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Pulsed Plasma Nitriding of Stainless Steel

Nitriranje nerjavnega jekla v pulzirajoči plazmi

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A use could be found for pulsed-plasma nitrided AISI 316L stainless steel in a wide range of biomedical applications, e.g. femoral and biarticular heads of joint prostheses. Forged samples of steel were pulsed-plasma nitrided at a temperature of 540°C for 24 hours to increase the hardness of the surface. During nitriding, the hardness of the 70 μm thick layer uniformly increased to 958 HV 0.1. The hardness of the steel below the nitride layer remained unchanged, 210 HV 0.1. The thickness of the layer depended on the process parameters. The research showed that nitriding of stainless steel for medical implants in pulsed plasma is feasible.

Key words: stainless steel, pulsed plasma nitriding, biomedical applications

Nerjavno jeklo AISI 316L, nitrirano v pulzirajoči plazmi, bi bilo mogoče uporabiti za biomedicinske namene, npr. za femoralne in biartikularne glave kolčnih vsadkov. Kovani vzorci jekla so bili nitrirani v pulzirajoči plazmi 24 ur na temperaturi 540°C. Med nitriranjem je trdota 70 μm debele plasti enakomerno narasla do 958 HV 0.1. Trdota jekla pod nitrirano plastjo je ostala nespremenjena, 210 HV 0.1. Debelina nitrirane plasti je odvisna od parametrov procesa. Raziskava je pokazala, da je nitriranje medicinskih implantatov v pulzirajoči plazmi izvedljivo.

Ključne besede: nerjavno jeklo, nitriranje v pulzirajoči plazmi, uporaba v biomedicini

1. Introduction

An increase of the wear resistance and surface strength of different steels and alloys with nitride forming elements can be obtained by pulsed plasma nitriding[1]. Usually, also the corrosion resistance of the surface is increased, while the corrosion resistance of stainless steel is sometimes slightly diminished. Modern nitriding devices and technology, however, maintain or even increase the corrosion resistance[2].

Nitriding in pulsed plasma is beneficial due to the low temperature (between 350°C and 660°C) of the process and the possibility of influencing the composition of the nitrided layer (γ', ϵ, $\gamma'+\epsilon$, and diffusion layer)[3]. The low temperatures of the process maintain the mechanical properties of the material below the layer unchanged. The process is ecological friendly and nontoxic.

Gases in normal state are nonconductive for electrical current. This property is changed by high voltage or at low pressure when lightning or glow discharging appears, respectively. In both cases, the nonconductive gas is transformed to an ionized plasma with a sufficient electrical conductivity. Nitriding in pulsed plasma is based on glow discharging pulsed current in a low pressure chamber. Electrons are released on the cathodic surface of the sample, sputtering off the surface atoms, and nitrogen ions migrate into the specimen.

At a distance of some millimeters above the cathodic surface of the specimen, the ions accelerate and hit the surface with high kinetic energy. About 90 % of this energy is transformed to heat, which warms the surface up to the nitriding temperature. The heat is controlled by electric power, and no additional heating is required.

Nitrogen ions are highly reactive in the plasma, and iron nitrides start to form on the sputtered surface. Because of the low temperature, FeN molecules on the surface of the tool or sample decompose into lower nitrides. At the decomposition $FeN \rightarrow Fe_2N \rightarrow Fe3N \rightarrow Fe4N$, nitrogen is released. A part of the released nitrogen diffuses into the sample and the rest is returned into the plasma. The process

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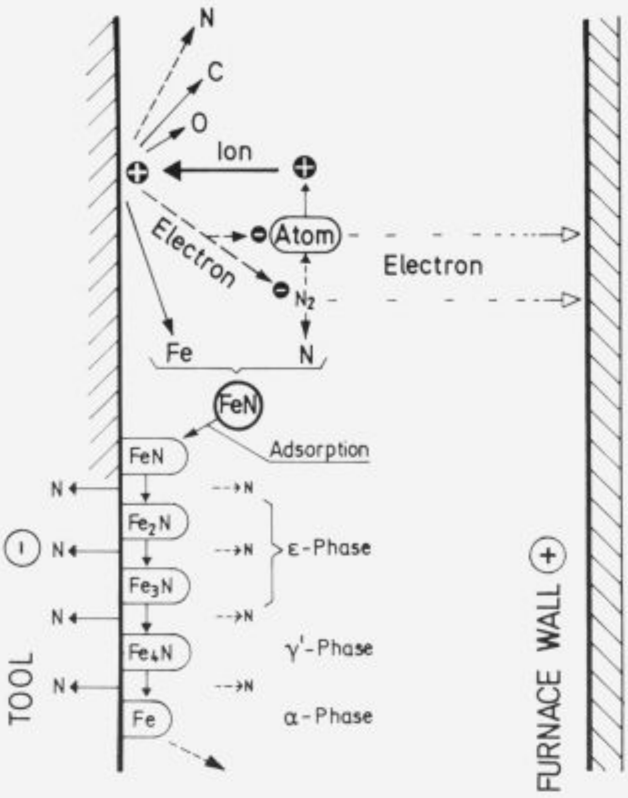

