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STUDY OF NEUTRON INDUCED OUTGASSING FROM TUNGSTEN ALLOY FOR ATLAS FCAL

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The use of sintered tungsten alloy slugs as absorber in the ATLAS Forward Calorimeter (FCAL) raised concern that it could possibly poison the liquid argon during the detector operation in the hard radiation environment expected at LHC. A vacuum container filled with tungsten slugs was exposed to the fast neutron fluence of $1.5 \cdot 10^{16}$ cm^{-2} at the IBR-30 reactor of JINR, Dubna. The residual gas pressure was analysed. The study was completed by mass spectrometer measurements. The irradiation and measurements were carried out at room temperature. An upper limit value of 0.1 ppm was determined for the pollution of liquid argon in FCAL due to outgassing from tungsten slugs under irradiation.

The investigation has been performed at the Laboratory of Particle Physics, JINR

Исследование индуцированного нейтронами обезгаживания вольфрамового сплава для форвард-калориметра детектора ATLAS

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При использовании кусков спеченного порошка вольфрамового сплава в качестве поглотителя внутри форвард-калориметра детектора ATLAS возникает вопрос о возможном загрязнении жидкого аргона в радиационных условиях, ожидаемых на коллайдере LHC. В связи с этим вакуумный контейнер, заполненный вольфрамовыми образцами, облучался быстрыми нейтронами реактора ИБР-30 до полного флюенса $1,5 \cdot 10^{16}$ см$^{-2}$, и проводился масс-спектрометрический анализ остаточного газа в контейнере до и после облучения. Облучение и измерения выполнялись при комнатной температуре. Определен верхний предел в 0,1 ppm для загрязнения жидкого аргона в форвард-калориметре, соответствующий обезгаживанию образцов под действием облучения.

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1. INTRODUCTION

The forward and end-cap regions of the ATLAS detector will be subjected to adverse radiation fields. The expected value of the neutron fluence at high pseudo-rapidity (the FCAL region) is about 10^{16} cm^{-2} for 10 years of LHC operation at high luminosity. Sintered tungsten alloy slugs will be used as an absorber material of the FCAL. There was concern that the calorimeter signal could be reduced by electronegative impurities present in the liquid argon as a result of the outgassing from the tungsten slugs under irradiation.

The «warm» tests at room temperature were carried out while the facility for irradiation tests at cryogenic temperatures was under construction. The effect of neutron irradiation on the rate of outgassing from the tungsten slugs placed into the vacuum container has been studied. Usually, the outgassing processes are more visible at higher temperatures, and outgassing rate measurements at room temperature, for instance, may serve as upper limit estimates on outgassing rate at liquid argon temperature. The residual gas pressure in the compartment with the tungsten slugs was monitored during a long period of time before and after irradiation by fast neutrons at the Dubna reactors. The composition of the gas was studied by means of a mass spectrometer.

2. SETUP OF THE EXPERIMENT

The sintered tungsten slugs of the FCAL calorimeter were placed into the vacuum container to be exposed to the beam of fast neutrons. The container schematic drawings are shown in Fig. 1.

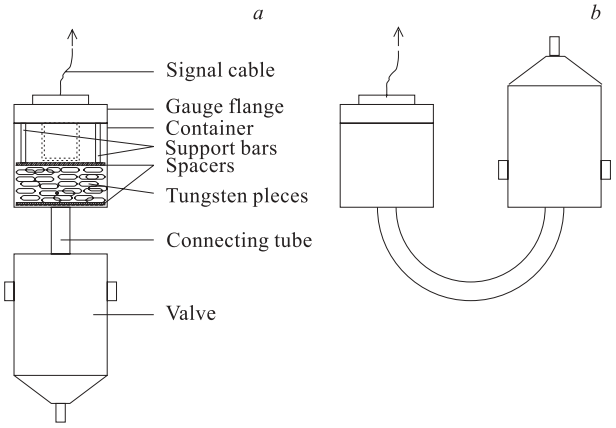

Fig. 1. Schematic drawings of the vacuum container used for *a*) the first and *b*) the second run of irradiation

The container of 200 cm^3 volume was equipped with an ionization gauge to measure the residual gas pressure in the interval of $(10^{-11} \div 10^{-1})$ torr. Conflat copper gaskets were used at all flanges. This allowed us to bake the container with the tungsten samples and to achieve a good level of initial vacuum by means of the BALZER turbo-molecular pump.

