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VACUUM GAUGE VERIFICATION METHODOLOGY FOR VACUUM COATING DEPOSITION PROCESSES

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Abstract

The paper analyses vacuum gauge accuracy problem, which leads to the pressure deviation in vacuum nano-coating deposition process. Pressure deviation leaves significant impact on vacuum coating properties and quality. Therefore, new vacuum gauge verification methodology has been developed and the results are presented in this paper. Methodology consists of combination of several verification methods, including a method of constant pumping speed, method of comparison to the reference gauge, Student's t-distribution and other methods and approaches. In the following research, several experiments with ionization and diaphragm vacuum gauges were performed to obtain data on their accuracy and to validate thereof. Proposed methodology focuses on vacuum gauge multifunctional verification under manufacturing conditions, resulting in correction coefficient implementation for vacuum gauges. Developed methodology determines a necessity and a period of calibration to be needed for each vacuum gauge

Keywords: Verification methodology; vacuum gauge; calibration period; vacuum coating; deposition process

1. Introduction

Vacuum nano-coating deposition is performed using specialized vacuum systems. In order to ensure consistent vacuum nano-coating properties, technological process parameters must remain unchanged and controlled at certain level. Vacuum gauges are used to measure a pressure with the vacuum chamber in result of which proper vacuum level is controlled. Vacuum gauge accuracy may leave a significant impact on deposition technological process resulting in low nano-coating quality and properties. In order to minimize associated risks two gauges are used in vacuum systems as a standard. Nevertheless, after certain operation time pressure measurements of the vacuum gauges start to vary considerably. Hence a vacuum gauge calibration is commonly used when the pressure measurement accuracy starts to fail and it is impossible to secure necessary nano-coating quality and properties [5]. Some studies analyse vacuum gauge accuracy and calibration period, but there are no clear approaches for vacuum gauge verification under manufacturing conditions before calibration operation. [14] There are two particularly important considerations to be made to determine vacuum gauge calibration period [14]. First, has the device been calibrated at least twice before, without adjustment, with one calibration being quite recent? [14]. Second, what measurement uncertainty is needed? For the majority of instruments the recalibration interval is about a year, but it is important that the historical data relates to the device when used in an environment and in a way that is similar to the way it is normally used [14]. If no historical data is available it is not possible to answer the question [14]. In addition, users have to stop their manufacturing process for at least several weeks during the period of calibration [6]. This is inappropriate in manufacturing conditions.

Moreover, some users are worried about the damage of their vacuum gauge during transportation [6]. Vacuum gauge calibration decision must be made as final step based on reliable information. That's why it is so important to develop vacuum gauge verification methodology. Than vacuum gauges can be tested, adjusted, necessary coefficient implemented and historical data obtained. In the following paper, we are representing multifunctional vacuum gauge verification methodology using custom-made vacuum system. Experiments were conducted using various methods to obtain full-scale data. In the result of the proposed verification methodology it is possible to perform a vacuum gauge analysis and conclude whether a tested vacuum gauge can be used further or calibration must be performed in accredited metrology laboratory.

Main purpose of this paper is to present the new vacuum gauge verification methodology. The new approach allows to acquire a full spectre of vacuum gauge performance data and perform correction on tested vacuum gauge, thus enhancing deposited vacuum nano-coating quality and properties.

2. Discussion of Prior Art

Oxide coating process, such as SiO2 deposition is a reactive process using metallic targets because of cheapness of metallic target in comparison with conductive ceramic targets [2]. Therefore, vacuum deposited coatings of metal oxides have a wide use in various applications [3]. The main problem in the vacuum nano-coating deposition process is unreliable accuracy of vacuum gauge. Incorrect pressure measurements and even small pressure deviations can reduce nano-coating properties and quality. For example, SiO2 nano-coating is very sensitive to pressure deviation. In confirmation of said statement, the experiment was performed where a SiO2 nano-coating was deposited using laboratory vacuum system UV 80 and diaphragm vacuum gauge CDG 025D INFICON®.

