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NATIONAL STANDARD OF THE
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CCS J 04

GB/T 40307-2021

**Non-Destructive Testing - Test Method for Texture by
Neutron Diffraction**

无损检测 材料织构的中子检测方法

Issued on: May 21, 2021

Implemented on: December 01, 2021

**Issued by: State Administration for Market Regulation;
Standardization Administration of PRC.**

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Non-Destructive Testing - Test Method for Texture by Neutron Diffraction

1 Scope

This Document specifies the method for detecting the texture of materials on the reactor by neutron diffraction technology.

This Document is applicable to the detection of texture of polycrystalline materials.

2 Normative References

The following documents are essential to the application of this Document. For the dated documents, only the versions with the dates indicated are applicable to this Document; for the undated documents, only the latest version (including all the amendments) is applicable to this Document.

GB/T 12604.8 Terminology for Non-Destructive Testing - Neutron Testing

GB/T 26140 Non-Destructive Testing - Standards Test Method for Determining Residual Stresses by Neutron Diffraction

3 Terms and Definitions

For the purposes of this Document, the terms and definitions given in GB/T 12604.8 and GB/T 26140 and the following apply.

3.1 Texture

The crystal grains in the polycrystalline sample are arranged in different directions with different orientation characteristics, which is manifested as the preferred orientation structure of the sample.

NOTE: The neutron detection method can obtain pole figures of multiple crystal planes, obtain accurate orientation directions, and obtain quantitative data of texture intensity.

3.2 Neutron diffractometer for texture measurement

Neutron radiation detection device for measuring texture of materials.

3.3 Pole figure

The projection map of the orientation distribution of the selected crystal plane of each crystal grain in the material in the sample coordinate system.

NOTE: The sample coordinate system refers to the rectangular orthogonal coordinate system established according to the sample shape, which is the basic expression form of texture intensity.

3.4 Inverse pole figure

The projection map of the orientation distribution of the selected crystal plane of each crystal grain in the material in the crystal coordinate system.

NOTE: The crystal coordinate system refers to the rectangular coordinate system established in accordance with the crystal orientation, which is the basic expression form of texture intensity.

3.5 Pole density

Polycrystalline materials are projected to the polar equator, and the weighted density distribution of the crystal grain volume represented by each point on the sphere.

3.6 Orientation distribution function

An optimized azimuth expression form of the three-dimensional spatial distribution of crystal elements.

4 Principle

After being irradiated by neutron rays, a polycrystalline sample with regularly arranged atoms shall produce coherent scattering at a specific angle. This process is called Bragg diffraction, and the process is given by Formula (1):

$$\lambda = 2d \sin \theta \quad \dots\dots\dots (1)$$

Where:

d – interplanar spacing, in nm;

θ – 1/2 diffraction angle, in °;

λ – neutron wavelength, in nm.

The texture shall cause the Bragg diffraction peaks of the sample in the three-dimensional space and cause the changes in intensity. The texture intensity of the material is obtained by collecting the diffraction peaks of different spatial angles in the three-dimensional space and analysing the diffraction peaks.

The monochromator is a device that uses crystals to select and reflect rays. The monochromator is mainly composed of single crystal, focusing device, adjustment table and other components. The energy range of the neutron beam selected by the monochromator should be 5meV~25meV; and the corresponding neutron wavelength range is 0.1nm~0.3nm. The monochromator should have vertical focusing or horizontal and vertical double focusing capabilities to increase the neutron fluence rate at the sample. The monochromators commonly used in neutron diffractometer for texture measurement include: silicon monocrystalline monochromator, pyrolytic graphite monocrystalline monochromator, monocrystalline germanium monochromator, and so on.

5.2.3 Euler ring

The Euler ring is used to realize the assembly and positioning of the sample, and to provide the rotation of the sample in the three-dimensional space coordinate. The Euler ring shall realize the sample rotation azimuth φ and the Euler ring rotation azimuth χ according to the set pace and ensure the angular accuracy. Among them, the φ angle should be in the range of $0^{\circ}\sim 360^{\circ}$, the χ angle should be in the range of $0^{\circ}\sim 360^{\circ}$, and the angular accuracy should be better than 0.1° .

5.2.4 Detector

The detector is a device for capturing and recording neutron signals. The detector can detect neutron energy in the range of 5meV~25meV. Commonly used detectors on neutron diffractometer for texture measurement include ^3He counter tube detectors, ^3He gas multifilament position sensitive detectors, and so on.

