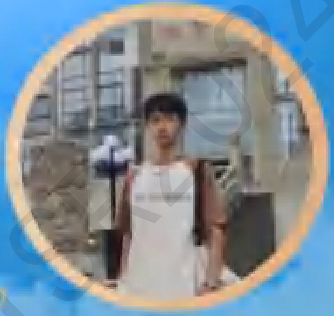
北京科技大学
University of Science and Technology Beijing

X射线断层扫描技术在定型耐火材料检测中的应用

Application of X-ray computed tomography in testing of shaped refractories

The 9th International Symposium on Refractories (ISR 2024)

Reporter： 职 佳 爽　　Time： 2024.10.17



职佳爽
JiaShuang Zhi

北京科技大学
材料科学与工程学院
School of Materials Science and Engineering
无机非金属结构材料研究室
Inorganic Non Metallic Structural Materials Research Laboratory
硕士三年级

导师：李勇　Tutor: Li Yong

Tel：18403797625
Wechat：zjs979133161
Email:zjs979144161@163.com

CONTENTS

01 Display of test results for shaped refractories

02 Introduction to X-CT technology

03 Our laboratory's CT equipment

04 Industrial CT testing operation steps

05 Summary and Outlook

The importance of pore structure in refractories

Pores, as an indispensable component of the gas-phase structure in refractories, a large number of pores will inevitably be introduced during raw materials, molding, and service processes. The internal structure and pore characteristics of refractories can directly affect their performance in high-temperature industries.

Pores

Porosity

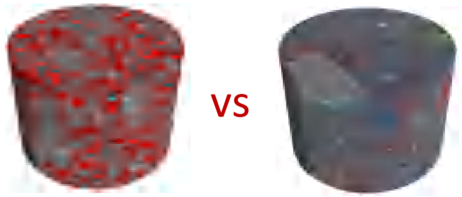
Normally, reducing porosity is beneficial for improving the strength and high-temperature creep performance of refractory materials, but thermal conductivity and thermal shock resistance may decrease.

Light heat-Insulation refractory

Dense refractory

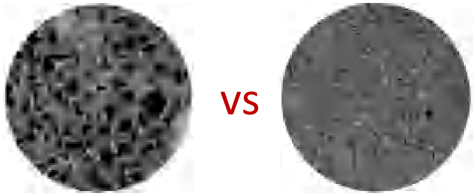
Pore distribution
(Through、closed pores, open pores...)

Penetrating pores facilitate the migration of liquid phase at high temperatures, while a large number of closed pores can lead to loose material structure.



pore size
(average、maximum、distribution...)

Reducing the average pore size, especially the maximum defect size, is the main way to solve the contradiction between porosity and strength[1] .



pore morphology

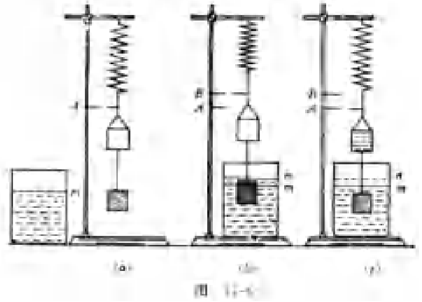
Pores that are closer to spherical shapes can withstand higher pressure, which is beneficial for improving the physical properties of refractories[2].

cracks

Similar to pore analysis

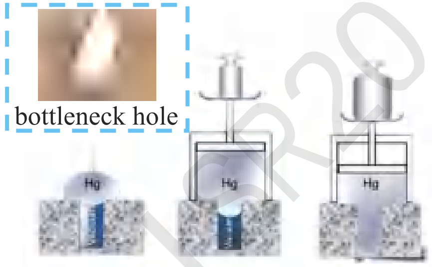
Common pore testing methods for refractories

Common pore testing methods include: **Archimedean drainage method, Mercury intrusion method, SEM...**



Archimedean drainage method

- Cannot test closed pores
- Cannot test samples that are too small or too large in size
- Cannot test easily hydrolyzed materials
- More suitable for measuring materials with relatively high density

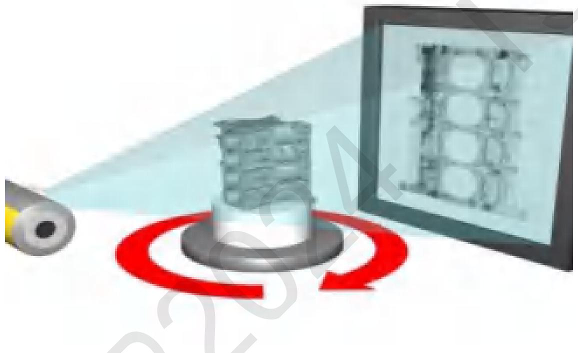


Mercury intrusion method

- Cannot test closed pores
- There are restrictions on size and shape requirements
- Difficult to measure “bottleneck” holes
- More suitable for measuring average pore size and pore size distribution

