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ORIGINAL PAPER

Irradiation of austenitic steel 10Cr12Mn14Ni4AlMo and titanium alloy Ti-Al-V by pulsed streams of fast nitrogen ions and plasma in a dense plasma focus

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Abstract. Austenitic steel 10Cr12Mn14Ni4AlMo and Ti-4Al-3V alloy were irradiated with nanosecond pulsed nitrogen ion and plasma streams in plasma focus devices. The two different modes of the treatment were applied: high power density ($\geq 10^8$ W/cm^2) irradiation with melting of the surface layer and irradiation with power density $\sim 10^7$ W/cm^2 below the melting threshold. Structure and phase changes as well as the mechanisms of modification and hardening of the surface layers of the steel and titanium alloy upon applied irradiation are discussed.

Key words: radiation • damage • phase transition • nanohardness • dense plasma focus (DPF)

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Introduction

It is known [2–8] that irradiation of materials by powerful streams of fast ions and hot plasma within different accelerators including the dense plasma focus (DPF) device results in changes of phase and structure states of surface layer (SL) and gives an opportunity to modify its physical, chemical, mechanical and some other characteristics.

Main feature of a DPF device that differs it from other irradiation facilities like pulsed plasma guns, injectors, plasma accelerators, fast ion and electron accelerators, is as follows. DPF generates hot plasma jets (plasma temperature is in the range from several hundred eV till several keV) with velocities (2–5) $\times 10^7$ cm/s produced simultaneously with fast ion and electron streams (energy of electrons and deuterons are in the range from 50 keV up to several MeV). Because the powerful phase of the DPF discharge lasts 10^{-8}...10^{-7} s, this device is able to irradiate targets with power flux densities of the above types of radiation on the level up to 10^{10} (for hot plasma), 10^{12} (for fast ions) and $> 10^{13}$ W/cm^2 (for fast electrons). For high-Z ions energy of them, obtained due to acceleration in a quasi-stationary electrostatic filed induced within the DPF may reach 100 MeV depending on their charge. Unlike to the case of powerful laser beams the above radiation types of DPF produce volumetric action upon surface layers of materials. These facets can be ensured not in one of the above-mentioned devices all together. It means that the DPF device can produce unprecedented changes of structure, phase composition and some other characteristics of the specimens under irradiation.

