Experimental leak tests of gas-filled instruments using automatic leak detection

S A Bushin and S S Galkin

Dukhov Automatics Research Institute (VNIIA), Federal State Unitary Enterprise, 22 Suschevskaya ul, Moscow 127055, Russia

E-mail: vniia4@vniia.ru

Abstract. The experimental leak tests of several gas-filled instruments (dischargers) are described. The tests are based on accumulation mass-spectrometry. The technique is implemented by means of VNIIA-developed and produced prototype for automated final leak test facility (FLTF). The tests show that a part of tested instruments have leakage within the range of filling gas (neon) equal to 1.5·10^{-12} – 10^{-7} Pa·m^{3}/s.

1. Introduction

In order to maintain a gas composition either inside a vacuum instrument or in gas-filled instrument, their enclosure has to be leak-tight and it depends on quality and mechanical integrity of leak-tight welded joints of parts and materials.

An automated final leak test facility (FLTF) was used for series of tests to check the key requirements of leak tightness testing technique developed to control the filling (control) gas leaks in small-sized gas-filled instruments [1–4].

An object to be tested is a process prototype for ceramic and aluminum alloy gas-filled instruments with a tube and basing (at the end of the tube there is a compound bead) fabricated in different time.

The following equipment was used for tests: 1) FLTF prototype [5] with neon detection limit as much as 5·10^{-14} Pa·m^{3}/s and 2) Adixen ASM142 Graph D leak detector [6] with detection limit as much as 5·10^{-13} Pa·m^{3}/s with accessories.

2. Testing technique

Leak tightness of gas-filled instruments was tested using an automatic final leak tightness testing technique [7, 8]. It is a leak tightness detection based on accumulation mass-spectrometry. When applying the detection like this the tested instrument is placed in an accumulation chamber. A filling gas (neon) which exhausts from the instrument accumulates in the chamber. When a gas portion is accumulated and transported to the ionization area of gas-detector, mass-spectrometer registers gas pressure. A gas leak is detected by mass-spectrometer signal, which is a function of time, provided that the tested instrument and gas-detector are isolated from the exhausted filling gas.

The instrument leak rate is calculated by expression (1):

$$Q = \frac{\Delta I \cdot V_z}{S \cdot \tau},$$

(1)
where: \( Q \) is a leak rate, \( \text{Pa} \cdot \text{m}^3/\text{s} \); \( \Delta I \) is a mass-spectrometer signal increment when the accumulation chamber is re-filled with gas (Figure 1), \( A \); \( V_c \) is a total volume of free space within the measurement system and the accumulation chamber, \( \text{m}^3 \); \( S \) is a sensitivity factor of measurement system, \( \text{A/Pa} \); \( \tau \) is an accumulation time, \( \text{s} \).

Four groups of instruments were tested in four stages:

Group I – The instruments were tested for leaks with FLFT;

Group II – The instruments were re-tested after being exposed to \((200 \pm 10) \, ^\circ\text{C}\) for 3.5 hours and exhausted at the same time;

Group III – The testing technique [7] was applied to the instruments having been pressurized in neon up to pressure \( \approx 1\ \text{atm.} \), which was directly set while the instrument is in the chamber without its removal and exposure to the ambient air for set exposure time. All instruments including test ones were pressurized (see Note);

Note:

1) **Pressurization** is a technique applied to leaky gas-filled instruments being stored. The procedure is used to restore an initial pressure of filling gas within the instruments to be unsoldered and removed from the pack rack in order to improve measurements of leak rate.

2) Two based instruments were used as test ones at different times. The first one was applied to the first group of instruments to be tested and the second – instead of the first – for tests of all other instruments.

Group IV – The instruments with neon leak more than \( 1 \cdot 10^8 \, \text{Pa} \cdot \text{m}^3/\text{s} \) were additionally pressurized in helium and then tested for leak tightness using helium mass-spectrometer leak detector.