SEM

- Observation limited to two-dimensional angles only
- Mechanical scratches, structural deformations, and other damages are inevitably introduced during the sample preparation process



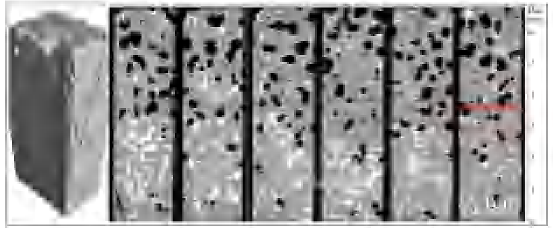
Industrial CT

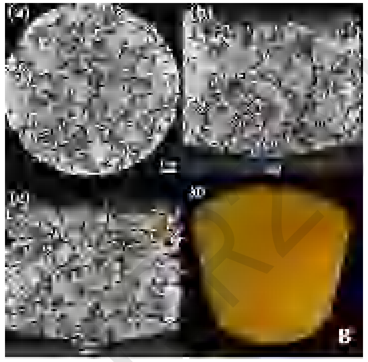
- Non-destructive testing
- 3D visualization
- high-precision (Advanced equipment can detect holes with a minimum aperture of 10 nm)
- Visual display
- Simple sample preparation
- Low testing requirements
- Suitable for almost all kinds of refractories