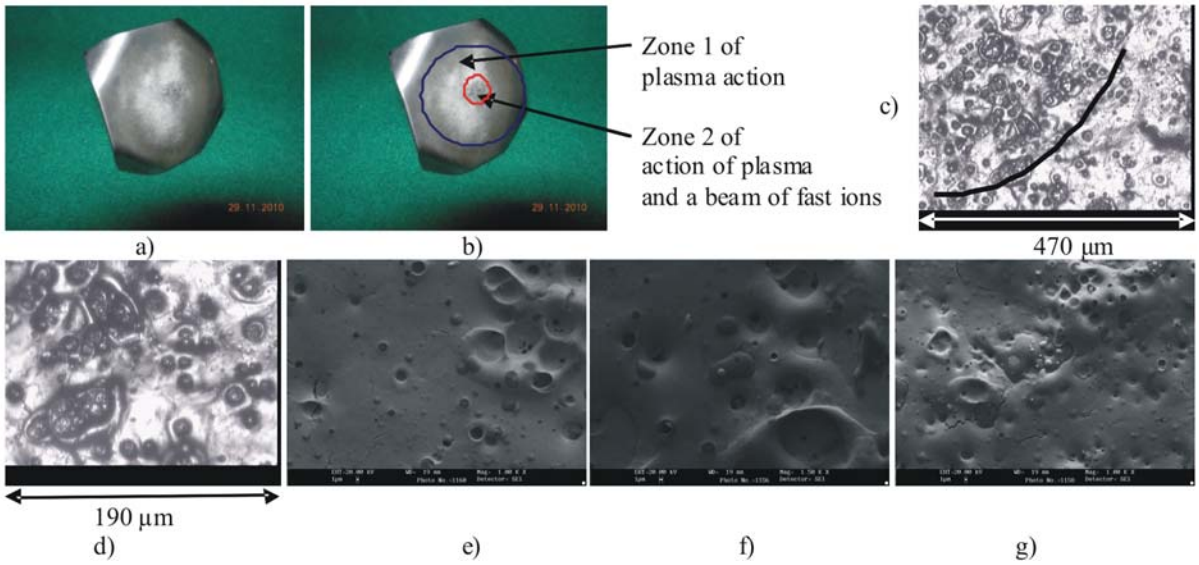


Fig. 1. Macroscopic photo of the austenitic steel sample irradiated in the "Bora" device (a) and (b) with regions irradiated by plasma only (1) and by plasma and a beam of fast ions (2); optical microscopy image of the region near the border of the first and second zones (c) and zone 2 with higher magnification (d) and electron microscopy of the regions (e, f, and g).

Implantation of the working gas ions at the cathode part of the DPF chamber may take place with and without SL melting at the power flux densities $\geq 10^8$ W/cm^2 and $\sim 10^7$ W/cm^2 correspondingly. It attains by variation of a distance from samples to the DPF anode. In both dissimilar cases its structure and properties will be different. Implantation of ions of various elements is one of the modern methods of modification of SL [3–5, 7, 8] aiming at improvements its properties.

Experimental results

In the present set of experiments nitrogen was used for a DPF filling (instead of deuterium as it was done in our previous tests of materials, candidates for fusion reactors [2, 6]). The main aim of the work was investigation of the SL modification of samples of the austenitic steel 10Cr12Mn14Ni4AlMo and titanium alloy Ti-3Al-4V irradiated by powerful streams of fast ions (peak energy $\sim$ 100 keV) and hot plasma (temperature about several hundred eV) within the above-mentioned parameters. We studied damage characteristics and we examined perspectives of the use of such irradiation by nitrogen streams generated in DPF devices for improvement of the mechanical properties of these materials.

In our experiments we used three DPF devices:
- PF-5M (IMET – Institute of Metallurgy and Material Sciences, Moscow, Russia),
- PF-6 (IPPLM – Institute of Plasma Physics and Laser Microfusion, Warsaw, Poland) and
- "Bora" (ICTP – International Centre for Theoretical Physics, Trieste, Italy).

Energies of the banks of these devices were varied from 2 kJ up to 5 kJ. Samples were placed in front of the anodes with distances from 5 to 11.5 cm to realize two regimes of irradiation – with melting of SL and without it. Numbers of irradiating pulses produced in each set of experiments were 50. Figure 1 presents the macroscopic photo, typical of optical microscopy and scanning electron microscopy (SEM) images of the specimen's SL of austenitic steel 10Cr12Mn14Ni4AlMo irradiated in "Bora" (4.8 kJ) and PF-6 (5 kJ) devices placed at a distance of 5 cm from the anode ($\geq 10^8$ W/cm^2). Damage is characterized in these cases by pores, bubbles and cracks of the size of several tens of μm. X-ray diffraction method of analysis has shown that during irradiation an implantation of nitrogen atoms into austenitic lattice noticeably increases a parameter of the gamma-phase (Table 1). Similar results were previously obtained by us for the 0.3Cr10Mn33WV austenitic steel irradiated within DPF in the same conditions [1].

An increase of nitrogen concentration in the solid solution, which is an effective stabilizer of gamma-phase, compensates a certain loss of manganese. Ferritic and martensitic phases inside the SL were not observed. After high-temperature irradiation with subsequent fast cooling, this matrix solution has a structure of the perfect gamma-phase. A small deformation of the martensite phases (ε- and α-martensite) presented in virgin samples of the austenitic steel turned by a diffusionless way into austenite during rapid heating at the irradiation by the first pulse of plasma. Table 2 presents the nitride phases created in the SL of this steel due to chemical activity of nitrogen atoms in relation to steel's components.