![Figure 1](image_url)

**Figure 1.** Typical frame of QMG 220 mass-spectrometer signal at mass 20 for filling gas leakage.

Ion current increment per mass 20 for each instruments, \( \Delta I' \), was calculated as difference of existing peak amplitude \( I_i \) with respect to the system background and ion current increment, \( \Delta I_i \). The last parameter is described by mass-spectrometer response to expansion of gas portion accumulated within the inspection equipment volume. The mass-spectrometer response is also overlapped by gas-detector response to relatively intensive gas escape from the surface of the tube with compound bead at the end. \( \Delta I' \) as calculated by expression (2):

\[
\Delta I' = I_i - \Delta I_i. \tag{2}
\]
For comparative analysis of test results obtained using accumulation mass-spectrometry (FLTF) and a sniffer probe of ASM142D Graph leak detector, a neon leak rate ($Q_{Ne}$) was converted into helium leak rate ($Q_{He}$) by expression (3):

$$Q = Q_{Ne} \cdot \frac{M_{Ne}}{M_{He}},$$

where: $Q_{Ne}$ is a neon leak rate, Pa·m$^3$/s; $M_{Ne}$, $M_{He}$ are $Ne$ and $He$ molecular masses.

3. Test results

The tests of group I (4 instruments) showed a leak rate of 1.2·10$^{-12}$ Pa·m$^3$/s (allowable leak rate).

Group I instruments were vacuum-heated. When the instruments were cooled, the neon portions were in each of accumulation volumes up to pressure 1 atm. Figure 2 (a) and (b) shows curves demonstrating the test results for two instruments. Neon leak rates were 7.12·10$^{-12}$ Pa·m$^3$/s and 1.99·10$^{-8}$ Pa·m$^3$/s (large leak); a leak rate for other instruments in the group does not exceed 1.2·10$^{-12}$ Pa·m$^3$/s.

In order to find a leak location in the instrument with large leak, the instrument after being removed from FLTF accumulation chamber was placed in special in-process storage box (V ≈ 1.5 l) and then exhausted and pressurized in He$_4$ at pressure ≈ 2.5...3 atm. for 35 days and tested by sniffer probe of helium leak detector. As a result, the location of compromised integrity on the instrument enclosure was found. It was a surface section of disk-shaped electrode with tube welded at the base. A leak rate registered by leak detector was (2.2...2.6)·10$^{-7}$ Pa·m$^3$/s.

The test of group II (6 instruments) did not reveal leaks. After heating at (190...195) °C the leaks were also not detected.

The test of group II of instruments being neon-pressurized at 1.1 atm. for 42 hours 50 minutes revealed a leak rate (after approximation and extrapolation while the instrument was unsoldered) (see Figure 3) equal to $Q=9.95\cdot10^{-8}$ Pa·m$^3$/s for Ne and $Q=2.22\cdot10^{-7}$ Pa·m$^3$/s for He$_4$, respectively.

The instrument when removed from the accumulation chamber was placed in special in-process storage box and pressurized in helium at pressure ≈ 1 atm. for 30 days. A sniffer probe of leak detector was then used to find a leak location, which was a ceramic and aluminum alloy joint directed towards the tube and a leak rate was (1...2)·10$^{-7}$ Pa·m$^3$/s.

**Figure 2.** Frames from single-piece leak tightness test record for instrument 3 (a) and 4 (b).
The test of group III (6 instruments) showed that only two of them after being pressurized in neon (at 1 atm. for 20h 41 min) had the leak traces. A total leak rate was $6.03 \times 10^{-12}$ Pa·m$^3$/s ($1.34 \times 10^{-11}$ Pa·m$^3$/s for H$_2$) and $2.39 \times 10^{-11}$ ($5.34 \times 10^{-11}$ Pa·m$^3$/s for H$_2$), respectively.

The test of instruments (5 pcs) from group IV after first and second stage showed that a leak rate did not exceed an allowable value. Three instruments revealed a higher value only after neon-pressurization at 0.995 atm, for 30 h 47 min. Their leak rate for neon was $5.57 \times 10^{-11}$ Pa·m$^3$/s, $6.96 \times 10^{-11}$ Pa·m$^3$/s and $2.04 \times 10^{-11}$ Pa·m$^3$/s, respectively (see figure 4).

![Figure 3](image_url)

**Figure 3.** Approximation (b) and extrapolation (a) curves for variable leak rate during leak test of neon-pressurized instruments of group II with large leak in mode Large Leak Rates

The instruments of group IV during leak tests revealed a leak rate higher than allowable one. A leak rate for three instruments subject to single-piece leak tightness tests was $1.32 \times 10^{-10}$ Pa·m$^3$/s, $9.63 \times 10^{-11}$ Pa·m$^3$/s and $1.83 \times 10^{-10}$ Pa·m$^3$/s (see figure 5(a)).