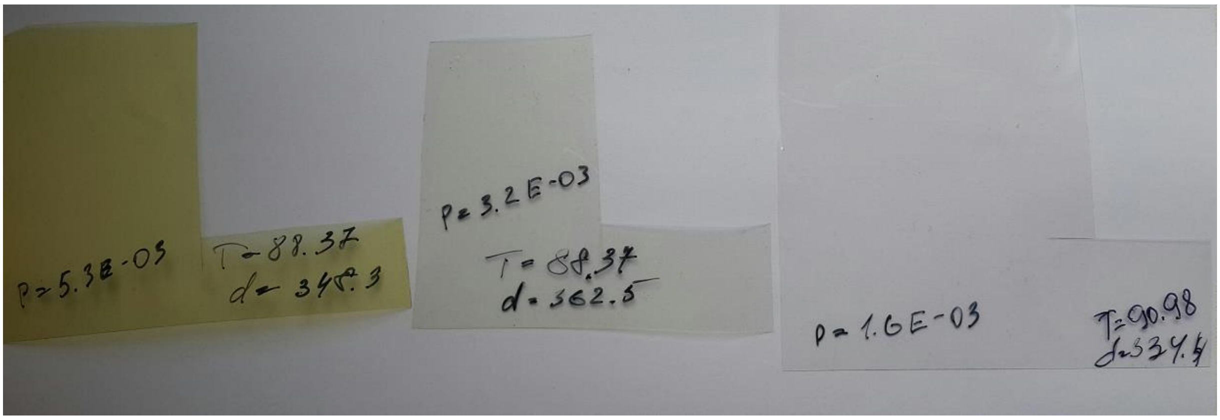

Fig. 1. SiO2 coating samples after vacuum coating deposition

Conducted experiment results are showed at figure 1 and table 1. Light transmission coefficients were measured using MC 122 spectrophotometer, but thicknesses were measured using FILMETRICS F20-UVspectroscopic reflectometer.

No.	Pressure, Torr	Transmission coefficient, %	Thickness, nm
1	5.3E-03	88.37	348.3
2	3.2E-03	88.37	362.5
3	1.6E-03	90.98	334.3

Table 1. Light transmission coefficients and thicknesses of SiO2 coatings under certain vacuum

After the preliminary analyses of experimental results it was concluded that transmission coefficient and coating properties changes depending on the pressure or vacuum value. In the following experiment the transmission coefficient values were above defined standard limit T = 87.5% for oxide coatings. Therefore, the coating quality at 3.2E-03 Torr and 5.3E-03 Torr was unacceptable and did not meet the necessary requirements.

Vacuum coating thickness also must be considered as one of the main parameters. It is well known that the technological process as such is combined from a set of factors and parameters, where a pressure or vacuum is very important. Vacuum gauge verification in certain periods of time should be performed, obtained data should be analysed and correction coefficient/-s should be implemented. The result should be in the form of a reliable and accurate vacuum gauge.

3. Vacuum gauge verification experimental approaches

Vacuum gauge verification using reference gauge. The first step of verification methodology was made using INFICION® diaphragm vacuum gauge CDG 025D and ionization vacuum gauge HPG 400. Any type of gas does not affect diaphragm vacuum gauge and it has high accuracy (inaccuracy is 0.2% of reading) [1, 4, 7]. Unlike CDG 025D, ionization vacuum gauge HPG 400 accuracy is ± 15% of reading [9]. Verification was performed in various pressure ranges, with argon correction coefficient option turned on for ionization vacuum gauge and without it. Experiment was performed using custom made vacuum system, where CDG 025D and two ionization HPG 400 vacuum gauges were verified. Ionization vacuum gauge coefficient was enabled. Argon flow was gradually reduced from 10 sccm to 1 sccm. In the same time a diaphragm vacuum gauge was considered as reference gauge. Results are showed at figure 2.