Actually, two irradiation runs took place. The first run was carried out at the IBR-2 reactor and the second one at the IBR-30 reactor of JINR, Dubna. During the first run, we obtained only the very upper limit on outgassing from the sintered tungsten samples because of air leakage in the valve of the vacuum container. It was confirmed later on by measurements of the residual gas composition and outgassing rate behaviour. The leakage was caused by small fragments of tungsten slugs (about 1 % of 700 g total weight) which reached the area of the gasket and screened the valve compression. The fraction of fragments in the second run was much less — about 0.2 % of the same total weight. Also, the tube connecting the container and the valve was bent in order to prevent dust motion. Finally, no leakage into the vacuum container was found. This was checked with the mass spectrometer measurements.

2.1. The Mass Spectrometer MS-25. We used the quadrupole mass spectrometer MS-25 («Vacuum», Prague) to identify the components of the residual gas in the vacuum container. It allows one to measure the composition of the gas probe in a wide interval $(2 \div 200)$ of atomic masses with high resolution and sensitivity. The ionization by electrons preserves up to 75 % of the H_2, N_2, CO, CO_2 — like combinations and light organics. The turbo-molecular pump of the spectrometer is working permanently and the gas probe for measurements is taken via the gas dosing valve. The pressure in the analyzing volume is changing with time. The measured values of the ion current (in A) are divided by the pressure value (in mbar) of the analysed gas probe in order to compare the results of various measurements carried out by means of the mass spectrometer.

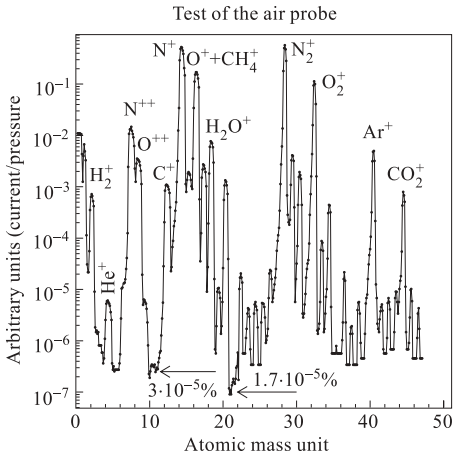

Fig. 2. Composition of the air probe in the operation room. Arrows indicate the mass spectrometer sensitivity level over some specific atomic mass regions

The composition of the air probe taken in the laboratory is shown in Fig. 2. All significant peaks are identified reliably and the sensitivity of the mass spectrometer is indicated by arrows in Fig. 2. Below, we will superimpose this «standard» picture on the figures obtained from the residual gas measurements. This will help one to recognize the presence (if any) of air leakage. In the Table, the composition measured with the mass spectrometer is compared with the standard

Table 1. Comparison of the mass spectrometer measurements with the «standard» air probe composition

A (a.m.u.)	Component	Measurements (%)	Standard (%)
4	Helium	$6 \cdot 10^{-4}$	$5 \cdot 10^{-4}$
18	Water	0.7	1.6
28	Nitrogen	79.3	78.1
32	Oxygen	19.3	21.0
40	Argon	0.5	0.9
44	CO_2	0.08	0.03

air probe composition. There is a fair agreement for the main components of the known «tabular» probe. The admixture of water is sensitive to the procedure followed for the setup preparation and the difference observed in the Table has no relevance for the present case.

3. PREPARATION OF THE SAMPLES FOR IRRADIATION

First, the empty container with the ionization gauge was baked at 400 °C for 5 hours with pumping by means of the diffusion pump equipped with a cryogenic trap. The gas composition in the empty container was measured three days after the annealing procedure. Results are shown in Fig. 3. Contrary to the air probe where fine steps measurements as a function of the atomic mass value were performed, we have carried out the residual gas measurements only around the most significant peak positions. A limited number of masses were used because of the small amount of residual gas in the container and the permanent pumping of the mass spectrometer analyzing volume (the pressure in the analyzing volume of the spectrometer has varied from $5.0 \cdot 10^{-8}$ to $2.2 \cdot 10^{-8}$ torr during the fast measurement procedure, although the background pressure remained stable at the level of $1.1 \cdot 10^{-8}$ torr).

