Microspectral Analysis of Substance with Help of the Nitrogen Laser

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Abstract – The results of experimental research aimed at developing an express method of testing and analysis of elemental composition of materials using a simple compact nitrogen laser are reported. The influence of intense laser radiation on surfaces was studied at atmospheric pressure of a surrounding gas. It was shown that the coating thickness can be determined in a range of 100–1000 nm.

1. Introduction

At present, one of the important tasks is the development of express methods for determination of thickness, structure, homogeneity, durability of coatings of nano- and micrometer thickness. This task can be solved using laser radiation. Recent research in the given area supports such possibility [1–3]. Under intense laser radiation, plasma radiates the lines of atoms which the material consists of. Thus, the main difficulty is the presence of a continuous plasma spectrum increasing the detection threshold of the material. Traditionally, a spectral analysis of laser plasma is carried out using a neodymium laser. It was shown in [4], that short-wavelength lasers (N_2 or excimer lasers) are more preferable from the point of view of increasing the signal/noise ratio and stability of plasma luminescence. In addition, it was shown in [5], that at a low pressure of surrounding gas, laser plasma consists of two parts: primary and secondary plasma. The primary plasma occupies a small area and gives off an intense continuous spectrum for a short time just above the surface of the target. While in the secondary plasma, it is the atoms of the material that mostly emit. Recording with a time-delay allows us to reduce the influence of continuous background in the spectrum under study. Another advantage of the nitrogen laser is suppression of the selective evaporation typical of neodymium lasers.

In the given work, the results of experimental research aimed at developing an express method of testing and analysis of elemental composition of material using a simple compact nitrogen laser are discussed. The influence of intense laser radiation on the examined surface was studied at atmospheric pressure of a surrounding gas. This allows us to increase the efficiency of diagnostics and remove the restriction on the sample size.

2. Equipment and materials

In the experiments, use is made of nitrogen laser developed at the High Current Electronics Institute of the Siberian Branch of the Russian Academy of Sciences (Fig. 1). The laser was pumped by a transverse electrical discharge. Laser radiation has the following characteristics: beam size 3×14 mm^2, pulse energy up to 1.5 mJ, pulse duration t = 4–6 ns, pulse repetition rate up to 100 Hz. The laser can work on pure N_2 (at 120 torr) or an N_2–He gas mixture (at 1.25 atm). Circulation of the laser mixture is carried out by a diametrical fan mounted in the laser working chamber.

Fig.1. Appearance of the N_2 laser. Dimensions of the laser: $l \times h \times w = 60 \times 42 \times 23$ cm

Radiation is focused by a lens with the focal length F = 60 mm. An HR4000 (Ocean Optics) or “Klavi” spectrometers with a resolution of 0.25 nm are used for registration plasma spectrum.

We have chosen samples which contain titanium, nickel, copper, chromium, as most attractive for research and analysis.

3. Results and discussion

The spectral lines intensities of laser plasma on the surface of a Ni–Cr alloy and pure Ni and Cr are shown in Fig. 2 (left). It is evident, that the spectral lines correspond to the transitions of the excited atoms alone, no transitions to ions are observed. The spectral lines intensities of laser plasma on the surface of titanium nitride coatings are shown in Fig. 2 (right). The lines of titanium atoms and scattered radiation of the nitrogen laser at the working wavelength 337.1 nm are clearly seen. The presence of strong spectral lines in the samples allows reliable determination of elemental composition of coatings. Typically, one can see only the lines of the excited atoms in all cases, though bright plasma is also observed. Plasma luminescence on the surface of a titanium nitride sample and on a substrate coating (12X18H10T) is shown in Fig. 3.

The significant difference in luminescence is, in our opinion, caused by internal crystal lattice strain of the coating. After repeated irradiation of the same point of the coating, sparks of luminescence are no longer observed.

