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GB/T 38913—2020

核级锆及锆合金管材氢化物取向因子检测方法

Test methods for hydride orientation fraction of nuclear grade zirconium and zirconium alloy tubes

（*English Translation*）

（征求意见稿）

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前　言

Forward

SAC/TC *243* is in charge of this English translation. In case of any doubt about the contents of English translation, the Chinese original shall be considered authoritative.

本标准按照 GB/T 1.1-2009 给出的规则起草。

This standard is drafted in accordance with rules given in the GB/T 1.1-2009

本标准由中国有色金属工业协会提出。

This standard is proposed by China Nonferrous Metals Industry Association.

本标准由全国有色金属标准化技术委员会（SAC/TC 243）归口。

This standard is administrated by National Technical Committee for Standardization of Nonferrous Metals

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1.范围

本标准规定了核级锆及锆合金管材氢化物取向因子检测的方法。

本标准适用于通过高压釜渗氢、气体渗氢后，对核级锆及锆合金管材进行氢化物取向因子检测。

1.Scope

This standard specifies test methods for hydride orientation fraction of nuclear grade zirconium and zirconium alloy tubes.

This standard is applicable to testing the hydride orientation fraction of nuclear grade zirconium and zirconium alloy tubes after hydrogenation permeation in autoclave or hydrogenation permeation in other device.

2.规范性引用文件

下列文件对于本文件的应用是必不可少的。凡是注日期的引用文件，仅注日期的版本适用于本文件。凡是不注日期的引用文件，其最新版本（包括所有的修改单）适用于本文件。

GB/T 13298金属显微组织检验方法

2 Normative reference documents

The following documents are indispensable for the application of this document. For dated references only the edition cited applied. For undated references, the latest edition of referenced documents (including any amendments) applied.

GB/T 13298 Inspection methods of microstructure for metals

3 术语和定义

下列术语和定义适用于本文件。

3 Terms and definitions

For the purpose of this document, the following terms and definitions apply.

3.1

氢化物

氢与金属性较强的金属形成的化合物。

3.1 hydride

A compound formed of hydrogen and a metal with strong metalicitity.

3.2

径向氢化物

在锆及锆合金管材某一确定的检查截面上，氢化物与管材径向夹角（θ）不大于产品标准、技术条件或需方要求的规定，且其实际长度大于等于15 μm的氢化物。

3.2 radial hydride

At a certain testing section on nuclear grade zirconium and zirconium alloy tubes, the radia angle （θ）between hydride and the tube shall be no more than the requirements of product standard, technical specification and requirements of the purchaser, and the actual length of which is greater than or equal to 15 μm.

3.3

氢化物数

在锆及锆合金管材某一确定的检查截面上，实际长度大于等于15 μm的氢化物数量计数。

3.3 quantity of hydride

N

At a certain testing section on nuclear grade zirconium and zirconium alloy tubes, the quantity of hydrides with actual lengths greater than or equal to 15 μm is counted.

3.4

径向氢化物数

在锆及锆合金管材某一确定的检查截面上，径向氢化物（3.2）数量计数。

3.4 quantity of radial hydride

N_θ

At a certain testing section on nuclear grade zirconium and zirconium alloy tubes, the quantity of radial hydride (3.2) is counted.

3.5

氢化物取向因子

F_n^θ

在锆及锆合金管材某一确定的检查截面上，径向氢化物数（3.4）与氢化物数（3.3）之比。

3.5 hydride orientation fraction

F_n^θ

refers to at a certain testing section on nuclear grade zirconium and zirconium alloy tubes, the ratio of the quantity of radial hydride (3.4) to the quantity of hydride (3.3).

4 原理

锆及锆合金管材与氢气或氢氧化锂反应，以氢化物的形式析出。在锆及锆合金管材某一确定的检查截面典型区域处，统计径向氢化物数和氢化物数，计算其比值。

4 Theory

The hydride is precipitated when nuclear grade zirconium and zirconium alloy tubes react with hydrogen and lithium hydroxide. Count the generated quantity of quantity of radial hydride and quantity of hydride at a certain testing section with typical position on nuclear grade zirconium and zirconium alloy tubes, and calculate the ratio between them.

5 试剂

除非另有说明，在分析中仅使用确认为分析纯的试剂和实验室用二级水。

5 Reagent

Unless otherwise stated, only reagents confirmed as analytically pure and class 2 water used in the laboratory.