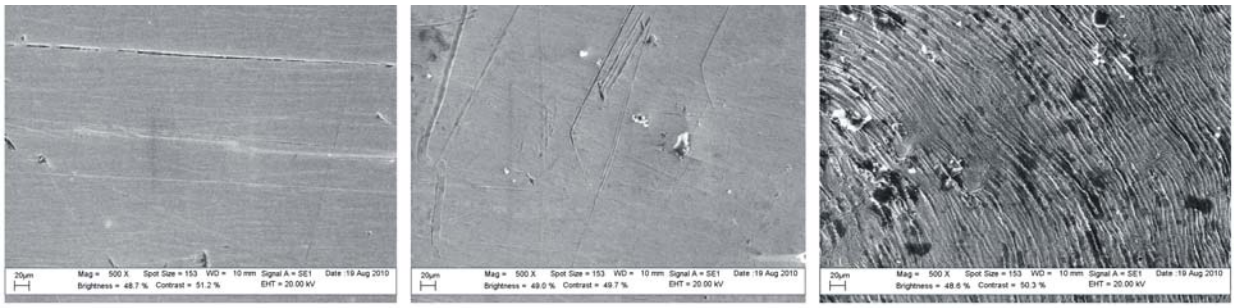
Thus the X-ray phase analysis has shown that different nitrogen atoms implanted into the SL of the steel under investigation behave themselves in the lattice of the gamma-austenite by a dissimilar manner: one part of them occupies places within the interstitial sites of the lattice (that results in the increase of the lattice parameter observed, see Table 1), whereas the other part of them "used" for the formation of fixed nitride phases.

Table 1. Parameters of the fcc lattice of austenitic steel irradiated by nitrogen ions and plasma

No. of sample	Sample state	a (Å)	V (Å^3)
1	Before irradiation	3.6012	46.70
2	After irradiation by fast ions and plasma of nitrogen	3.6234	47.57

Table 2. Type and quantity of nitride phases in SL of the above steel after irradiation

No. of specimen	Nitride phase	Lattice type	Quantity (%)
1	$FeN_{0.0880}$	fcc	8.5
2	$FeN_{0.0939}$	bcc	< 2
3	Fe_4N	B1	< 2
4	Fe_3N	hexagonal	< 2
5	Fe_2N	orthorhombic	< 2



a) b) c)

Fig. 2. Electron microscopy images of specimens: virgin sample (a), sample #2 irradiated without melting (b) and sample #1 irradiated with melting of a SL (c).

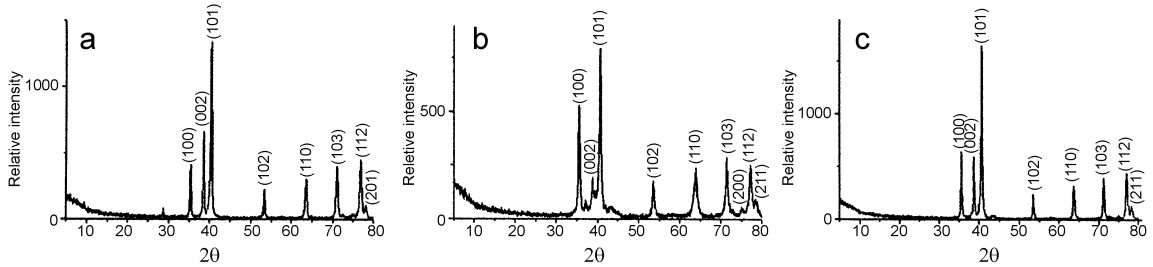


Fig. 3. X-ray diffraction patterns of the virgin sample (a) of the titanium alloy Ti-4Al-3V and samples after irradiation: the sample #1 (b) and the sample #2 (c).