Figure 1:
Schematic presentation of the ion nitriding process
Slika 1:
Shematski prikaz postopka ionskega nitriranja

of ion nitriding is shown schematically in **Fig. 1**. In principle, all materials based on iron can be nitrided, since with glow discharging, ions of nitrogen are active enough to recombine on the surface. In some minutes of treatment, the nitride layer is formed and the steep gradient of concentration accelerates the diffusion of nitrogen into the specimen[4].

The objective of the present research was to check the nitriding of AISI 316L stainless steel in pulsed plasma.

2. Experimental work and results

2.1. Experimental procedure

The samples for nitriding were machined from the AISI 316L stainless steel bars with diameter of 30 mm and the composition of the steel was: 0.049 % C, 17.9 % Cr, 13.1 % Ni, 2.5 % Mo, 0.03 % Al and were pulsed-plasma nitrided at 540°C for 24 h.

After nitriding, the samples were prepared for microstructure examination and hardness tests. The hardness was measured with a Vickers indenter, using a 100 g load and a 10 s load duration. The microstructure was examined, after etching with Marble's reagent, by optical microscopy, while the fracture surfaces of the nitride layer were examined by scanning electron microscopy (SEM).

2.2. Experimental results

In **Fig. 2 a** and **b**, the optical micrographs of unetched and etched nitrided layers are shown. The nitrided layer is light-gray in an unetched condition, and only inclusions are found by optical or scanning microscopy.

After etching in Marble's reagent, the nitride layer is darker and it looks homogeneous.

The optical micrographs **(Fig. 3 a and b)** of the transverse section through the base steel and nitride layer show Vickers imprints and a needle scratch, both of which confirm the increased hardness of the nitride layer. The needle scratch, easily visible in the base steel, disappeares when crossing the harder nitride layer. The increased hardness of the nitride layer is connected to the reduction of its fracture toughness and the appearance of a brittle fracture, whereas the fracture of the steel below the layer remains ductile. In **Fig. 4 a** the cracks in the nitride layer, formed at bending the nitrided surface to an angle 180° are shown. The fracture is brittle and the crack stops in the interface of the nitride layer-steel, because the base material has a higher fracture toughness. The topology and morphology of the nitrided surface with a bending crack is shown in **Fig. 4 b**. The nitride surface can be polished to a high brilliancy, which is important for medical use, as i.e. for femoral and biarticular heads of joint prostheses.

The modification of the surface morphology and topology may be beneficial for an improved biological performance or improved bone-bonding[5] at the uncemented modular stem or cement-bonding at the cemented stem.

The base steel shows after nitriding a ductile transgranular fracture **(Fig. 5)**.

Hardness measurements show an average and homogeneous hardness of 958 HV 0.1 in the layer **(Fig. 3 a)** and a hardness of 210 HV 0.1 in the base material. The usual hardness transition zone, found in nitrided steels, was not found. The results confirm that the process of ion nitriding is suitable for increasing of the relatively soft AISI 316L stainless steel surface. It is regarded as promising for increasing the wear resistance of such materials.

3. Conclusions

Ion nitriding of AISI 316L steel in pulsed plasma increases the surface hardness from 210 HV 0.1 to 958 HV 0.1, while the hardness of the base alloy is unchanged.