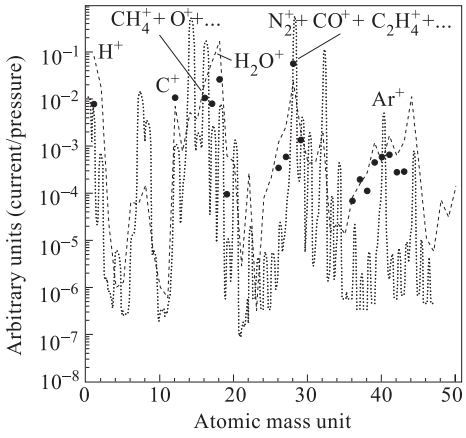

Fig. 3. Composition of the residual gas inside the empty container (filled circles). The dashed line shows results for the residual gas (the spectrometer background) and the dotted line represents the air probe composition measured earlier

It is seen that outgassing from the inner surface of the container into the vacuum volume gives a composition similar to that of the residual gas in the analyzing volume of the mass spectrometer and differs from the air probe composition. So, one could conclude at that stage that the vacuum container had suffered no air leakage. It has been cleaned, annealed, ready for vacuum tests.

Then, the tungsten pieces (with no additional cleaning) were loaded into the container for annealing (with pumping) at high temperature. During the first ten minutes of the annealing procedure, when the temperature grew up to 195 °C, the pressure inside the container increased by $0.75 \cdot 10^{-2}$ torr. Evidently, both — samples and ionization gauge, accumulated some substances (namely, water vapour), while the container was open and slugs were in contact with air.

In order to investigate the radiation effect on the outgassing rates more precisely (at higher vacuum level), we heated the compartment with the tungsten slugs at 400 °C for 8 hours. In Fig. 4, the composition of the residual gas after the annealing procedure is presented. The gas composition during and after the annealing is similar, and it is quite different from the composition of the air probe presented by the dotted line. This kind of desorpted gas is typical for vessels with cleaned and annealed metallic surfaces.

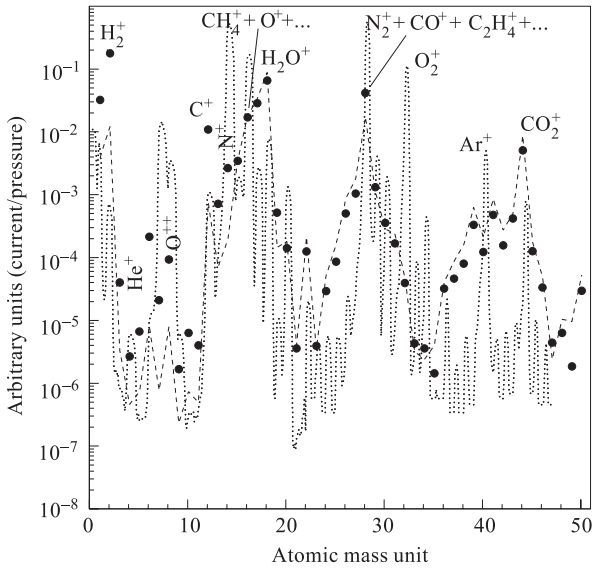

Fig. 4. Gas composition after the annealing procedure (filled circles). The dashed line is the spectrometer background and the dotted line represents the air composition measured earlier

4. RESULTS OF THE IRRADIATION RUN

After the mass spectrometer measurements, the container was pumped out to a high vacuum level and locked. The pressure of the residual gas in the container was measured several times before the irradiation run and after it. The results of the pressure measurements as a function of time are shown in Fig. 5 by filled circles. The outgassing rate behaviour with time is presented in the insertion as well.

The container was mounted very close to the moderator surface of the IBR-30 reactor active core in order to increase the neutron dose rate. The total fast neutron fluence ($E_n > 100$ keV) of $1.5 \cdot 10^{16}$ cm^{-2} was collected in four days of the reactor operation. It was not possible to make the pressure measurements during the irradiation time at the IBR-30 reactor. Thermal neutrons induced high activation of the container (4 Roenthen/hour) making access and handling impossible.