![Figure 4](image_url)

**Figure 4.** Amplitude behavior at mass 20 for six neon-pressurized instruments including a reference one (ref.) and an instrument X [with leak rate equal to $4.5 \times 10^{-9}$ Pa·m$^3$/s for comparison] during single-piece leak tightness tests.
Note that the leak rates in figure 5(a) are much different from those in figure 5(b) (the processed data of large leak rates). The leak rate history in figure 5(b) (instrument X) is almost unchanged and a leak rate is \( \approx 4.5 \times 10^{-9} \text{ Pa}\cdot\text{m}^3/\text{s} \). It indicates an actual leakage and is proved by relevant calculations and observations over the instruments for relatively long time (since August 2013) unlike the leak test results for instrument A, B, C in figure 5(a).

It should be noted that the instrument external surface cleaned with ultrasound when manufactured and made of ceramics (70% of surface) has a grained physical structure contrary to its geometric area. There can be microvoids and holes which can quite intensively inlet the filling gas in the vacuum volume after pressurization. Moreover, the micro-bubbles on the compound bead spread over plasticizer and partially extricated and filled during pressurization can greatly distort the results. Point-by-point measured leak rate in 10 – 15 minutes after pressurization reaches about \((1...3) \times 10^{-9} \text{ Pa}\cdot\text{m}^3/\text{s}\) which is a level of integral gas leak rate from the surface. In 5–8 hours after high vacuum evacuation a level of leak rate drops to about \((0.5...2) \times 10^{-10} \text{ Pa}\cdot\text{m}^3/\text{s}\). The initial background (before pressurization) is resumed in 1.5–3 days with total evacuation time per a shift equal to 3–5 hours.

![Figure 5. Curves of point-by-point neon leak tests made at different time on three pressurized instruments A, B, C (a) and a leaky instrument X (b), with approximation (solid line) and extrapolation (dash line).](image-url)
Upon the above results there are no reasons to say that the instruments A, B, C are leaky.

4. Summary

The leak tightness tests of instruments carried out with FLTF had the following results:
  - Two instruments showed a neon leak rate as much as $1.99\times 10^{-8}$ Pa·m$^3$/s, $9.95\times 10^{-8}$ Pa·m$^3$/s, respectively;
  - Eight instruments demonstrated a neon leak rate (after pressurization) as much as $1.5\times 10^{-12}$ to $10^{-11}$ Pa·m$^3$/s.

Other instruments had a leak rate within an allowable value.

5. Result analysis

The tests performed showed that detection of the filling gas leak rate using FLTF enabled to find both relatively large ($10^{-7}$–$10^{-9}$ Pa·m$^3$/s) and small (less $10^{-15}$ Pa·m$^3$/s) leakages. But the leak test results will be reliable if a number of requirements are followed: the leak tests should be conducted right after pressurization of instruments and before coating the external surfaces of enclosure. If the requirements are ignored, prior to leak tests additional measures should be undertaken: the instruments have to be vacuum-heated at high temperature to degasify the external surfaces and exposed to high pressure in filling gas medium to restore pressure inside the leaky enclosure. So the leak tests prove that only two instruments are leaky and eight instruments can have a probable leakage. Other ten pieces are considered to be leak-tight.

6. Conclusion

The key requirements of filling gas leak tightness testing technique applied to small-sized gas-filled instruments using FLTF were checked. The technique is showed to enable highly sensitive tests of small-sized gas-filled instruments.

References

[1] Bushin S A 2011 Materials of 18-th Research Conference “Vacuum Science and Technique” (Sudak)
[2] Bushin S A and Galkin S S 2014 Vacuum Equipment and Technology 1 39–41
[3] Bushin S A 2017 Collection of papers of 21-st All-Russian Conference for Non-destructive Tests and Technical Diagnostics (Moscow) pp 275–88
[4] Bushin S A and Kozlovskaya T I 2017 Proc. 15th Asia-Pacific Conference on Non-Destructive Testing (APCNDT2017) (Singapore) URL: http://www.ndt.net/events/ APCNDT2017/app/content/Paper/31_Bushin_Rev4.pdf.
[5] Smirnov G A, Khapov A S, Kozlovskaya T I, Bushin S A, Bakanov A P, Chernykh E V, Galkin S S RF Patent No. 101072 for Prototype G 01 M 3/02, 3/26. Automated Final Leak Tightness Facility 28.11.16
[6] Adixen Vacuum Products by Pfeiffer Vacuum, France
[7] Neon Leak Tightness Tests Instruments: Technique No. PM T401/22-2012 (Moscow: VNIIA) 2012
[8] Bushin S A, Kamenev V G and Galkin S S 2015 Vacuum Equipment and Technology 2 77–82