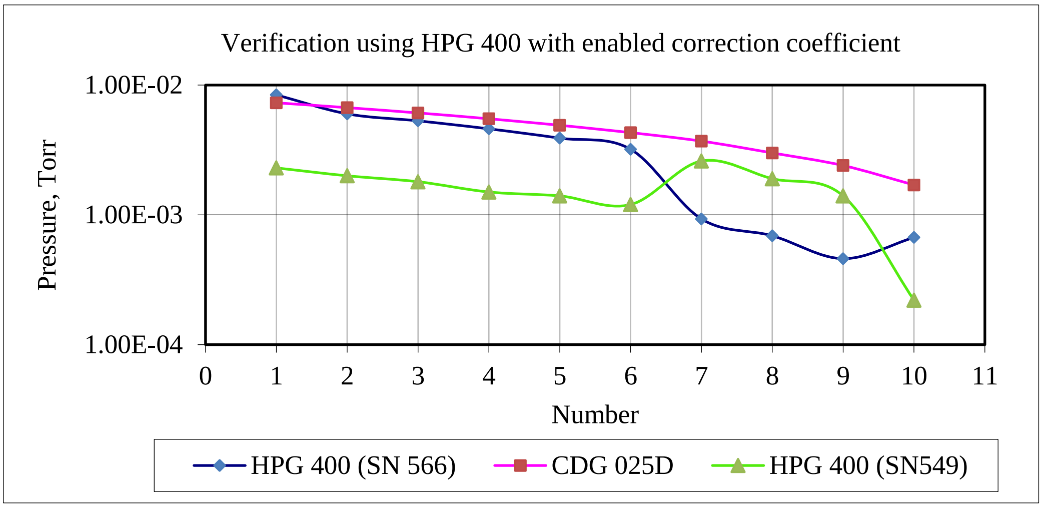

Fig. 2. Performance comparison of HPG 400 and CDG 205D vacuum gauges

After preliminary analysis of the results, it was concluded that the ionization vacuum gauge HPG 400 (SN 549) pressure measurement values differ significantly comparing to the reference gauge. Lack of accuracy exceeds acceptable boundary ±15% of reading, thus no further operations can be performed with this gauge. HPG 400 (SN 566) ionization vacuum gauge pressure measurements and graphic line is more similar to the reference gauge. Ionization vacuum gauge HPG 400 (SN 566) starts to show significant changes in pressure from seventh measuring point to ninth. Identical pressure deviation character was observed in several experiments with disabled argon correction coefficient. Overall ten experiments with enabled and disabled gas correction were conducted and necessary data obtained. This verification experiment combines several approaches such as: testing in different pressure ranges, gradual reduction of gas, and comparison to the reference gauge. All this approaches create vacuum gauge full-scale performance map necessary for the evaluation.

Ionization vacuum gauge correction coefficient test. Vacuum gauge built-in correction coefficient option may have its own inaccuracy. Accordingly, ionization pressure can be determined by following equation [9]:

$$p_{eff} = K \times pressure\ indicated \tag{1}$$

No.	Argon flow, sccm	HPG 400 Measured pressure (correction off), Torr	HPG 400 Estimated pressure, Torr	HPG 400 Measured pressure (correction on), Torr	Uncertainty, %
1	10 sccm	3.10E-03	2.48E-03	2.32E-03	6.4%
2	9 sccm	2.70E-03	2.16E-03	2.10E-03	2.8%
3	8 sccm	2.40E-03	1.92E-03	1.84E-03	4.2%
4	7 sccm	2.15E-03	1.72E-03	1.55E-03	9.8%
5	6 sccm	1.80E-03	1.44E-03	1.37E-03	4.9%
6	5 sccm	1.50E-03	1.20E-03	1.10E-03	8.3%
7	4 sccm	1.30E-03	1.04E-03	1.02E-03	1.9%
8	3 sccm	9.5E-04	7.6E-04	7.4E-03	2.6%
9	2 sccm	8.0E-04	6.4E-03	6.25E-03	2.3%
10	1 sccm	6.8E-04	5.44E-04	5.35E-04	1.7%

Table 2. Measured and estimated pressures values of HPG 400 vacuum gauge

Ten identical experiments were conducted to check ionization vacuum gauge performance with enabled gas correction coefficient. Experiment results are shown in table 2. After preliminary analysis of experimental results, it was concluded that measured pressure values are lower than estimated pressure values. A maximum value of inaccuracy is 9.8%, which should be considered high because measuring device has inaccuracy ± 15% of reading. This approach shows that measuring device inaccuracy is not the only value to be considered.

Vacuum gauge verification using constant pumping speed. For this experiment, custom-made vacuum gauge verification system was developed and used together with turbo-molecular pump TURBO – V 3K – T for high vacuum and back up mechanical pump НВЗ-100Д for low vacuum. Prior art discusses that pumping speed can be constant at certain point, see figure 3 [8].