……

CT testing for refractories

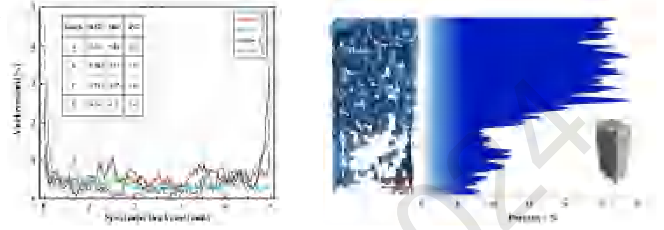
All internal information





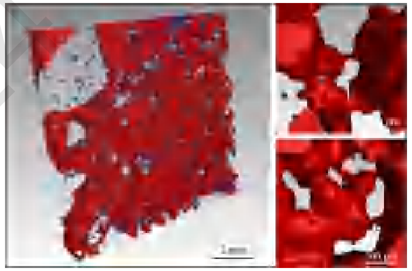
Test closed pores

Layer by layer variation of porosity

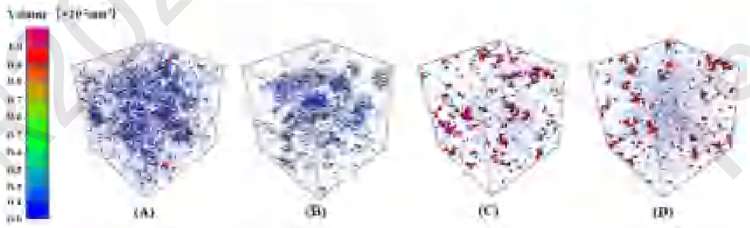


Extract different pores and structure

Pore morphology

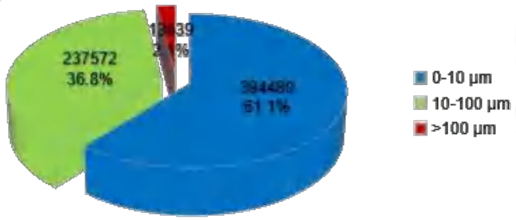


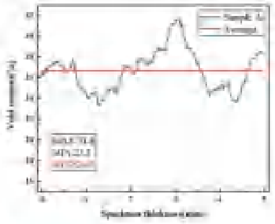
Distribution of pores



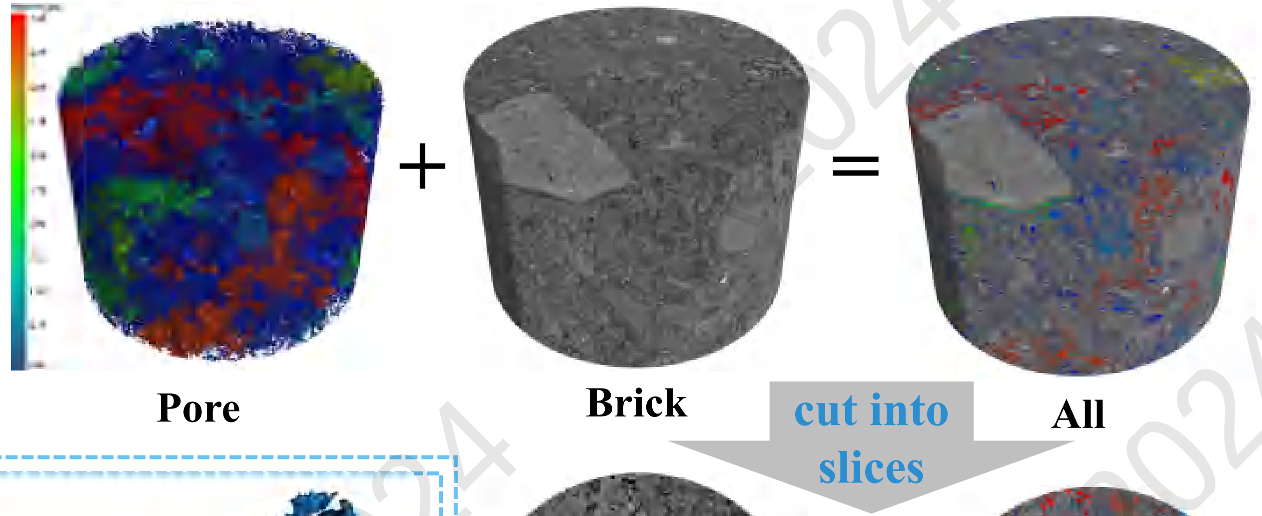
(Pore size, sphericity, volume, tightness...)

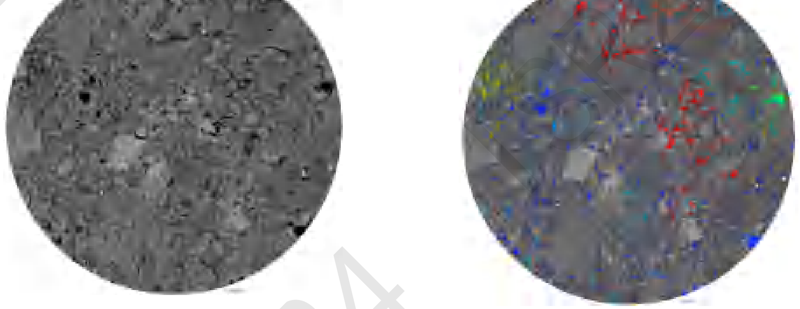
Pore information statistics





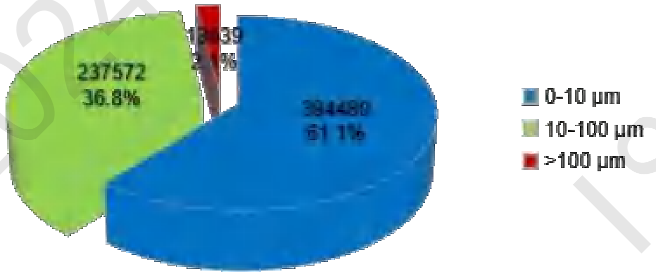
Display of test results for shaped refractories