The period of the reactor operation is marked in Fig. 5. The pressure measurements were started three weeks after the irradiation because of the induced activity. These results are also presented in Fig. 5. *Generally, no effect of irradiation is seen.*

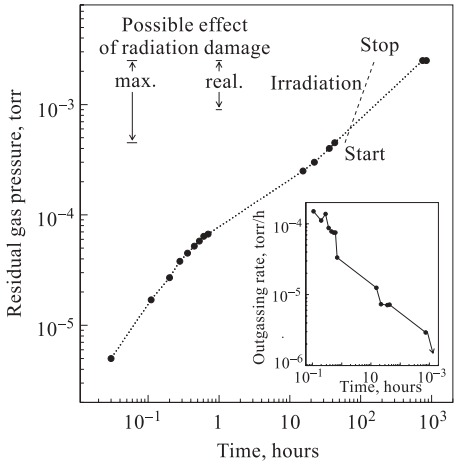

Fig. 5. Residual gas pressure inside the vacuum container as a function of time. The figure inserted shows the outgassing rate behaviour over the same period of time

Nevertheless, one could obtain an upper limit on a possible irradiation effect by assuming that the whole amount of gas released in the container was solely due to irradiation. This limit is marked in Fig. 5 as «maximal» and shown by the dashed line. The pressure has grown by $2.0 \cdot 10^{-3}$ torr. For simplicity let us consider the worse situation assuming that oxygen is the dominant component (this will not change results significantly). The additional 0.002 torr of partial pressure could be provided by 0.75 μg of oxygen gas. For the volume ratio of W:LAr = 5:1 expected in the FCAL module [1], the upper limit on the LAr pollution could be estimated as 0.07 ppm after a fast neutron fluence of $1.5 \cdot 10^{16}$ cm^{-2}. A more realistic (and smaller!) estimate could be obtained by assuming some «natural» growing of the pressure due to outgassing independent of the irradiation. Such an estimate marked in Fig. 5 as «realistic», for the linear increase of pressure, gives a value 20 % less for the upper limit.

Figure 6 shows the residual gas composition measured three months after the irradiation. Such a delay was caused by the high induced activity of the container and of the tested samples.

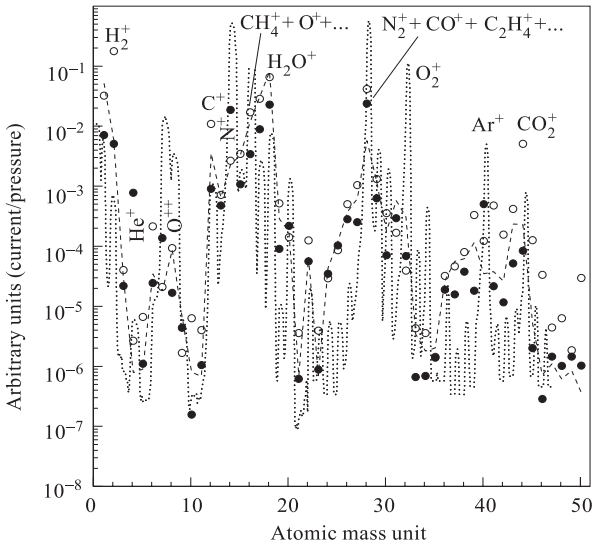

Fig. 6. Residual gas composition measured three months after the irradiation (filled circles). The dashed line is the spectrometer background and the dotted line represents the air probe measured earlier. The measurements carried out before the irradiation (see Fig. 4) are presented (open circles) for comparison

We have not seen a significant difference between the data taken before and after the irradiation. Thus, the electronegative substances (if any) are not dominating after irradiation, and the upper limit is overestimated.

5. CONCLUSION

We have not found considerable influence of the irradiation on outgassing of the sintered tungsten slugs after a fast neutron fluence of $1.5 \cdot 10^{16}$ cm^{-2}, which corresponds to 10 years

of LHC operation. The upper limit on the possible pollution of the liquid argon caused by the irradiation of the tungsten slugs in FCAL was defined to be less than 0.1 ppm. This value of the poisoning rate is quite acceptable. The result is in agreement with a low value of the «natural» outgassing rate measured at the University of Arizona [2].

One has to note that some important aspects are ignored in the estimate based on the irradiation at room temperature. In particular, low mobility of molecules in cold and the contribution from nonelectronegative components could make this estimate even smaller. The measured values of the outgassing rate are also influenced by the preparation procedure of the tested compartment, the ionization gauge and the tungsten slugs themselves.

The vacuum measurements at room temperature could supplement adequate tests performed under cold conditions. The next irradiation tests carried out later at liquid argon temperature [3] confirmed the positive conclusion about the negligible poisoning of liquid argon by the tungsten alloy slugs used by the ATLAS FCAL.

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