THE USE OF RESONANCE NEUTRON METHOD FOR DETERMINATION OF PALLADIUM CONTENT IN THE ELEMENTS OF THE PROTON ROCKET ENGINE

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Abstract

We performed an elemental analysis of a detail from the Proton-M rocket-carrier engine at the pulsed white-spectrum neutron source IREN (JINR FLNP), using the resonance neutron spectroscopy and prompt gamma resonance neutron capture method.

For soldering the rocket engine structural walls with the fuel injector of the gas generator RD-0210 (second stage) or RD-0212 (third stage), instead of the G70NH alloy (nickel, chromium, and manganese, Ni-Cr-Mn) was used PJK-1000 alloy (palladium, nickel, chromium, and silicon, Pd-Ni-Cr-Si), which led to the crash of the "Proton" rocket. To avoid further potential crashes, it is desirable to develop a method of non-destructive elemental analysis of the rocket engine or its parts, in particular aiming at determination of Pd, which indicates the presence of the PJK alloy. The resonance neutron method is a potentially suitable tool for such a task, as the resonance capture cross section of palladium is extremely high, which may result in excellent sensitivity to this particular element. The method is non-destructive and practically doesn't induce any residual radioactivity in the investigated sample.

In the present work we performed a feasibility study for the determination of the palladium content in a part of the "Proton" rocket. The presence of palladium was detected independently from our method using the X-ray fluorescence analyzer. We could confirm the presence of palladium in the sample by our method and determined its sensitivity on the order of ~ 2 mg/g.

Keywords: Gamma-rays, spectrometry, multichannel analyzer, neutron source, IREN, Proton-M.

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1. Experimental setup

The experimental setup consisted of the pulsed neutron source IREN [1] and gamma-ray detectors NaI(Tl)-array [2]. The scheme of the experimental setup is shown in Fig.1 (left) and the photo of the detection system in Fig.1 (right). The system was installed on beam-line 4 of the IREN neutron source at a distance of ≅11.4 m from the neutron producing target. A 10-cm-thick borated polyethylene (BPE) ring was located in front of the system to protect the gamma detectors from the neutrons scattered on the last neutron collimator. To keep the neutrons scattered on the sample from hitting the detectors, the sample was additionally shielded by a cylindrical screen of 10-mm-thick boron carbide (B_4C) enriched to 94% of ^{10}B.

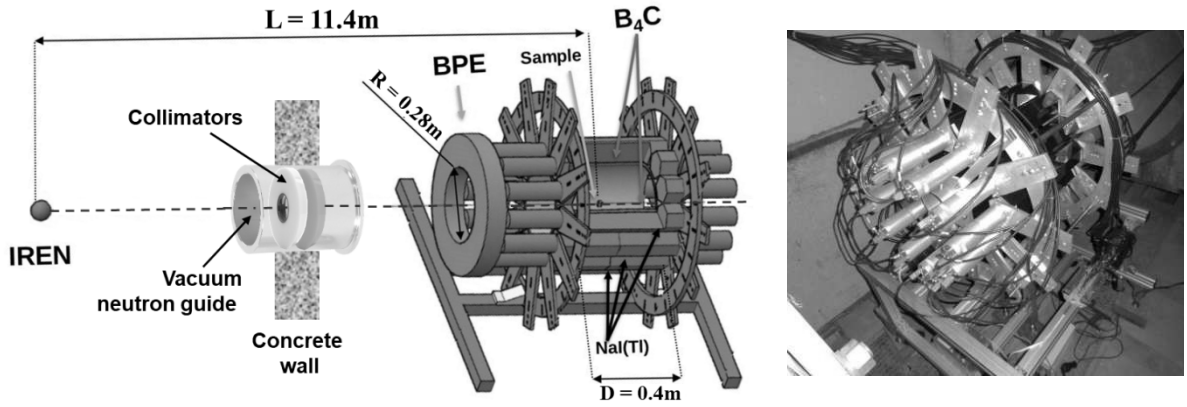
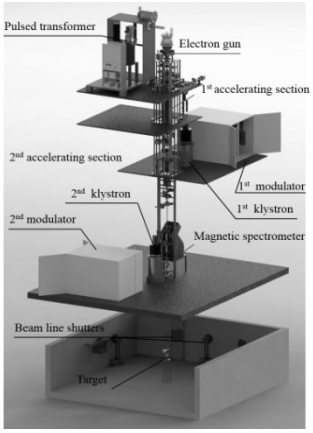


