Standards fabrication to providing metrology traceability in micromass and nanoforce measurements results

Thaiane Vargas Pereira[1], **Anderson Beatrici**[1]

[1] Mass Laboratory (Lamas), Scientific and Technological Metrology Division (Dimci) - Inmetro

E-mail: tvpereira@colaborador.inmetro.gov.br

Abstract: Some of the more sensitive weighing equipment available nowadays has its repeatability close to tenth of microgram. OIML characterize mass standards bigger them 1 mg, so in this range doesn't exist direct traceability to the kg prototype. The ASTM has a characterization of mass standard 50, 100, 200 e 500 micrograms. This work have a purpose of providing traceability to mass measurement in microgram scale (nanonewton scale in force) with the confection and calibration of a standard weights collection. At this time were studied two materials, Tungsten and MetGlass 2705M (MetGlass), and produced 12 mass standards.

Keywords: Micromass. Traceability. Nanoforce. Nanometrology.

Resumo: Alguns equipamentos de pesagem mais sensíveis disponíveis atualmente possuem uma repetibilidade na faixa de décimo de micrograma. A OIML só caracteriza padrões de massa maiores que 1 mg, assim nessa faixa de pesagem não há rastreabilidade direta ao protótipo do kg. Na ASTM existe a caracterização dos padrões de massa de 50, 100, 200 e 500 microgramas. Esse trabalho tem o propósito de prover rastreabilidade em medição de massa na escala de micrograma (escala nanonewton em força) com a confecção e calibração de uma coleção de pesos-padrão. Até o momento foram estudados dois materiais, o Tungstênio e o MetGlass 2705M (MetGlass), e confeccionados 12 padrões de massa.

Palavras-chave: Micromassa. Rastreabilidade. Nanoforça. Nanometrologia.

1. INTRODUCTION

The American Society for Testing and Materials (ASTM) recognized the advances in the area of weighing equipments that currently can achieve less than one microgram in the repeatability. The ASTM has specifications for precision mass standards (ASTM E617-13 revised in 2013 for including definition of weight specifications submilligrams with maximum permissible error.

"Weight manufacturers must be able to provide evidence that all new weights comply with specifications in this standard (material, density, magnetism, mass values, uncertainties)"[1].

In Brazil, specialized metrology regulations for the manufacturing and use of standard weights Nº. 233/1994 [2] don't exist in 50 µg up to 500 µg range (500 nN up to 5 µN in the force case). The OIML R-111, specifies the format, dimensions, nominal values, nature of the material used in mass standards construction and assigns accuracy classes, has international classification and definitions related to mass standards and in this recommendation mass standards have their nominal value starting in 1 mg.

The US government is the one that most invests in micro and nanotechnology. In 2000, the Institute of Technology of the National Initiative of Nanotechnology was created, with initial investments of US $ 495 million and continues being the country that invests more in this area.

The science current state increasingly requires traceability in the microgram range, being relevant for balances sensitivity determination, in nanotechnology, in biotechnology, in fine chemistry, and others. Researches in the Chemical, Biological, Pharmaceutical and Materials areas often involve weighing-based measurement processes whose results must be traceable to mass units in order to ensure, on the micro scale, the quality of their measurement results. Usually gravimetric measurements have the small uncertainties involved in this process.

This paper shows the process of choosing the appropriate material for standards production and stability monitoring. We used aluminum to make the mass standards, as it is recommended in OIML to nominal values in a milligram range, and we are testing the manufacturing with others materials, for instance, MetGlass and tungsten which showed good stability during the monitoring tests.

2. MATERIAL CHARACTERISTICS

The manufacture of microgram standards requires proper material and precise production. It is very hard to handling these mass standards, than the size and shape are critical characteristics of these standards. We used Aluminum (Al), MetGlass2705M and Tungsten, but we will describe just the MetGlass (MG) and Tungsten (W) characteristics because Aluminum is already recommended for use in standard weights.

2.1. Tungsten characteristics

The Tungsten was confirmed by determining its specific mass. The value of its mass was obtained from weighing, and its dimensions determined by an optical measurement system [3], resulting in the confirmation of tungsten [4], as shown in table 1. The tungsten wire diameter is 0.0767 mm obtained in an optical device (figure 1).

Table 1. Tungsten characterization.