The above-mentioned features make usually in favor of an increase of hardness of material. Tribological measurements will be our next step in these researches.

Titanium alloy Ti-4%Al-3%V was irradiated at PF-5M (2 kJ) with the same nitrogen streams of plasma and fast ions at distances of 8 cm ($\leq 10^8$ W/cm^2, with SL melting, sample #1) and of 11.5 cm ($\sim 10^7$ W/cm^2, without melting, sample #2) from the DPF anodes. Electron microscopy (Fig. 2) has shown that, dissimilarly with the case of steel (Fig. 1), the morphology of the SL of the titanium alloy irradiated at 10^7 W/cm^2 was not changed (b), whereas for $\sim 10^8$ W/cm^2 a wave-like structure with crests of $\sim$1 μm widths and of several hundred μm lengths were observed (c).

Elemental analysis of samples has shown that concentration of nitrogen implanted to the SL of the alloy is higher in the case of its melting. It is so in spite of the fact that in this harsh mode of irradiation the SL was not only melted at each pulsed discharge, but it was also evaporated, i.e. previously implanted nitrogen was partially removed from the SL.

Observation of the X-ray diffraction patterns (Fig. 3) has shown that nitrides of the main components of the titanium alloy are presented inside the implanted SL: Ti_2N, Ti_2AlN, AlN, V_2N. Besides nitrides that appeared due to the reactions of nitrogen with the titanium alloy, some dopant materials deposited on the SL from the chamber elements were found: Ni_3N, $(Cr,Fe)_2N$, Fe_2N, CrN.

In the sample #1 the maximal volume part corresponds to the nitride Ti_2N, whereas in the sample #2 – to the compound Ti_2AlN. All nitrides are concentrated in the thin layer having a thickness of about $h \leq 200$ nm independently of the regime of irradiation.

X-ray phase analysis has also shown that surfaces of samples are covered by an oxide film of titanium TiO_2, whereas the basis of the SL is composed of α-solid solution with a small percentage of β-phase. Parameters *a* and *c* of the crystal lattice are decreased after irradiation (see Table 3). It means that nitrogen implanted into titanium is not in the interstitial positions (as it was happened in the lattice of gamma-austenite), but in

Table 3. Lattice parameters of the α-phase of Ti-alloy

Sample type	Power flux density q (W/cm^2)	Number of irradiation pulses (N)	Lattice parameters of the α-phase (nm)	
			a	c
Virgin	–	–	0.2927	0.4671
After irradiation	10^8	50	0.2913	0.4659
After irradiation	10^7	50	0.2918	0.4654

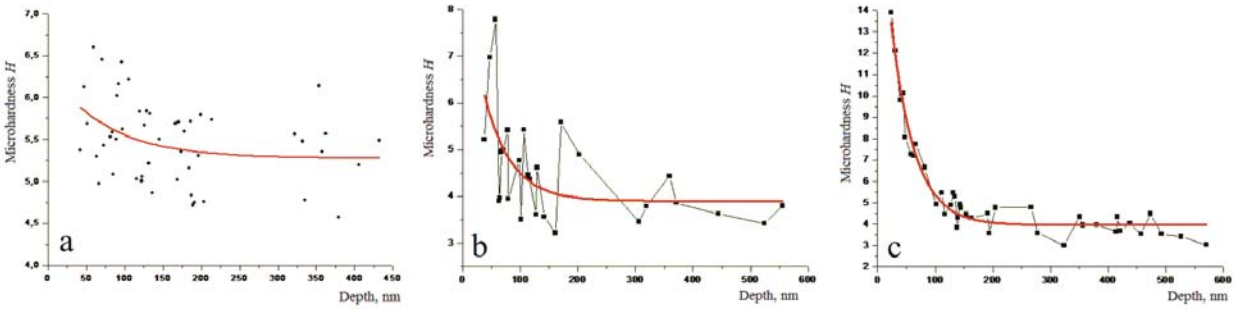


Fig. 4. Variations by depth of nanohardness H (in hPa) of samples of the titanium alloy of the type Ti-4Al-3V in the proximity of the irradiated surfaces of samples: virgin (a), sample #1 (b) and sample #2 (c).