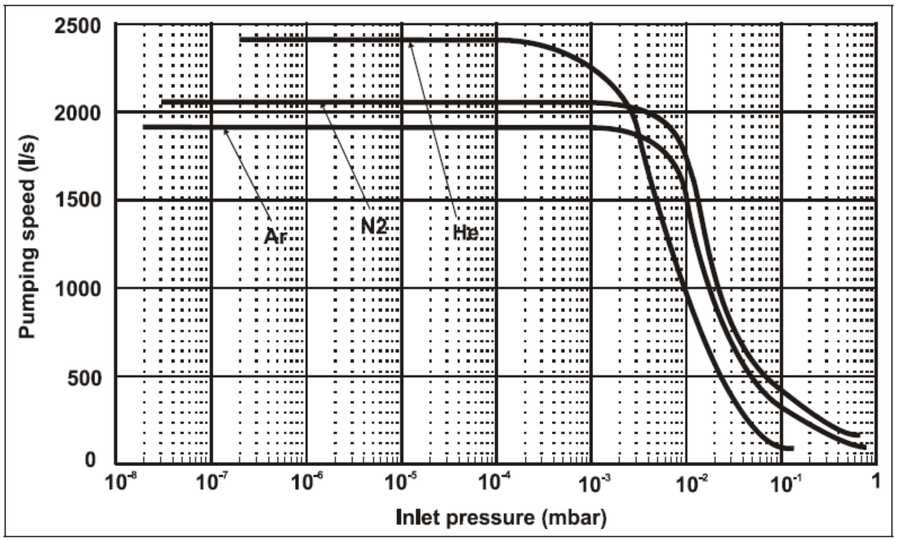

Fig. 3. Pumping speed curves

In this experiment diaphragm vacuum gauge CDG 025D was used to measure pressure within a vacuum chamber and to analyse its performance. Argon (Ar) and Oxygen (O2) gases were fed into the chamber in certain amounts. Argon pumping curve at figure 3 rapidly rises from atmosphere pressure till approx. 10^{-3} mbar, but after that point pumping speed is constant up to approx. $5x10^{-7}$ mbar. Therefore, the experiment was conducted in constant pumping speed range. Experiment results are shown in table 3.

No.	Ar, sccm	CDG 025D, Torr	Estimated pressure, Torr	Inaccuracy, %
1	1 sccm	1.70E-03	Input data	0%
2	2 sccm	2.40E-03	3.4E-03	29.41%
3	4 sccm	3.70E-03	6.8E-03	83.78%
4	6 sccm	4.90E-03	1.02E-02	108.16%
5	8 sccm	6.10E-03	1.36E-02	122.95%
6	10 sccm	7.30E-03	1.7E-02	132.88%

Table 3. Vacuum gauge estimated pressure and inaccuracy

The pressure can be determined by following equation [10]:

$$P = \frac{Q \times K}{S} \tag{2}$$

Where: P is pressure; Q is gas flow amount; S is pumping speed, K is unit conversion value.

Based on the experimental results, which are shown in table 3, it was possible to calculate pumping speed when a pressure and injected gas amount is known. Unit of throughput conversion is necessary for further calculations, thus $sccm = 1{,}27 \times 10^{-2} \frac{torr \cdot l}{s}$ [10].

First measurement in table 3 was taken as input data for pumping speed calculations [10].

$$S = \frac{Q \times K}{P} = \frac{1 \times 0.0127}{0.0017} = 7.47\frac{l}{s} \quad (3)$$

When pumping speed is known it is possible to estimate pressure in the remaining gas flow. Calculated pressure is acquired by using (2) and results are showed in Table 3.

After preliminary analysis of results, we concluded that diaphragm vacuum gauge indicated pressure and estimated pressure values are significantly different. Furthermore, the changes should be linear i.e., pressure value should change twice if gas flow amount is changed twice. Inaccuracy exceed 100% boundary and reached maximum value of 132.88%, which is unacceptable and high. Preforming vacuum coating deposition process using this vacuum gauge may result in poor coating quality.

This experiment proved that it can be performed for fast accuracy check of vacuum gauge when typical pumping curves are known.