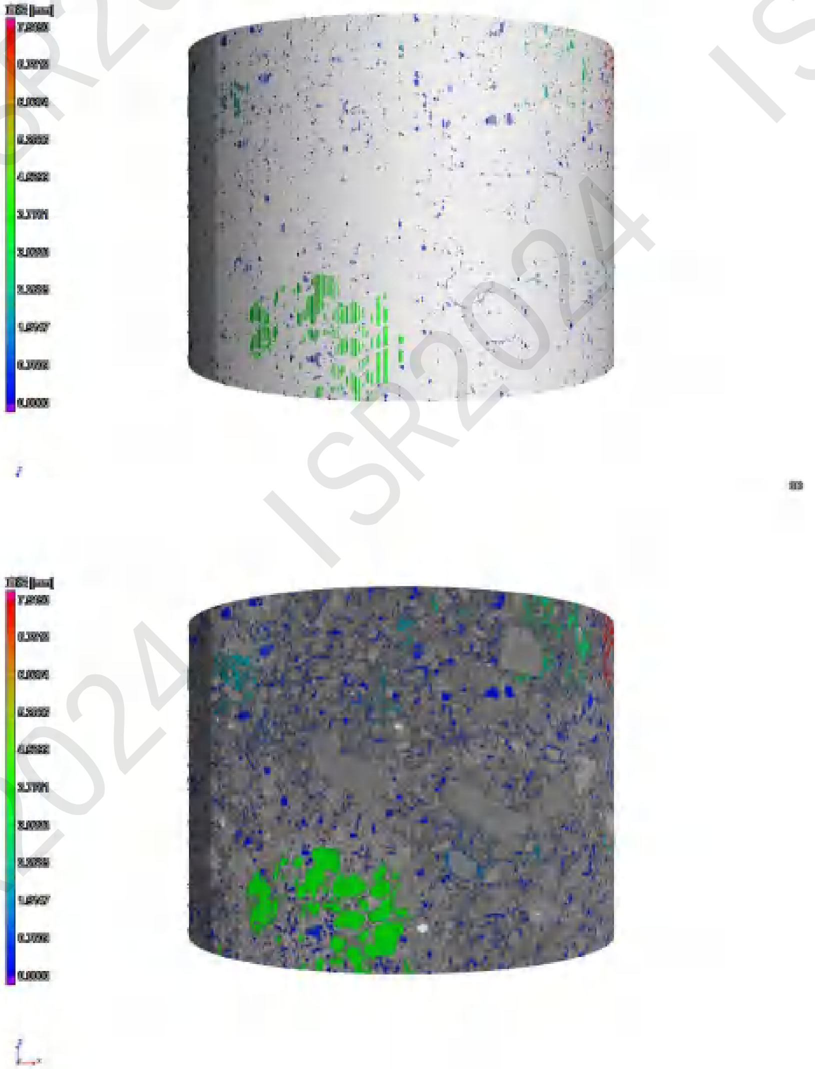
Slice image

3D image of pore structure



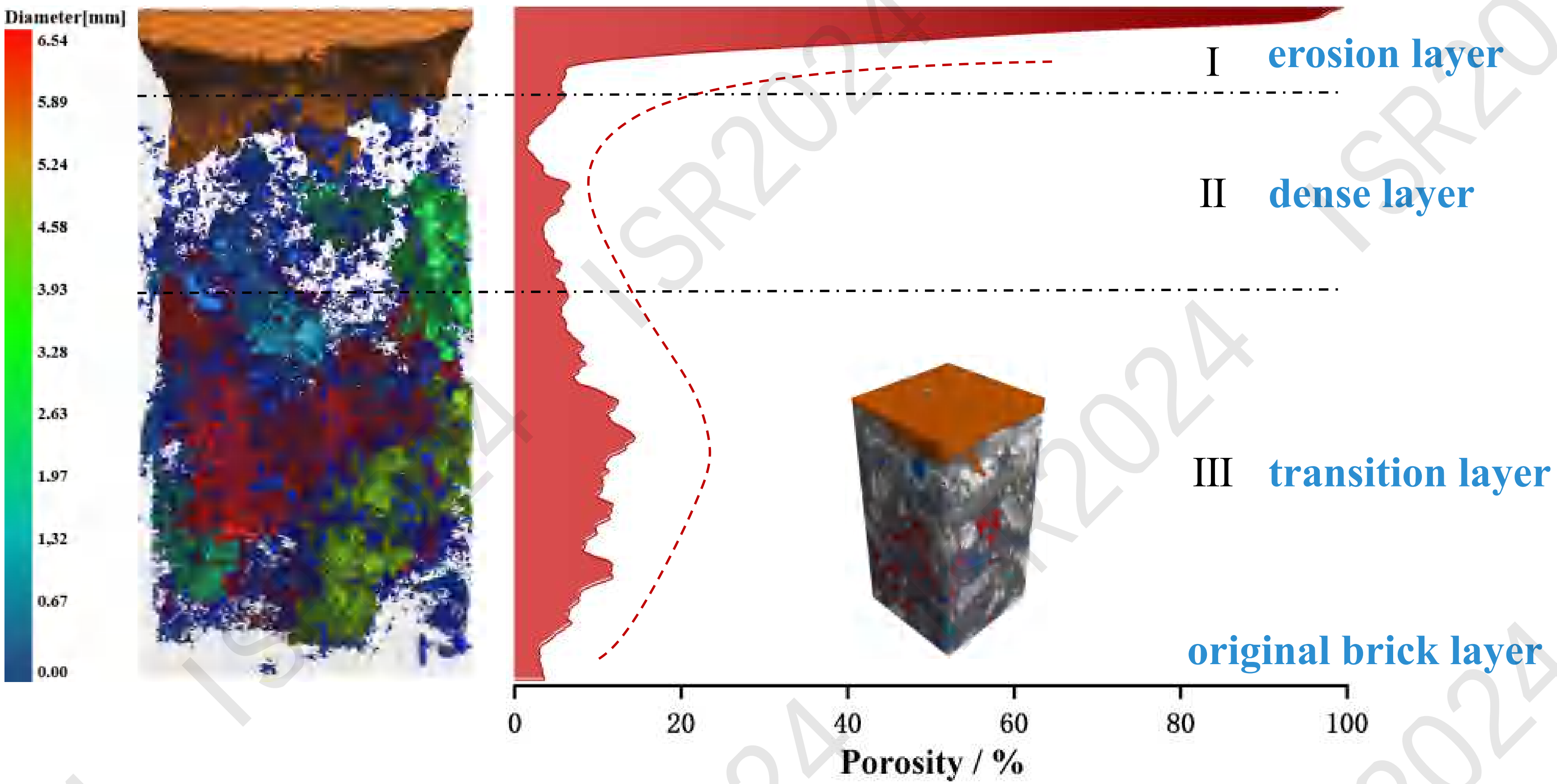
Statistical image of pore size extraction