5.1 氢氧化锂Lithium hydroxide, ρ=1.46g/cm3。

5.2 硝酸Nitric acid, ρ=1.42g/ mL。

5.3 氢氟酸Hydrofluoric acid, ρ=1.12g/mL。

5.4 过氧化氢Hydrogen peroxide, ρ=1.13g/mL。

5.5 乳酸Lactic acid, ρ=1.20g/mL。

5.6 无水乙醇Anhydrous ethanol, ρ=0.789g/mL。

6 仪器设备

6.1 高压釜：应具有温度、压力测量及时间控制功能。

6.2 气体渗氢设备：具有温度、流量控制功能。

6.3 金相显微镜：应配备图像采集系统和金相分析软件，金相分析软件应具有长度、角度测量功能。

6 Apparatus

6.1 Autoclave: temperature, pressure measure and time adjustable.
6.2 Gas hydrogen permeation equipment: temperature and flow control adjustable.
6.3 Metallographic microscope:be equipped with image acquisition system and metallographic analysis software with the function of length and angle measurement.

7 样品

7.1 取样部位与数量按产品标准或技术条件规定。推荐高压釜渗氢样品长度尺寸为 30 mm±3 mm，推荐气体渗氢样品长度尺寸为 13 mm±3 mm。

7.2 样品在制备过程应避开因剪切、加热影响的区域。不能使用有改变样品应力状态的方法进行样品制备。

7 Sample

7.1 Sampling location and quantity shall be accordance with product standard and technical specification. It is recommended that the sample tested in autoclave with hydrogenation permeation should be 30±3 mm, and hydrogenation permeation should be 13±3 mm.
7.2 Areas affected by shearing and heatting shall be avoided during sample preparation. The method which can change stress status of the sample can not be used during sample preparation.

8 试验步骤

8.1 渗氢过程

渗氢过程应按照附录A的方法进行。渗氢量上下限可参考附录B所对应的形貌图。

8 Testing procedure

8.1 hydrogen permeation process

Hydrogen permeation process shall be conform to Annex A. Refer to morphology pictures in Annex B for upper and lower limits of hydrogen permeation.

8.2 金相制备

8.2.1 渗氢后的样品禁止对管壁进行处理，对任一截面按 GB/T 13298 规定的方法制备金相样品，确保足够的去除量，金相样品制备过程中不允许加热和加压。

8.2.2 样品制备完成后，采用浸蚀方式进行样品截面的浸蚀，推荐选用下列浸蚀剂之一：

a) 乳酸:硝酸:氢氟酸=45:45:8(体积比)
b) 过氧化氢:硝酸:氢氟酸=45:45:1(体积比)

也可采用其他能够清晰显示出氢化物的浸蚀剂，浸蚀时间为 10s～40s。

8.2 Metallographic preparation

8.2.1 No treatment of tube walls of sample is allowed after hydrogen permeation process. Prepare metallographic samples on any section with methods stipulated in GB/T 13298 and sufficient removal amount should be ensured. No heating and increasing pressure are allowed during metallographic samples preparation.

8.2.2 The corrosion method is adopted on section of samples. One of the following corrodent is recommended:

a. Lactic acid: Nitric acid: Hydrofluoric acid=45:45:8(volume ratio)

b. Hydrogen peroxide: Nitric acid: Hydrofluoric acid=45:45:1(volume ratio)

Using other corrodents which can also identify hydride clearly is acceptable. Corrosion time: 10s～40s.