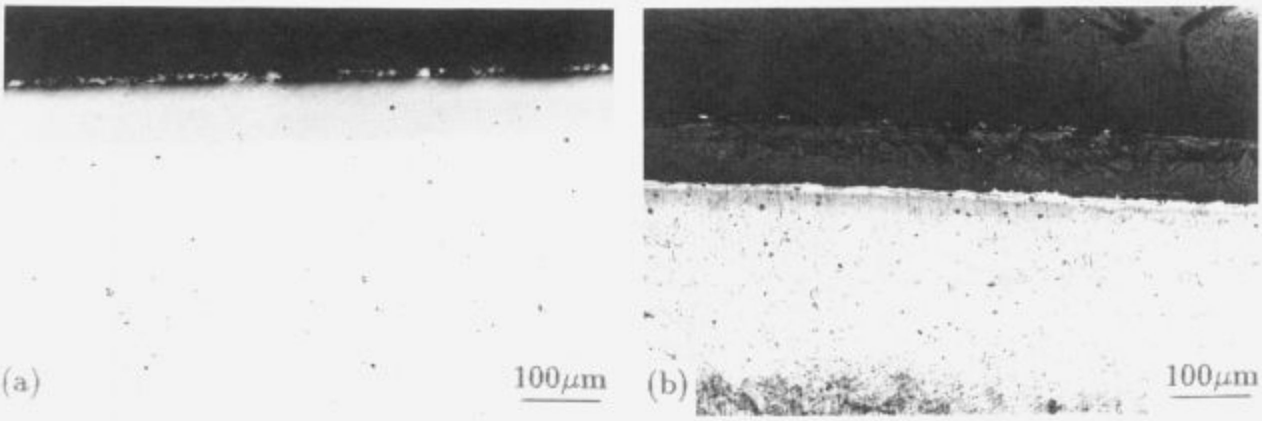

Figure 2 a and b: Optical micrograph of the nitrided layer after nitriding in pulsed plasma for 24 hours at 540°C, **(a)** - unetched, **(b)** - etched with Marble's reagent

Slika 2 a in b: Optični mikroposnetek nitriranega sloja po nitriranju v pulzirajoči plazmi, 24 ur na temperaturi 540°C, **(a)** - nejedkano, **(b)** - jedkano v Marble jedkalu

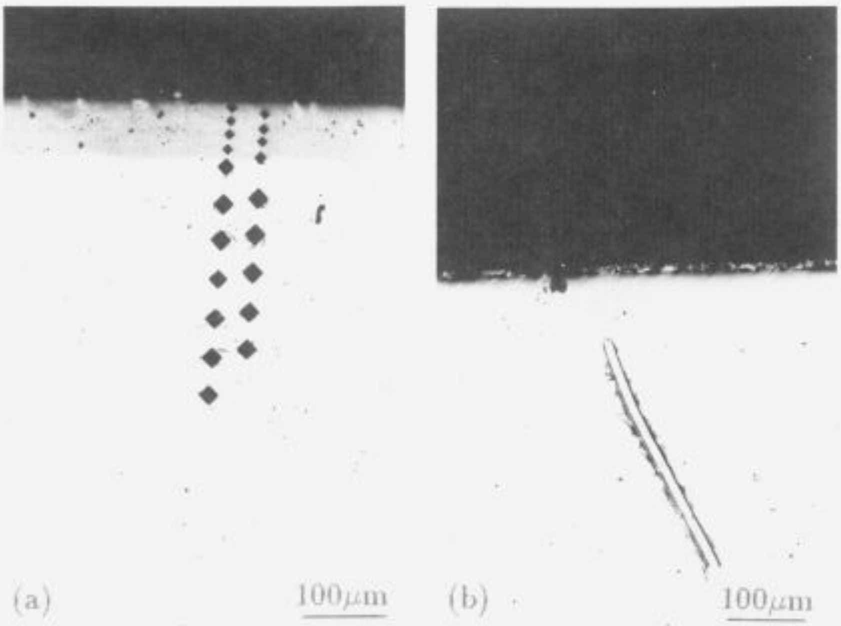

Figure 3 a and b: Optical micrograph of the nitride layer, **(a)**- Vickers indentions and **(b)**- a needle scratch

Slika 3 a in b: Optični mikroposnetek nitriranega sloja, **(a)**- odtisi merjenja trdote po Vickersu in **(b)**- raza preko nitriranega sloja, napravljena z iglo

The thickness of the layer after 24 hours of nitriding at 540°C is up to 70 μm.

The propagation of the crack opened in the nitride layer stopps at the interface nitrided layer-base steel due to higher ductility of the base steel. This indicates to a difference in fracture toughness between the nitrided layer and the matrix.

The modification of the surface morphology and topology may be beneficial for an improved surface bonding with bone or cement.