the intermetallic compounds – nitrides observed by us. Besides the doping elements (Fe, Cr, Ni, Si) deposited on the surface under irradiation have lower atomic radius compared with titanium. Being substitutional atoms, these elements at their penetration into the lattice decrease also its parameters. Maximal decreasing of the parameter a was observed for the samples irradiated with melting of their SLs.

Measurements of nanohardness within SLs were provided by means of the device „NanoTest" having a wide range of loads upon indenters and equipped by a computerized data acquisition system. Their results are presented in Fig. 4. One may see that a formidable increase of nanohardness H is observed within the layer having a thickness about 100 nm. Thus inside the non-irradiated sample the value of H near its surface is equal to ≈ 6.5 hPa, whereas within the sample #1 it increases up to $H \approx 7$–8 hPa and in the sample #2 this magnitude is $H \approx 14$–15 hPa. The same values of H were observed for the alloy Ti-6Al-4V at its pulsed irradiation by low-temperature plasma in a more soft regime of treatment (plasma temperature was 1.5–2.0 eV, power flux density $\sim 10^6$ W/cm^2 and long pulse duration ~ 1 ms) [7].

Our estimations have shown that the observed decrease of nanohardness of the alloy in the range of depth 200 nm < h < 600 nm may result from its thermal softening in this zone because of its heating by several hundred degrees at every pulsed action of the stream of energy upon the sample. In these conditions the alloy lost its hardness due to possible phase conversion $\beta \rightarrow \alpha$ and stress relief within the lattice. Strong increase of nanohardness H within the nanolayer h < 100 nm is determined by the presence in its contents of nitride phases and deposited dopant elements.

Quite unexpected result is that an increase of H within this layer is higher in the sample irradiated without melting compared with the sample with a production of liquid phase on its surface. It may be connected with the fact that at melting the initial reinforced state (cold hardening) of SL, provided by rolling at the stage of the sample's preparation, disappears whereas its resulted hardening is reached due to quenching of melted layer enriched by dopant elements and production of nitride phases. If there is no melting of the surface layer (sample #2), its initial reinforced state is preserved and implantation of nitrogen ions into the alloy's lattice accompanied by a production of nitrides with basic and dopant elements of the anode's materials results in additional strengthening of the alloy.

Conclusions

1. It is shown that irradiation of the austenitic stainless steel 10Cr12Mn14Ni4AlMo and titanium alloy Ti-3Al-4V by nitrogen hot plasma (< 1 keV) and fast ion (> 100 keV) streams at the DPF device allows modification of their surface layers by means of two mechanisms simultaneously – by implantation of nitrogen ions into materials and by additional doping by some elements.
2. It is found that in the DPF operational regimes realized (at power flux densities of about 10^7–10^8 W/cm^2) nitrogen ions implanted into alloys on the basis of iron and titanium have different behavior. In the lattice of the gamma-austenite one part of them occupies places within the interstitial sites of the lattice (that results in the increase of the lattice parameter observed), whereas the other part of them takes part in the formation of nitride phases, while in the titanium alloy nitrogen ions participate mainly in the formation of nitride phases only.
3. A noticeable strengthening of thin nanolayers (depths about several tens of nm) of the titanium alloy after their treatment by powerful pulsed streams of nitrogen plasma and fast ions was observed. Magnitudes of nanohardness in these layers ($H \approx 15$ hPa) are higher by several times than the initial values. These figures are formed due to the production of nitride phases with basic (Ti, Al and V) and dopant (Fe, Cr and Ni) elements deposited on the alloy's surface.

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