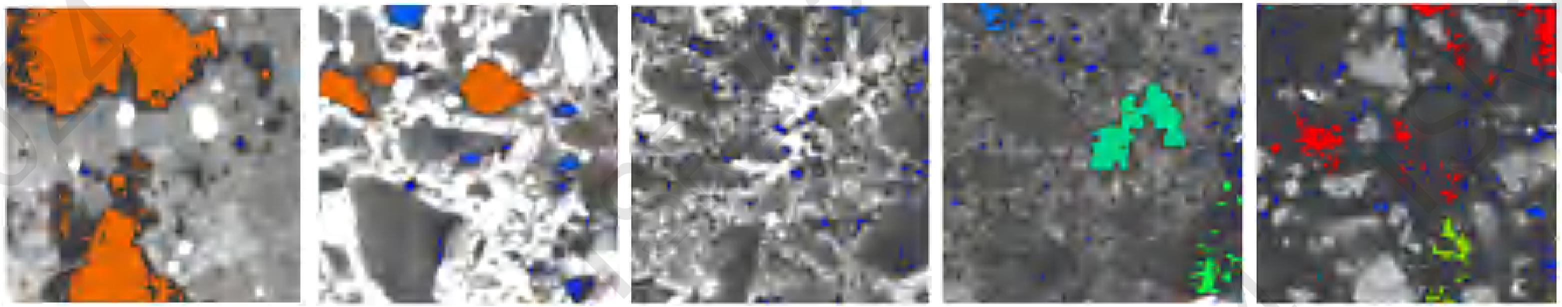
X-ray tomography results of dense high-alumina brick



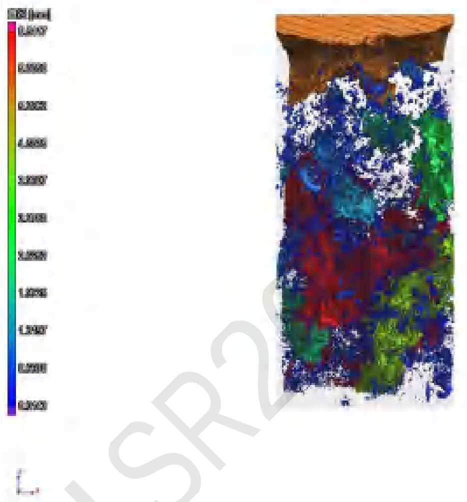
3D animation

Failure analysis of melt erosion brick





I I→II II II→III III



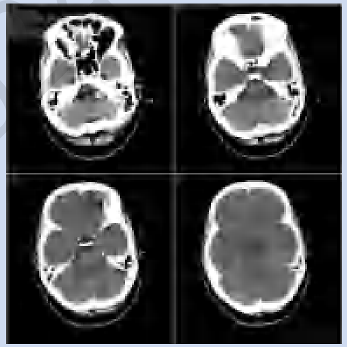
3D animation

Introduction

X-ray computed tomography (**X-CT**), also known as **X-ray absorption imaging** or **industrial CT**, as an advanced non-destructive testing technology, is widely used in medicine, industry, materials science, earth science and other fields.