Fig. 1. The scheme of the experimental setup (left) and the photo of the Romashka gamma-ray spectrometry system (right).

The IREN is a pulsed source of resonance neutrons produced by an electron accelerator and a nonmultiplying neutron-production target. With a pulsed source, one can determine the incident-neutron energy by time-of-flight (TOF) method and measure the energy-dependent neutron–nucleus cross sections and related quantities. In particular, one can probe the elemental and isotope composition of a sample by characteristic neutron resonances [3]. In Fig. 2 is shown the schema of the neutron source IREN and some of its current characteristics, at the moment of measurements of sample, as well as those of the new project, which will be realized in the future.



Current characteristics:	**New project:**
• pulsed electron beam current – 2.0 A • electron energy – 40 MeV • pulse width – 100 ns • repetition rate – 25 Hz • integral neutron yield $(3\div5)\times10^{10}$ n/s.	• 200-MeV linear accelerator LUE-200 with a beam power of ~10 kW • Subcritical neutron multiplying target • Integral neutron yield of ~10^{15}n/s and pulse width of ~0.6 μs.

Fig. 2. The scheme of IREN and its characteristics.

2. Sample

We analyzed a 120-g rocket-engine component provided by the Roscosmos Corporation that was later cut in two nearly equal parts (see Fig. 3). Of these, only one part with a mass of ~60 g contained Pd in its solder, as was earlier established by an X-ray fluorescence analysis carried out at the Institute of Physical and Technical Problems (Dubna). As a control sample for measuring the Pd abundance in the solder, we used a 5-g palladium foil with a natural isotope composition.

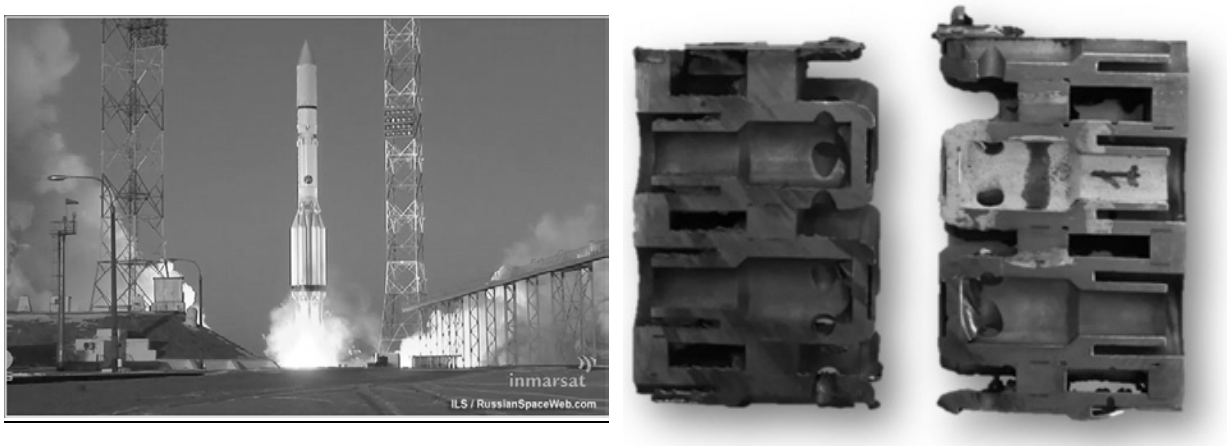


Fig. 3. The Proton-M rocket (left) and the photo of the part of the rocket engine (right). The piece containing palladium is marked as "1".