8.3 氢化物取向因子检测

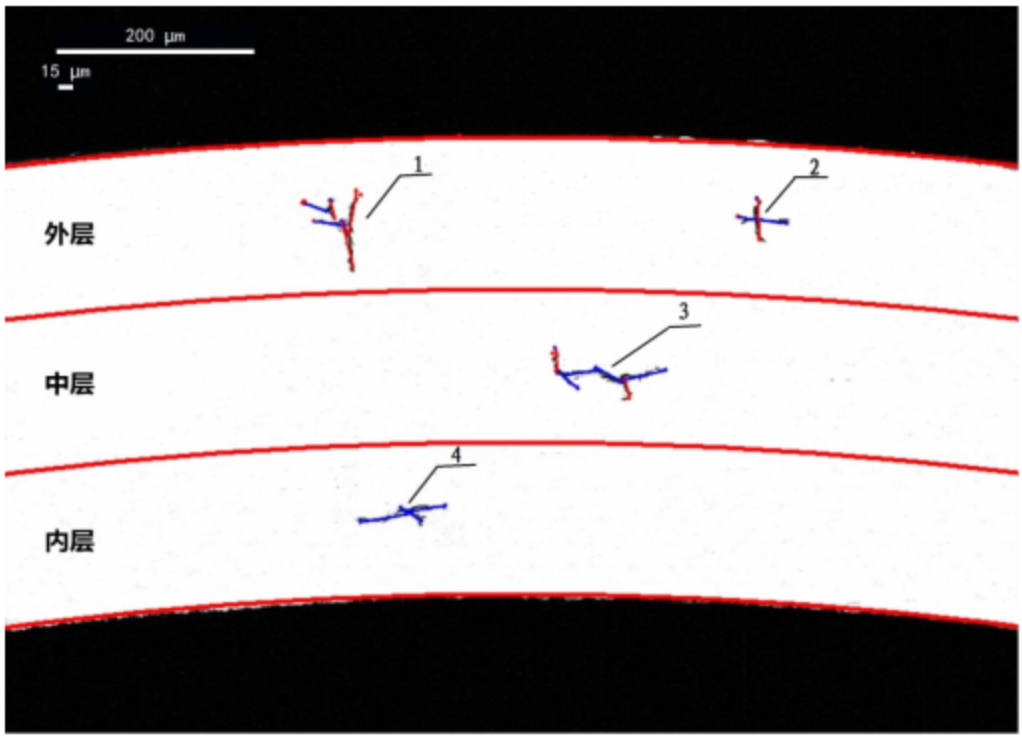
检测截面区域应为整个管材样品截面中典型氢化物形貌的区域。推荐采用放大倍率为 100 倍。

使用金相分析软件，选定待测氢化物形貌照片中管材外径边界，确定圆心位置（如图 1 所示），再沿管材壁厚方向等分为外层、中层、内层三个区域。

8.3 Test of hydride orientation fraction

8.3.1 The testing cross section shall be in the typical morphology areas in whole section of tube samples. The 100 times magnification is recommended.

8.3.2 Use metallographic analysis software to select the external radial boundary in morphology pictures from samples to be tested and define center position (as shown in Figure 1). Divided it along tube wall thickness direction equally into three sections named outer layer, middle layer and inner layer separately.



说明：

1、2——外层氢化物；

3——中层氢化物；

4——内层氢化物。

Illustration:

1、2——hydride in outer layer;

3——hydride in middle layer;

4——hydride in inner layer。

氢化物取向因子检测示意图

Figure 1 Schematic diagram of hydride orientation fraction

分别对外层、中层、内层三个区域的氢化物数和径向氢化物数进行记录。

若所测氢化物相交，在交点处分割，若氢化物长度较长，无需分割。如图 1 所示，外层氢化物 1 可分割为 5 片氢化物，外层氢化物 2 可分割为 2 片氢化物，中层氢化物 3 可分割为 6 片氢化物，内层氢化物 4 可分割为 2 片氢化物。

Record the quantity of hydride and quantity of radial hydride in three sections of outer layer, middle layer and inner layer.

If hydride to be tested intersects, divide it at the intersection. No division is required when length of the hydride is longer. hydride1 in outer layer can be divided into 5 pieces of hydride, hydride 2 in outer layer can be divided into 2 pieces of hydride, hydride 3 in middle layer can be divided into 6 pieces of hydride, hydride 4 in inner layer can be divided into 2 pieces of hydride, as shown in Figure 1.

9. 试验数据处理

采用测量法按照公式（1）分别计算外层、中层、内层氢化物取向因子，计算结果保留小数点后两位。

$$F_n^{\theta} = \frac{N_{\theta}}{N} \cdots\cdots (1)$$

式中：

F_n^{θ}—氢化物取向因子；

N_{θ}—径向氢化物数；

N—氢化物数。

9. Test data processing

The measurement method is used to calculate hydride orientation fraction in outer layer, middle layer and inner layer according to the formula. The result of the calculation is reserved for two decimal places.

$$F_n^{\theta} = \frac{N_{\theta}}{N} \cdots\cdots (1)$$

where:

F_n^{θ}—hydride orientation fraction;

N_{θ}—quantity of radial hydride

N—quantity of hydride

10. 试验报告

试验报告应至少包括下列内容：

a) 本标准编号；
b) 样品信息及标识；
c) 渗氢方法及试验条件；
d) 试验结果；
e) 试验人员及试验日期。

10 Test report

Minimum contents in the report should include:

a) Number of this standard;
b) Sample information and identification;
c) Hydrogen permeation method and test condition;
d) Test result;

e) Test personnel and date.