Acknowledgement

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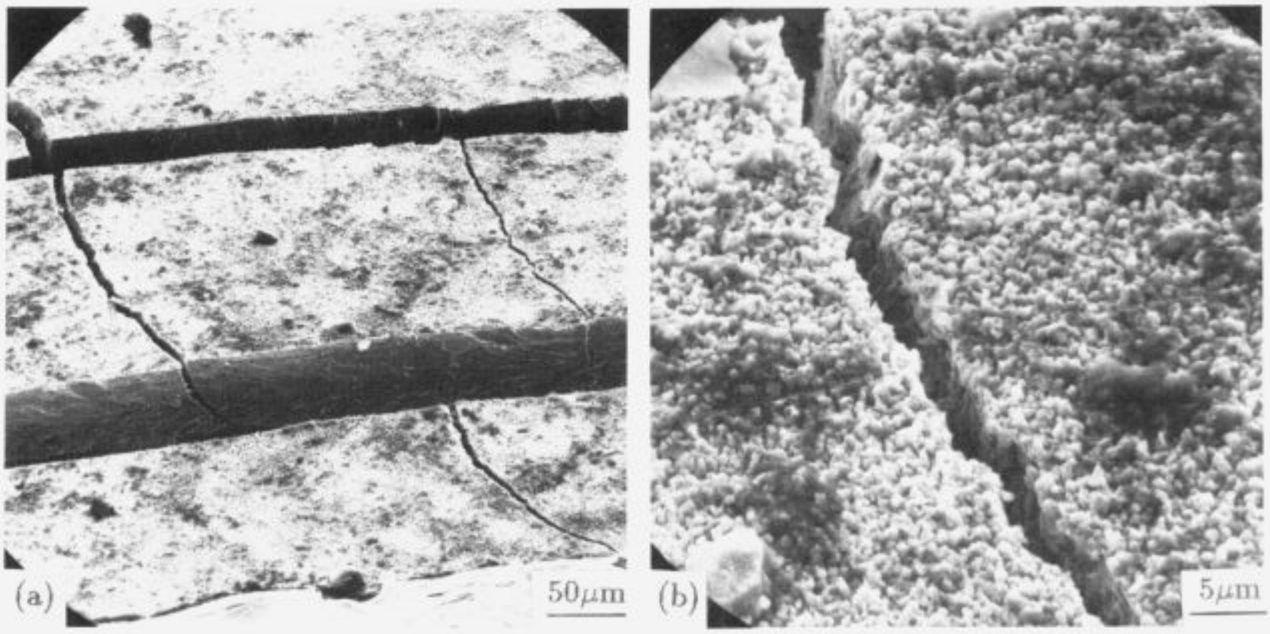

Figure 4 a and b: SEM micrographs. Nitrided surface with bending cracks **(a)**- bending angle 180° and **(b)**- detail of the surface

Slika 4 a in b: SEM posnetek nitrirane površine z razpokami, nastalimi pri upogibu, **(a)**- kot upogiba 180° in **(b)**- detajl površine

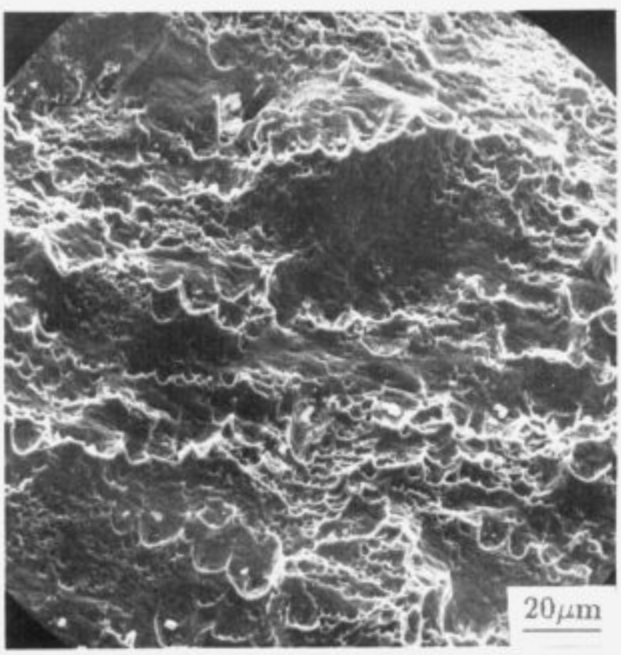

Figure 5: SEM micrograph. Ductile fracture of the base material

Slika 5: SEM posnetek žilavega preloma jekla pod nitriranim slojem

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