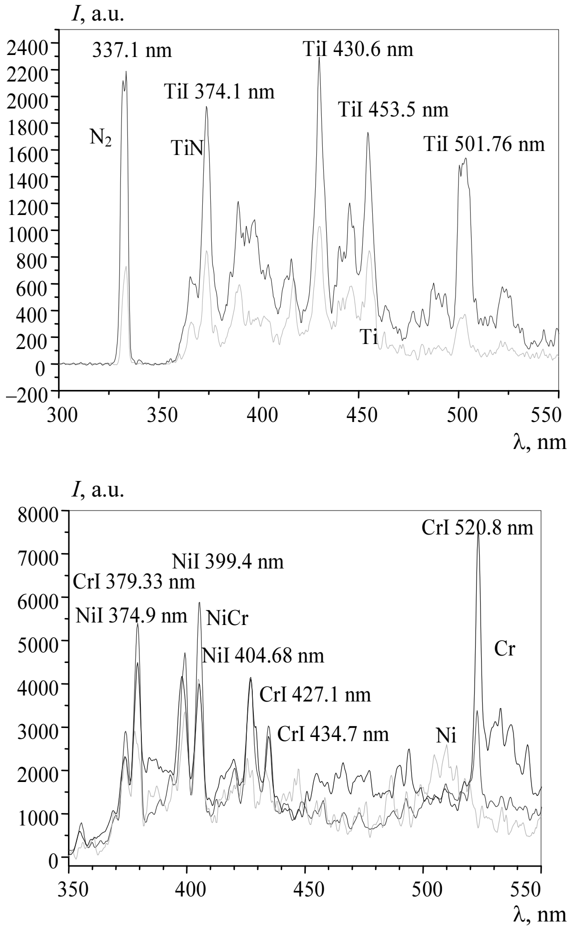

Fig. 2. Spectral lines of laser plasma of a Ni–Cr alloy, Ni and Cr (top) and TiN (bottom) intensity

We have determined characteristic luminescence times of plasma: it was about hundreds μm with sparks and less than 50 μm without sparks (Fig. 4). Focusing of laser radiation to a small spot allows us to carry out detailed research of homogeneity of composite alloys which, for certain tasks is of primary importance. For example, the hydrogen power engineering needs thin and homogeneous YZr films on the surface of porous materials. In order to produce them, it is necessary to have an initially homogeneous YZr alloy. The results of research into homogeneity of a YZr cathode are in Fig. 5. The radiation intensity ratio of YII ions with $\lambda = 4374.9$ Å and Zr atoms with $\lambda = 4710.1$ Å was chosen as a parameter to be measured.

Since our N_2 laser works in a pulse-periodic mode with the parameters controlled in each pulse, the spectral composition of laser plasma could be determined at the moment of coating removal when the spectral