Appendix A
（规范性附录）
核级锆及锆合金管材渗氢方法

(annex normative)
Hydrogenation permeation method of nuclear grade zirconium and zirconium alloy tubes

A.1 渗氢前样品处理 Sample preparation before hydrogenation permeation

A.1.1高压釜渗氢前样品制备Sample preparation before hydrogenation permeation in autoclave

A.1.1.1 使用无水乙醇浸泡擦洗样品，擦洗干净后用水冲洗 3 次，单次时间为 2 ～3 min。

A.1.1.2 使用体积配比为：氢氟酸:硝酸:水=1：4.5：4.5 的酸洗液（或其他酸洗液）进行酸洗。

A.1.1.3 酸洗后的样品迅速转移到流动水中冲洗，冲洗时间为 1 ～2 min，然后冷风吹干。

A.1.1.1 Soak and scrub the sample with anhydrous ethanol. Flushing the sample with water three times with duration 2～3 min each time after cleaning.

A.1.1.2 Pickling with solution(volume ratio of Hydrochloride acid: Nitric acid: Water=1: 4.5: 4.5)or other pickling solution.

A.1.1.3 The sample is quickly transferred to running water to rinse after pickling. Rinse for 1 ～2 min and then dry it with cold air.

A.1.2 气体渗氢前样品制备Sample preparation before hydrogenation permeation.

A.1.2.1 使用无水乙醇浸泡擦洗样品，擦洗干净后用水冲洗，时间为 2 ～3 min。

A.1.2.2 使用体积配比为氢氟酸 :硝酸 :水=35：90：484 的酸洗液进行酸洗，酸洗后将样品迅速转移到流动水冲洗，反复酸洗、冲洗 4 次，每次酸洗时间为 20 s，冲洗时间为 1 ～2 min。

A.1.2.3 冲洗后的样品在 50 ℃中性清洗溶液中超声清洗，时间为 10 min，接着用水漂洗，时间为 1 ～2 min，最后在无水乙醇浸泡后，冷风吹干。

A.1.2.1 Soak and scrub the sample with anhydrous ethanol. Flush with water after scrubbing it clean. Rinsing for 2 ～3 min.

A.1.2.2 Pickling with solution (volume ratio of Hydrofluoric acid: Nitric acid: water=35: 90: 484). The sample is quickly transferred to running water to rinse after pickling. Repeat the pickling and rinsing 4 times with 20 seconds each time. Rinse for 1～2 min.

A.1.2.3 Put the sample after rinsing for ultrasonic cleaning in neutral cleaning solution at 50 ℃ for 10 min. Soak the sample with anhydrous ethanol and then dry with cold air.

A.2 渗氢过程 Hydrogenation permeation process

A.2.1 高压釜渗氢：用氢氧化锂配制浓度为1 mol/L或其他浓度的氢氧化锂水溶液，在温度为360 ℃±6 ℃，压力为18.6±1.4 MPa条件下进行渗氢。

A.2.2 气体渗氢：通有氢气、氩气混合气（建议氢气体积分数为：1.8 %～2.2 %，其余为氩气，也可使用其他安全的氢气体积分数)的气体渗氢设备中,采用预充氢氩混合气10 min～20 min,再加热至399 ℃±14 ℃的方式进行渗氢。若需方同意，样品可以在温度不超过414 ℃的惰性气体中保温5 h±1 h进行热处理，如果使用真空热处理，为防止脱氢，压力不应小于1.33 mPa。降温过程中建议冷却速率应低于14℃/min。

A.2.3　渗氢时间：以测量区域内，100倍条件下氢化物数量等于或大于100片所需的渗氢时间，作为高压釜、气体渗氢的时间。

A.2.1 Hydrogenation permeation in autoclave: Prepare solution with a concentration of 1 mol/L with lithium hydroxide or other concentration of lithium hydroxide solution. Permeating in temperature 360±6 ℃ and ambient pressure 18.6±1.4 MPa.

A.2.2 Hydrogenation permeation: Filling the mixture of hydrogen and argon into the hydrogenation permeation equipment(The recommended hydrogen volume fraction is 1.8 %～2.2 % and the rest of volume is argon. Other safety hydrogen volume fractions is acceptable.). Hydrogenation permeation by the filling for 10～20 min and then heat to 399 ℃±14 ℃. Sample could be in heat treatment in inert gas for 5±1 h if purchaser agrees. The pressure shall not be smaller than 1.33 MPa if in vacuum heat treatment in order to keep from dehydrating.

