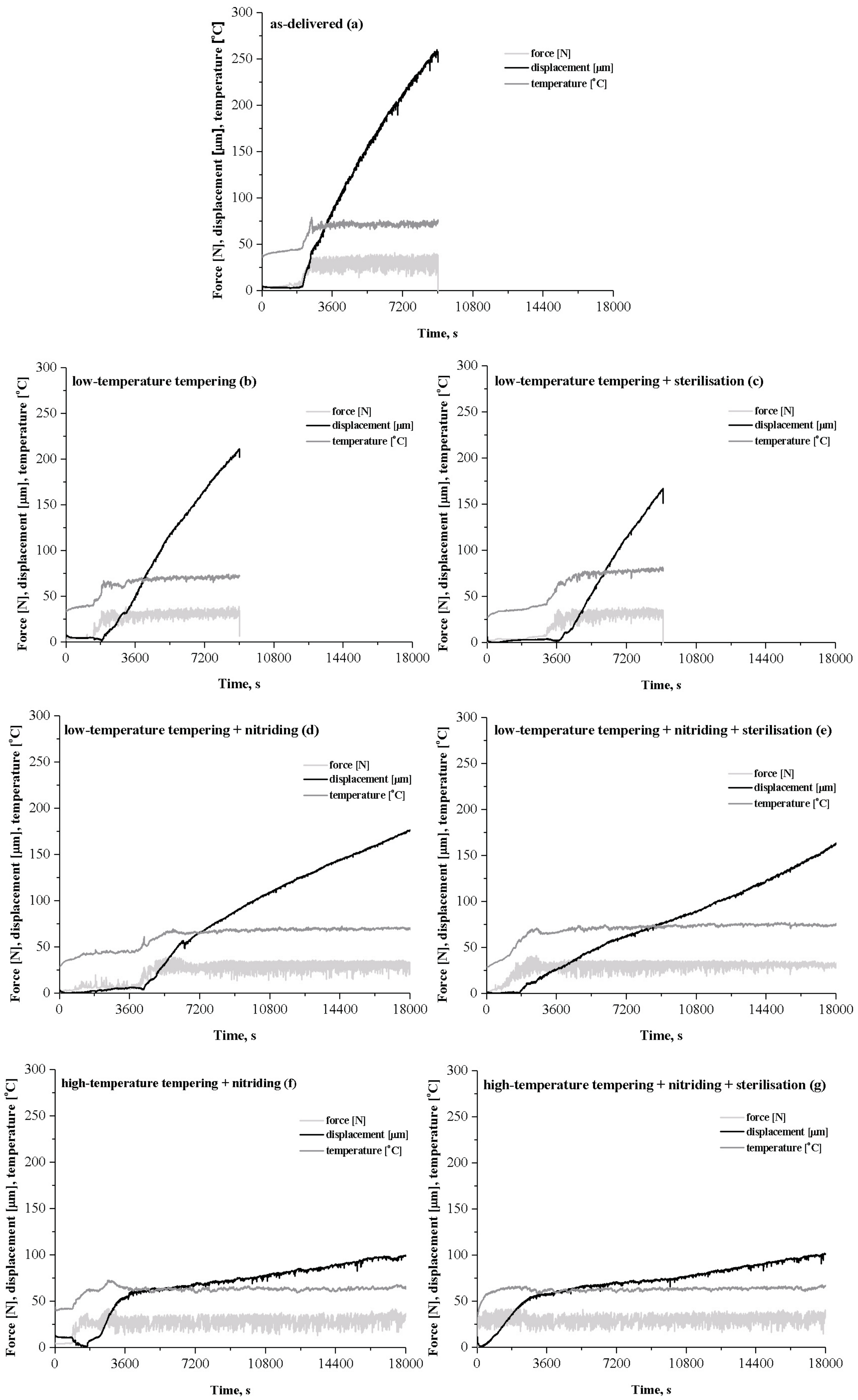

Fig. 3. Results of laboratory wear tests

Heat treatment (low-temperature tempering) causes increased wear resistance as compared with the as-delivered state, at the application of the same test duration parameters. However, only plasma nitriding of X39Cr13 steel resulted in significant increase in wear resistance. For nitrided specimens the test duration was extended from two and half hours (as-delivered and low-temperature tempering) to five hours. This increase is strongly related to the heat treatment preceding the nitriding. Specimens, which were high-temperature tempered prior to nitriding, feature much lower mass decrement as compared with low-temperature tempered and nitrided specimens. So significant differences in abrasive wear of steel nitrided in the same conditions but tempered at two different temperatures may result from substantial differences in hardness of the core and of the surface layer. Previous authors' studies [11, 12] have shown that the hardness after high-temperature tempering is around 300 units lower than the hardness of low-temperature tempered steel. Additionally, the results obtained are confirmed by studies carried out for as-delivered nitrided steel, which are presented by authors in paper [13], and by authors' studies on only quenched and then nitrided steel, not published so far. Substantially lower degree of abrasive wear was observed for as-delivered nitrided steel than for quenched and nitrided steel. This is caused by significantly lower hardness of as-delivered steel (~200 HV0.3) as compared with quenched steel (~800 HV0.3). The sterilisation carried out for various specimen lots did not result in substantial changes of wear resistance.

4. Findings and conclusions

1. The heat treatment applied (quenching and low-temperature tempering) causes an increase in wear resistance as compared with the as-delivered state.
2. The plasma nitriding of martensitic steel at the surface treatment parameters applied results in substantial increase in wear resistance. The highest wear resistance was obtained for high-temperature tempered and nitrided steel.
3. The sterilisation does not have a significant influence on the degree of abrasive wear.

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