Mass flow controller performance experiment. During technological process vacuum gauge measures pressure within vacuum chamber while gas is fed into it. Mass flow controller (MFC) is responsible for gas flow delivery and control. Therefore, it is possible that MFC inaccuracy will influence vacuum gauge indicated pressure. This vacuum system element check is necessary to ensure good deposition process and perform preliminary analysis of obtained data.

MFC verification experiments were performed by separately delivering argon (Ar) and oxygen (O2) in various amounts into vacuum chamber and obtaining pressure measurements. Experiment results are shown in table 4.

No.	Ar, sccm	O2, sccm	CDG 025D, Torr
1	50	0	3,40E-03
2	75	0	5,00E-03
3	100	0	6,68E-03
4	0	50	3,20E-03
5	0	75	4,67E-03
6	0	100	8,09E-03

Table 4. Measured pressure values depending on used gas type

Next step was to perform manipulations with gas delivery system by connecting argon flow channel though O2 mass flow controller. Schematic representation of gas connection establishing through O2 MFC is showed in figure 4.

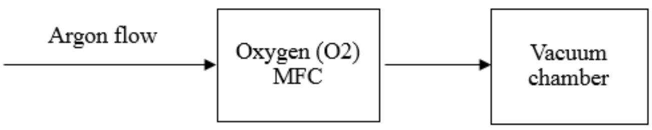

Fig. 4. Experimental gas connection scheme

Since argon gas is delivered through the O2 mass flow controller, gas correction factor must be implemented. For argon, a gas correction factor according to the @MKS data is 1.39 [11]. Obtained experimental results are showed in table 5.

No.	Argon flow value, sccm	O2 converted value, sccm	CDG 025D indicated pressure through O2 MFC, Torr	CDG 025D indicated pressure through Ar MFC, Torr	Inaccuracy, %
1	50	36	3.58E-03	3,40E-03	5,00%
2	75	54	5.26E-03	5,00E-03	4,94%
3	100	72	6.92E-03	6,68E-03	3,47%

Table 5. Vacuum indicated pressure values through different MFC's

After preliminary analysis of obtained data, it was concluded that measured pressure of the diaphragm vacuum gauge can be different delivering the same amount argon or oxygen into the chamber. Gas connection through Oxygen MFC did not result in accurate measurements. It was concluded that oxygen MFC has increased uncertainty. Pressure values should be identical, when argon is delivered into the chamber through Ar and O2 MFC.

Vacuum gauge verification using Student's t-distribution. T-distribution is used to obtain vacuum gauge confidence interval, thus its accuracy is checked. Experiment was performed using ionization HPG 400 vacuum gauge. Argon gas flow equal to 30 sccm was fed into the chamber, after pressure stabilization injection was stopped. When gas injection is stopped, pressure value should return to the initial value.

Pressure inside vacuum chamber was set up 4.05E-4 Torr. Experimental data and estimated values are shown at table 6.

The sample mean can be determined by following equation [12]:

$$\bar{x} = \frac{\sum x_i}{n} = 4{,}1E - 04\ (Torr) \tag{4}$$

Where: $\sum x_i$ – all pressure values, n – number of measurements.

No.	$\mathbf{x_i}$ HPG 400 pressure, Torr	$\Delta \mathbf{x} = \bar{\mathbf{x}} - \mathbf{x_i}$, Torr	$\Delta \mathbf{x}^2$, Torr
1	3.99E-04	+ 0.11E-04	0.0121E-04
2	4.11E-04	+ 0.001E-04	0.0001E-04
3	4.25E-04	+ 0.15E-04	0.0225E-04
4	3.80E-04	- 0.3E-04	0.09E-04
5	4.15E-04	+ 0.05E-04	0.0025E-04
6	4.22E-04	+ 0.12E-04	0.0144E-04
7	4.51E-04	+ 0.41E-04	0.1681E-04
8	4.35E-04	+ 0.25E-04	0.0625E-04
9	3.91E-04	- 0.19E-04	0.0361E-04
10	3.86E-04	- 0.24E-04	0.0576E-04

Table 6. Ionization vacuum gauge measured and estimated pressure values

When the sample mean estimation is complete, then a pressure standard deviation is calculated using following equation [12]:

$$S = \sqrt{\frac{\sum_i^n (x_i - \bar{x})^2}{n-1}} = 0.227E - 04\ (Torr) \tag{5}$$