A.2.3 Hydrogenation permeation time: Take hydride quantity equal to or greater than 100 pieces required hydrating time with 100 times in the testing areas as hydrogenation permeation in autoclave or hydrogenation permeation time.

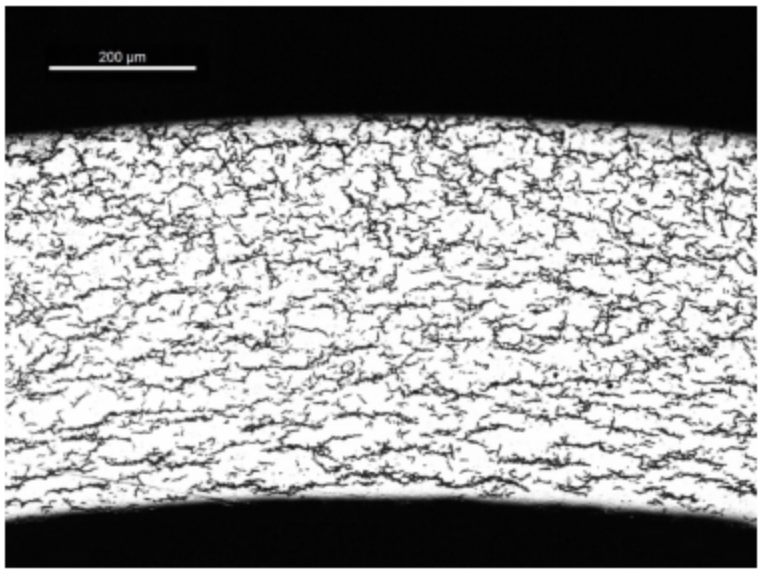
（资料性附录）
核级锆及锆合金管材渗氢量范围形貌图

Appendix B
(annex informative)
Morphology pictures of hydrogenation permeation range of nuclear grade zirconium and zirconium alloy tubes

B.1 渗氢量上限形貌图 Morphology pictures of upper limit of hydrogenation permeation

核级锆及锆合金管材渗氢量上限形貌图如图B.1所示。

Morphology pictures of upper limit of hydrogenation permeation of nuclear grade zirconium and zirconium alloy tubes (As shown in Figure B.1)



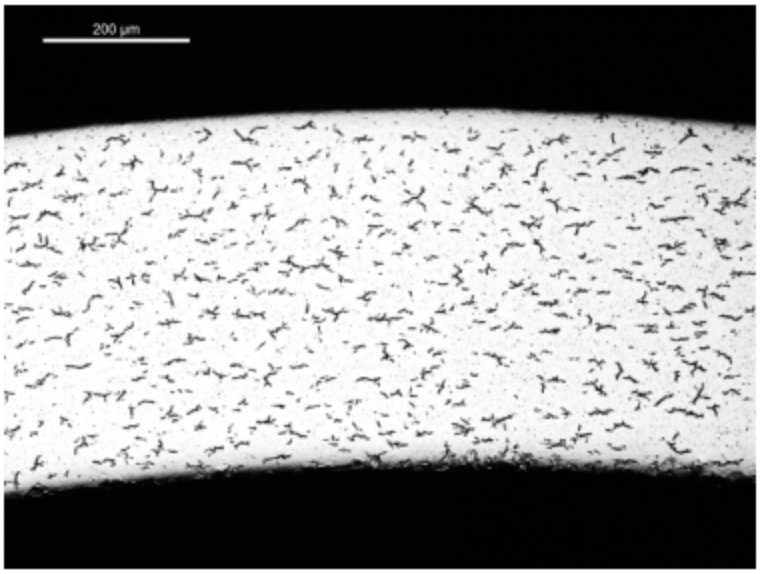
核级锆及锆合金管材渗氢量上限形貌图 100×

Figure B.1 Morphology pictures of upper limit of hydrogenation permeation 100×

B.2 渗氢量下限形貌图Morphology pictures of lower limit of hydrogenation permeation

核级锆及锆合金管材渗氢量下限形貌图如图B.2所示。

Morphology pictures of lower limit of hydrogenation permeation of nuclear grade zirconium and zirconium alloy tubes (As shown in Figure B.2)



核级锆及锆合金管材渗氢量下限形貌图　100×

Figure B.2 Morphology pictures of lower limit of hydrogenation permeation 100×