6 Preparation for Measurement

6.1 Neutron wavelength selection and calibration

6.1.1 Confirm the crystal structure information of the sample.

6.1.2 Select the crystal plane to be tested and neutron wavelength of the sample.

6.1.3 According to the selected neutron wavelength, set the monochromator take-off angle and focus parameters to a neutron beam obtained at that wavelength of the sample.

6.1.4 After the neutron wavelength is set, the standard calibration sample is used to measure the diffraction spectrum of multiple crystal planes of the sample; and the actual neutron wavelength is obtained through the peak position fitting analysis and calculation of the diffraction spectrum. The calibration of the neutron wavelength and the error value is given by Formula (2). According to the recommendation of the International Neutron Scattering Agency, the neutron wavelength resolution $\Delta\lambda/\lambda_{\text{test}}$

should be better than 1×10^{-2} . The commonly used calibration samples include silicon powder, iron powder, aluminium oxide powder, and so on.

$$\Delta\lambda = \lambda_{\text{test}} - \lambda_{\text{theory}} \dots\dots\dots (2)$$

Where:

λ - neutron wavelength, in nm;

λ_{test} - neutron wavelength in test, in nm;

λ_{theory} - neutron wavelength in theory, in nm.

6.2 Calibration of mechanical positioning accuracy

6.2.1 Set the detector rotation angle, Euler ring inclination angle and sample rotation angle, respectively; and build an angular accuracy detection device at this angular position.

6.2.2 Drive each rotation angle to control the motor; so that make each rotation angle that deviates from the set position to the set angle; and the repeatability accuracy is obtained by measurement.

6.2.3 Arrange the theodolite at the position corresponding to the centre of circle for each rotation angle; measure the absolute angle value from the beginning of the rotation angle to the set value; and obtain the absolute accuracy by the measurement.

6.3 Calibration of spectrometer resolution

6.3.1 The resolution of the spectrometer usually refers to the resolution of the interplanar spacing.

6.3.2 The standard calibration sample is installed in the centre of the sample bench; and select the measurement crystal plane and its corresponding diffraction angle.

6.3.3 Set the focus parameters and diffraction angle of the monochromator of the spectrometer; and carry out the collection of the diffraction spectrum of the selected crystal plane.

6.3.4 Obtain the measured d value of the standard sample according to the diffraction peak position.

6.3.5 Use the measured interplanar spacing d value and the sample theoretical interplanar spacing d_0 to calculate the spectrometer resolution ($\Delta d/d$), which is given by Formula (3):

2 - regular diffraction peaks on the background;

I --- diffraction peak intensity;

BG - diffraction background, the signal caused by stray neutrons;

2θ - diffraction angle, in $^{\circ}$.

Figure 6 -- Background Deduction Method of Diffraction Peaks

8.2 Correction of temperature influence

8.2.1 The experimental temperature change causes the change of the sample lattice spacing d , which indirectly leads to the change of the diffraction peak intensity.

8.2.2 When the temperature change during the texture detection experiment is within 5°C , the fluctuation of the diffraction peak intensity caused by the temperature change (uniformly changed to the intensity change) is less than one ten-thousandth, then its influence is negligible.

8.2.3 When the temperature change during the experiment exceeds 5°C , the change in interplanar spacing shall be calculated according to the thermal expansion coefficient of the sample; and the fluctuations of diffraction peak intensity caused by this change shall be included in the texture intensity error analysis. The temperature of the sample shall be monitored during measurement.

8.3 Data analysis

8.3.1 Polar density distribution function

The polar density distribution function $p_{hkl}(\chi, \varphi)$ is given by Formula (6):

$$p_{hkl}(\chi, \varphi) = \frac{I_{hkl}(\chi, \varphi)}{2 \sum_{\chi=0(\Delta\chi)}^{\pi/2} \sum_{\varphi=0(\Delta\varphi)}^{2\pi} I_{hkl}(\chi, \varphi) \sin\chi \Delta\chi \Delta\varphi} \dots\dots\dots (6)$$

Where:

χ - rotation azimuth of Euler ring, in $^{\circ}$;

φ - rotation azimuth of sample, in $^{\circ}$;

$I_{hkl}(\chi, \varphi)$ - integrated intensity of the (hkl) crystal plane diffraction peak of the specimen in the (χ, φ) direction.

8.3.2 Calculation of orientation distribution function and texture strength

Confirm the orientation direction y of the sample. The orientation direction y of the sample is related to the Euler angle χ and φ ; and their relationship satisfies Formulas

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