After standard deviation, confidence interval of the mean is determined. We used 95% confidence level for (*f*) degrees of freedom:

$$f = n - 1 = 9, \tag{6}$$

Where: n is number of measurements. Multiplier $t = 2.26$ for 95% confidence level and 9 degrees of freedom [12]. Confidence interval equation is as follows [12]:

$$\Delta\bar{x} = \sqrt{\frac{S^2}{n}} = \frac{S}{\sqrt{n}} = \frac{2{,}26 \times 0{,}227}{\sqrt{10}} = 0.16223E - 04\ Torr \tag{7}$$

After calculations it was concluded that standard deviation confidence interval is 4.1±0.16E-04Torr. This approach shows vacuum gauge possible inaccuracy interval, which should be considered performing coating deposition process.

Vacuum gauge zero adjustment. As vacuum gauge verification final step, a zero adjustment must be performed. If the vacuum gauge accuracy if very low and exceeds accepted limits, then it is necessary to perform vacuum gauge zero adjustment.

For this experiment diaphragm vacuum gauge CDG025D was selected. The zero can be adjusted via the control button on the same gauge. Pressure in the vacuum chamber must be reduced to a pressure according to the manufacturers table, which is $<5x10^{-6}$ Torr [13].

Fig. 5. Diaphragm vacuum gauge zero adjustment

The gauge was operated for at least 2 hours until signal was stable according to the instructions from manufacturer of the gauge [13]. The final step was to briefly press the zero button with a pin [13]. The zero adjustment runs automatically and LED flashed until the adjustment (≈ 8 sec.) was completed [13]. CDG 025D capacitance gauge experimental zero adjustment is shown at figure 5.

Diaphragm vacuum gauge accuracy test after zero adjustment. This operation is necessary if vacuum gauge accuracy is low after performing its verification by methods described in this paper. After zero adjustment vacuum gauge must be verified once again using all described methods. For example, based on the obtained experimental data CDG 025D diaphragm vacuum gauge pressure measurement accuracy was low. Therefore, zero adjustment was performed and re-verified. Adjusted vacuum gauge was checked using (3, 4) and calculating inaccuracy. Experimental results are shown in table 7.

No.	Ar, sccm	O2, sccm	CDG 025 D indicated pressure, Torr.	Estimated pressure	Inaccuracy, %
1	50	0	2.86E-03	Input data	0
2	75	0	4.33E-03	4.29E-03	0,93%
3	100	0	5.75E-03	5.72E-03	0,52%
4	150	0	8.52E-03	8.58E-03	0,70%

Table 7. Vacuum gauge estimated pressure and inaccuracy

After preliminary analysis of obtained experimental data, it was possible to conclude that diaphragm vacuum gauge pressure measurement accuracy was high. After the adjustment, the indicated pressure values were precise and uncertainty was low comparing to the diaphragm gauge experimental results before zero adjustment as seen in table 3.

4. Vacuum gauge verification methodology

The overall vacuum gauge verification methodology includes all the above described vacuum gauge verification approaches, which are represented as block scheme in figure 6.