3. Energy calibration of the time-of-flight spectra

The time-of-flight (TOF) spectra were calibrated using a 5g foil from natural Pd as a control sample. One of the TOF spectra is shown in Fig. 4, where the resonance-neutron energies, corresponding to peak positions, are quoted. We identified the resonances at energies 11.79, 13.22, 25.15, 55.21, and 77.71 eV corresponding to the neutron radiative capture by ^{105}Pd nuclei, and at 33.10 and 90.81 eV – by ^{108}Pd nuclei.

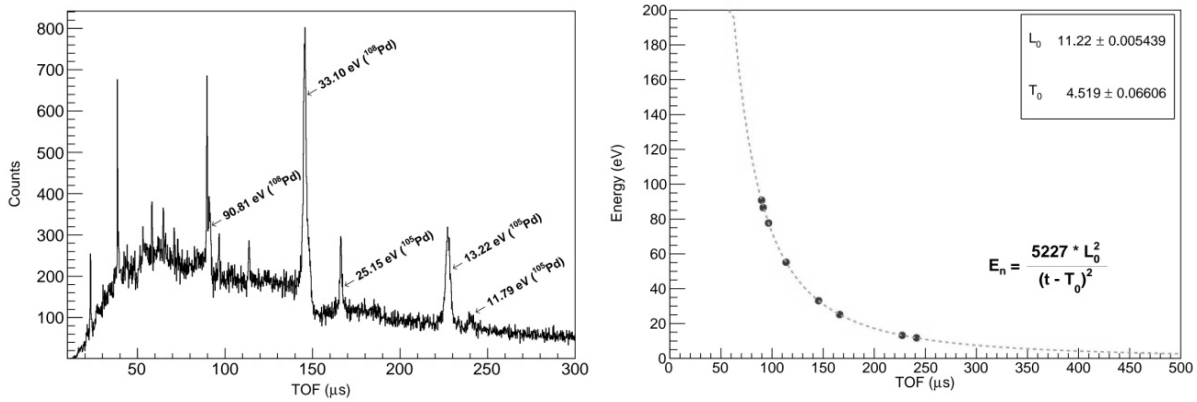


Fig. 4. Time-of-flight spectrum (left) obtained for the Pd control sample and Energy calibration of Time-of-flight spectra (right).

4. Measurements and results

The two samples from the rocket engine were measured during 45 hours each. The mean neutron intensity and the pulse width of the IREN neutron burst amounted to some 3×10^{11} s^{-1} and 100 ns, respectively. The results of the measurements for multiplicity 5 of

coincident signals from γ-detectors are shown in Fig. 5. The solid and dashed curves are the spectra obtained with the palladium-free and palladium-containing samples, respectively; the dotted lines are the spectra from the 5g palladium sample. Left side of the figure shows the full energy range, right side is the region around 13.22 resonance of ^{105}Pd.