Tungsten	Tabulated value	Measured value
Density	19.2 g/cm³	19.1 g/cm³
Elastic Modulus	400 GPa	

2.2. MetGlass characteristics

Amorphous metal alloys, MetGlass, are a new class of material, are composed of atoms, molecules or ions that don't have a long range ordering. The MetGlass properties are direct related with its homogeneous structure, thus allowing a different behavior of the amorphous alloys in relation to the crystalline alloys. These alloys are produced by the fast cooling of their liquid phase (glasses), therefore metals without crystalline structure are called "metallic glasses" [5, 6, 7]. MetGlass2705M have a chemical composition of 69% Co, 12% B, 12% Si, 5% Ni and 2% Mo [8]. And it has the following properties [9] as shown in table 2:

Table 2. MetGlass 2705M characterization.

Density (g/cm³)	Vicker's hardness (50g load)	Tensil strentgth (GPa)	Elastic Modulus (GPa)
7.80	900	1-2	100-110

2.3. Stability

An important step in this research is mass monitoring and stability. During 3 months of monitoring we obtain the following results shown in the table 3.

Table 3. Standards mass in microgram.

Standard weigh identification	Mean Value (µg)	Standard deviation (µg)
1 (MG)	47.00	0.87
2 (Al)	47.50	0.58
3 (MG)	50.00	1.04
4 (MG)	52.50	2.18
5 (MG)	96.50	1.60
6 (MG)	97.50	0.76
7 (MG)	99.00	0.76
8 (MG)	102.50	1.29
9 (MG)	201.00	1.92
10 (MG)	201.00	3.21
11 (W)	497.50	1.80
12 (MG)	499.50	2.32

The weighs collection has the following distribution, table 4.

Table 4. Nominal value standard mass number.

Amount	Mass value (µg)
4	50
4	100
2	200
2	500

3. TRACEABILITY

The traceability to the SI unit mass, the kilogram, is widely used and stablished in all countries. In Brazil, the mass measurements results are traceable of 1 mg up to greater nominal values. A lot of the modern weighing equipment has resolution much less than 1 milligram, in fact 0.1 microgram. In this case the traceability can be reached by interpolation (or extrapolation) [10] to determine the corrections and the respective measurement uncertainties [11].

Thus to provide a better reliability and reach lower uncertainties (Type A) is necessary to provide traceable standards weights in the microgram range.

3.1 Equipments and materials

The following materials were used in the standards mass production:

i) Tungsten wire.

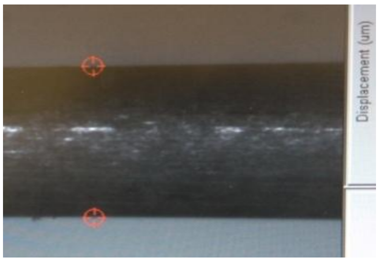

Figure 1. Wire tungsten in system optical.

ii) MetGlass strip.

Figure 2. MetGlass strip.

iii) Aluminum strip.

Figure 3. Aluminum strip.

The equipments used are:

i) Sartorius C5 mass comparator (ultramicro). Used in the samples mass determination and stability monitoring.

ii) Standard weight collection PP016, 1 mg up to 20 mg [12]. To perform the mass comparator linearity and sensitivity.

iii) Digital caliper with 0.01 mm resolution. Just to samples length measurements.

iv) Optical dimensional system. 0.001 mm of resolution [3]. To determine the volume of tungsten wire to calculate the density.

4. MEASUREMENTS AND MONITORING

4.1. Balance calibration

To improve the measurements results reliability we perform a mass comparator linearization in nominal value below 20 mg. The measurements points (table 5) were verified using the E_1 accuracy class standard weight collection PP016.

Table 5. Measurement results of balance.

	1 mg	2 mg	5 mg	10 mg	20 mg
1	0.9885	2.0003	4.9987	10.0087	19.9890
2	0.9887	2.0008	4.9988	10.0109	19.9909
3	0.9887	2.0006	4.9985	10.0086	19.9897
4	0.9889	2.0014	4.9984	10.0092	19.9901
5	0.9895	2.0005	4.9981	10.0098	19.9896
m	**0.9888**	**2.0007**	**4.9985**	**10.0094**	**19.9898**

Using the Calibration Certificate [12] we obtain the corrected values, table 6.