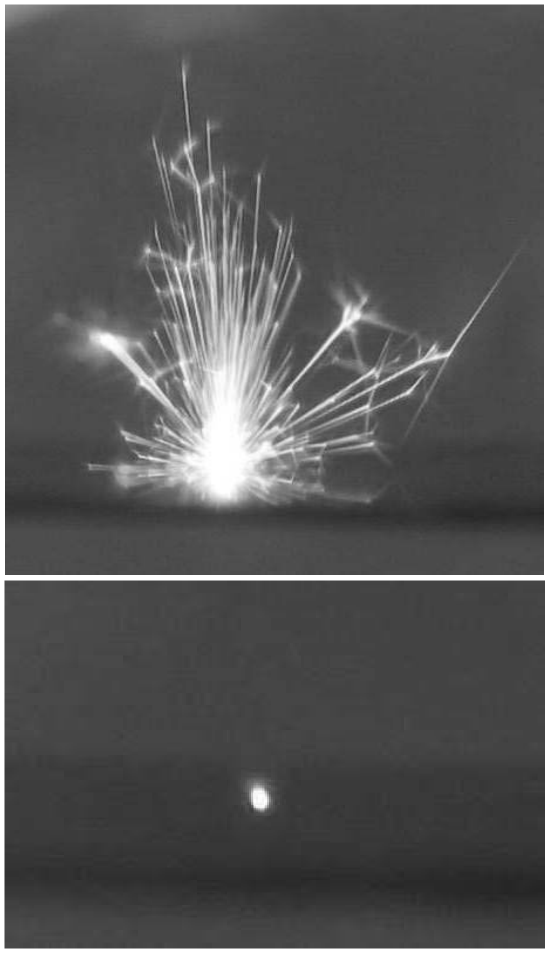

Fig. 3. Plasma luminescence on a TiN coating surface (top) and a stainless steel substrate (bottom)

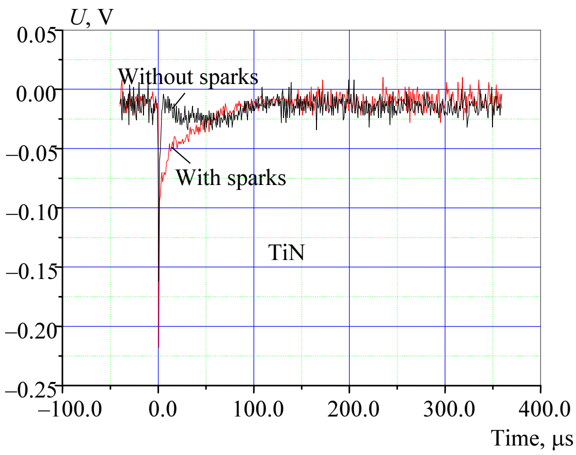

Fig. 4. A change in plasma luminescence in the presence and absence of sparks

lines of the substrate material appear instead of the spectral lines of the coating. If one knows the coating thickness which is carried away during one laser pulse, it is possible to use this method for determination of the total coating thickness. To test the method, the experiments on removal of TiN coatings of dif-

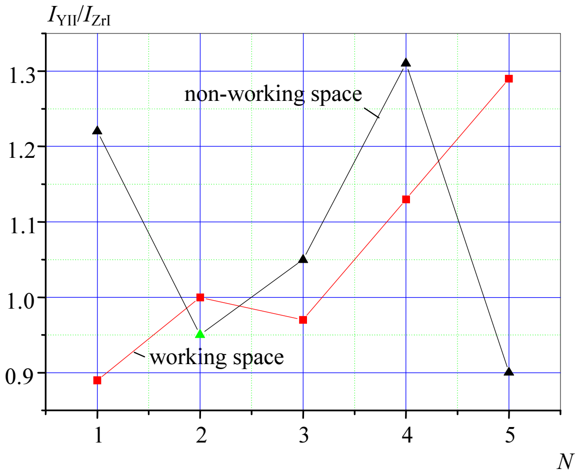

Fig.5. The relation of intensity radiation of YII ions with wave length λ = 4374.9 Å to intensity radiation of Zr atoms with λ = 4710.1 Å at different points of YZr alloy

ferent thickness have been carried out. It was important to choose optimum irradiation intensity on the coating surface. In our experiments the radiation intensity varied from 10^7 W/cm^2 up to $3 \cdot 10^9$ W/cm^2. The intensity was provided at a focal spot with a diameter of 50–200 μm. Reliable removal of the material was observed at the highest intensities. A change in the coating thickness from 30 nm up to 10 μm affected the amount of material removed during one shot but slightly. As a result of research into a TiN coating it was found that at 2 GW/cm^2, a 20-nm layer is ablated during one pulse. We removed a 60 nm coating during 3 pulses, and 1 μm coating – during 50 pulses.

4. Conclusion

Thus, a simple nitrogen laser can be used for determination of elemental composition and homogeneity of materials under atmospheric pressure. Fast determination of coating thicknesses in the range 100–1000 nm is also demonstrated. The developed method can be applied to any materials and differs from the known methods by simplicity and high speed of information acquisition.

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