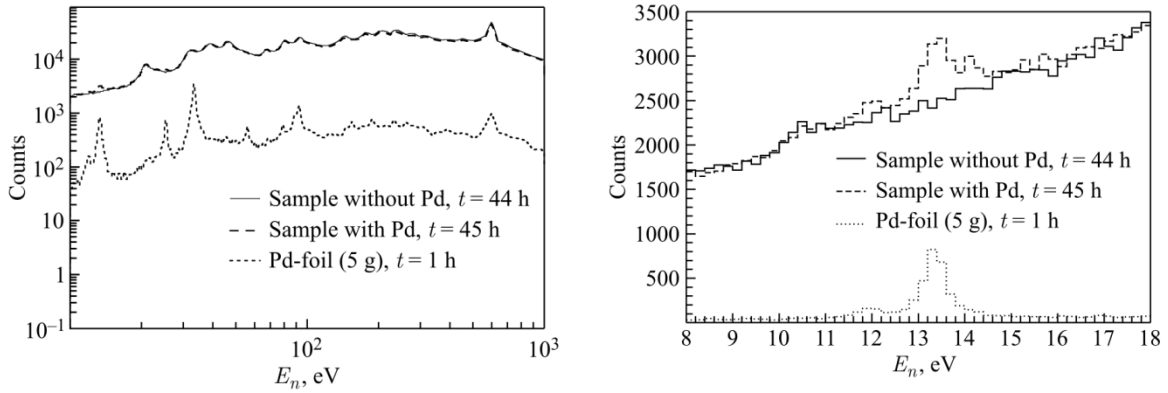


Fig. 5. Total number of 5-fold coincidences from any of the 24 γ-detectors, as a function of neutron energy in the 9–1000 eV range (left) and in neutron energy range of 8–18 eV (right) for the samples with and without palladium, and for the 5g palladium sample.

In the 60-g palladium-containing sample shown on the right in Fig. 3, the amount of Pd was determined by the yield of γ-quanta in the 13.22-eV resonance of the ^{105}Pd isotope. The thus estimated Pd mass content of the investigated sample, obtained using γ-detector coincidences with multiplicities from 4 through 7, is shown in Fig. 6. Upon averaging over the 4–7 range of the coincidence multiplicity, the Pd mass content is estimated as m_{Pd} = 0.1002 ± 0.009 g.

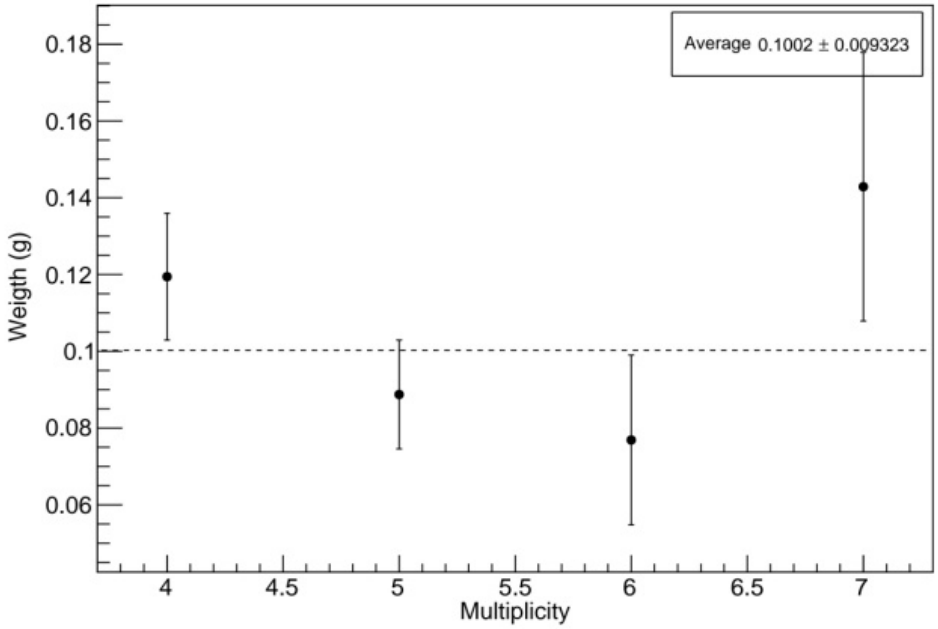


Fig. 6. Values of Pd mass content of the sample obtained from the neutron-energy spectra of coincidences with multiplicities of 4, 5, 6, and 7 (points) and their mean value (dashed line).

5. Conclusions

The presence of Pd in one of the samples was proved and its content was determined. Further improvement of the experimental conditions will increase the sensitivity of this method and may allow performing elemental prompt-gamma resonance neutron capture analysis of bigger samples and more complex substances.

References

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