Table 6. Mass corrected measurement results.

Nominal value (mg)	Mean value (mg)	Corrected values (mg)
1	0.9888	0.9904
2	2.0007	2.0006
5	4.9985	4.9995
10	10.0094	10.0083
20	19.9898	19.9932

In figure 4 are shows the plotted data of table 6 corrected values and we calculate the linear *b* and angular *a* coefficients (sensibility).

a = 0.9998 mg/mg

b = -0.0015 mg

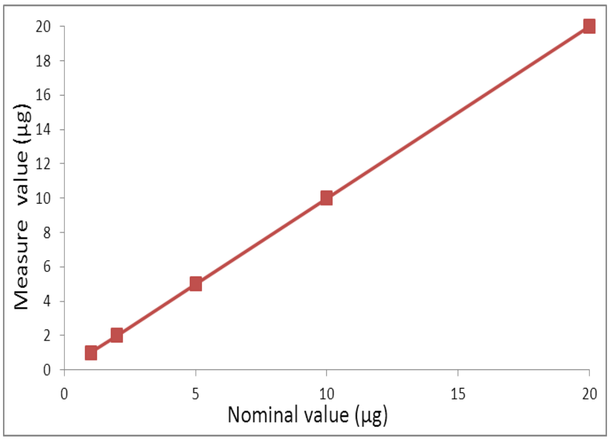

Figure 4. Nominal value *vs.* measurement result.

5. CONCLUSIONS

Nowadays, scientific researches have greater requirements for traceability at lower mass values, lower than OIML R-111 recommendation provides. This lack accurse in all fields that requires measurements results in micro or nano scale in mass or force measurements.

This research has the purpose of determining the methodology of mass standards production, including the material choice and its metrological characterization. The initial results point out that in this range of mass all of materials (Tungsten, MetGlass and Aluminum) are stable at least in these three months of monitoring. Aluminum is already recommended by OILM as a material for mass standards, especially for its low density which allows better handling of the standards. Tungsten has very high density, creating a difficulty for its handling despite its advantages of homogeneity and chemical stability. The MetGlass alloy has an intermediate density, but it is very versatile and can be manufactured in very thin strips which allows for better standards manipulation, the disadvantage are its magnetic properties, in many cases the cutting process already cause magnetization, what is a factor against its use. The next steps are the calibration of the mass standards and the continuous evaluation of the long term stability.

6. REFERENCES

[1] Standard Specifications for Laboratory Weights and Precision Mass Standards, Nashville, 2013.

[2] INMETRO, Regulamentação Metrológica Instrumentos de Medição e Medidas Materializadas. Março, 2010.

[3] Optical Grade Calibration Certificate: DIMCI 0926/2016. Lamed/Inmetro.

[4] Tungsten – Mechanical Properties and Material Applications, AZoM, December 20, 2012.

[5] HOLT, M. L. Less-common metals and alloys. Metal Finish, p. 48-55, 1956.

[6] NAGEL, S, R. Advanced chemical physics. New York: Wiley, 1982. p. 227-275

[7] SEARSON, P. C.; NAGASKAR, P. V.; LATANISION, R. M. In: WHITE, R. E.; BOCKRIS, J. O. M.; CONWAY, R. B. (Ed.). Modern aspects of electrochemistry.New York: Plenum Press, 1990. p.121-161.

[8] MATERIALS - Conference proceedings. Techology & Engineering. **Volume II**, SPE/ANTEC, 1996.p.1769.

[9] MAGNETIC ALLOY 2705M, Metglass - Technical Bulletin, Ref: 2705M08022014.

[10] EURAMET, Guidelines on the Calibration of Non-Automatic Weighing Instruments, version 3, Edition 2011.

[11] BIPM, IEC, IFCC, ILAC, ISSO, IUPAC, IUPAP and OIML 2008 Guide to Expression of Uncertainty in Measurement (GUM: 1995 with Minor Corrections) JCGM 100:2008 (Sèvres: Bureau International dês Poids et Mesures).

[12] Standards Weighs Calibration Certificate (PP016): DIMCI 1247/2015. Lamas/Inmetro.

ACKNOWLEDGEMENTS

This work was funded by the National Program to Support the Development of Metrology, Quality and Technology (Pronametro) and Inmetro Mass Laboratory (Lamas).