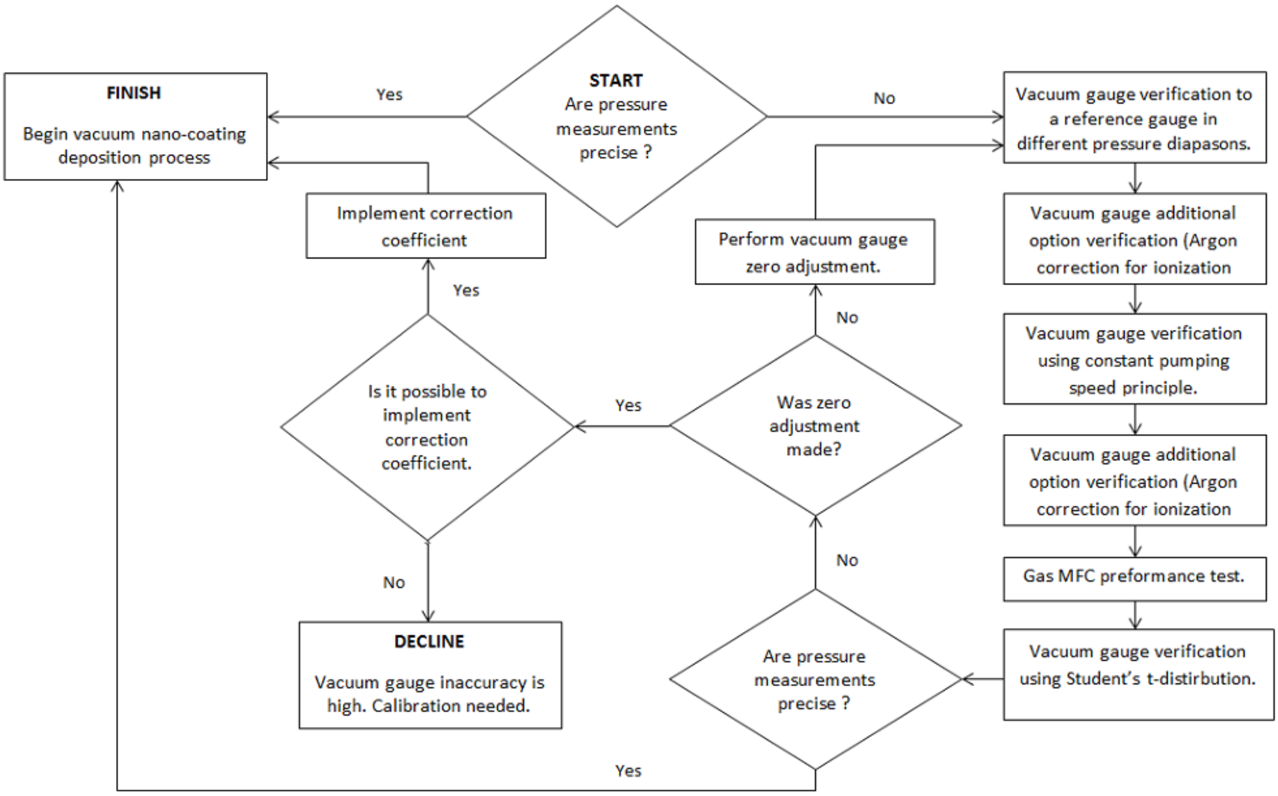

Fig. 6. Vacuum gauge verification methodology

Main goal of verification is to obtain reliable data under different conditions. Vacuum gauge performance will influence a quality of deposited nano-coating during manufacturing process, therefore it is necessary to secure reliable pressure indication. Each approach focuses on critical and possible vacuum gauge inaccuracies, which may take place or emerge. It was concluded that there is no need to perform expensive and long-term vacuum gauge calibration in accredited laboratories, because using the developed method it is possible to obtain full spectre data and act accordingly. If vacuum gauge verification was performed and even after adjustment pressure measurement uncertainty is high, then evaluation of current situation is needed. Evaluation includes technological process parameter and verification experimental data analysis to introduce a correction coefficient. If correction coefficient introduction is not possible, then vacuum gauge should be declined for further use and send to recalibration. Diaphragm vacuum gauge CDG 025D was verified using described methods. After adjustment vacuum gauge inaccuracy did not exceed 2% limit. In this situation correction coefficient implementation was not necessary.

5. Conclusion

The paper analyses vacuum gauge pressure deviation problems and its influence on coating quality. It was concluded that even small pressure changes will influence a visual look and parameters of the coating. To prevent following disadvantages a precise and reliable vacuum gauge shall be used for vacuum nano-coating deposition process. Vacuum gauge verification should be performed before deciding, whether a vacuum gauge recalibration is needed in special accredited laboratories. In a result of presented research a vacuum gauge verification methodology was designed. The method allows to obtain complete data on vacuum gauge pressure measurement accuracy and uncertainty thereof. Developed vacuum gauge verification methodology allows to perform all the verification operations under manufacturing conditions in short period of time in order to rapidly resume the coating deposition process. The methodology can be used as a guide in any company, which focuses on deposition of various vacuum nano-coatings. Further research should focus on correction coefficient implementation in vacuum gauges, its testing and observation.

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