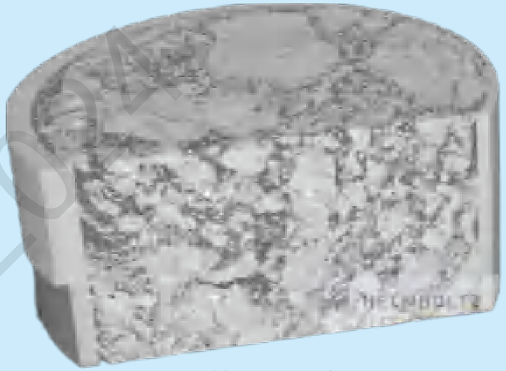
Medical CT equipment

It was originally used as a medical device for scanning the human body in the medical field, with a scanning resolution of about 0.5 mm.

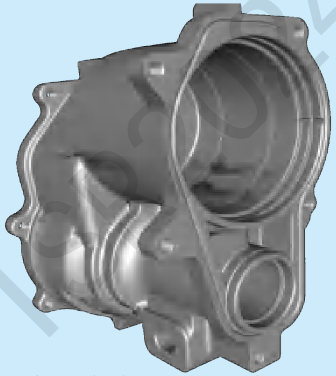


CT image of the lung of a COVID-19 patient、 teeth and head

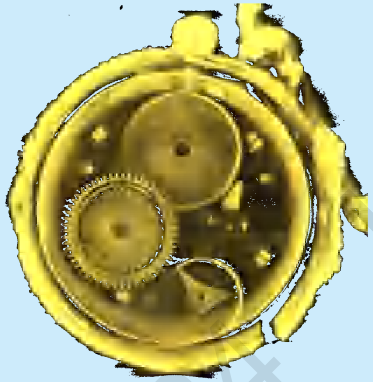
With the advancement of technology, CT technology has gradually been applied in industrial and scientific research fields, and micro-focus X-ray tomography technology (referred to as micro-CT) has been developed, with a scanning resolution of 0.3-0.005 mm or even lower.



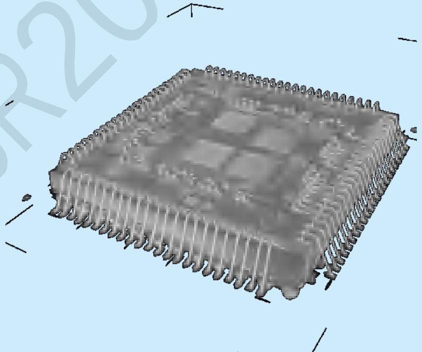
Soil Sample



Aluminium Casting



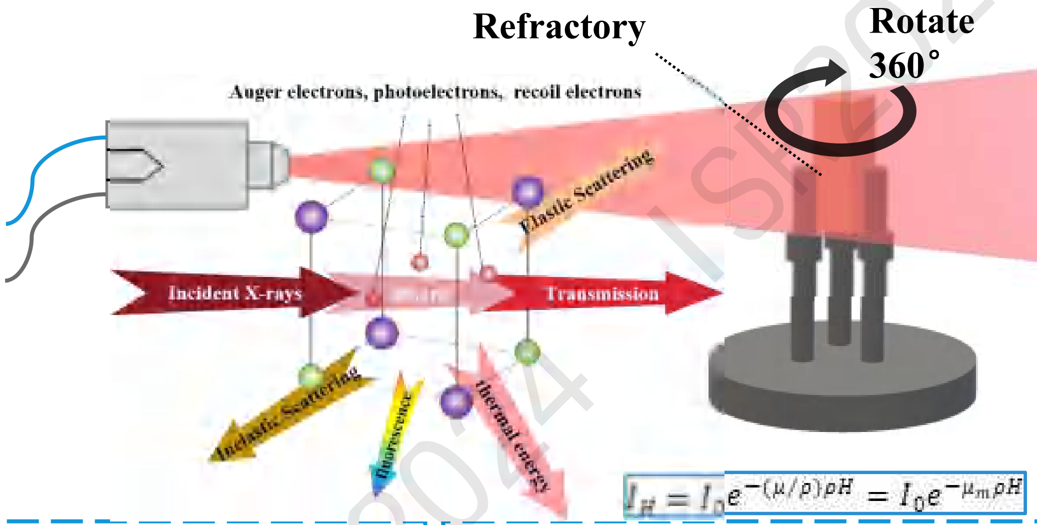
17th Century Watch

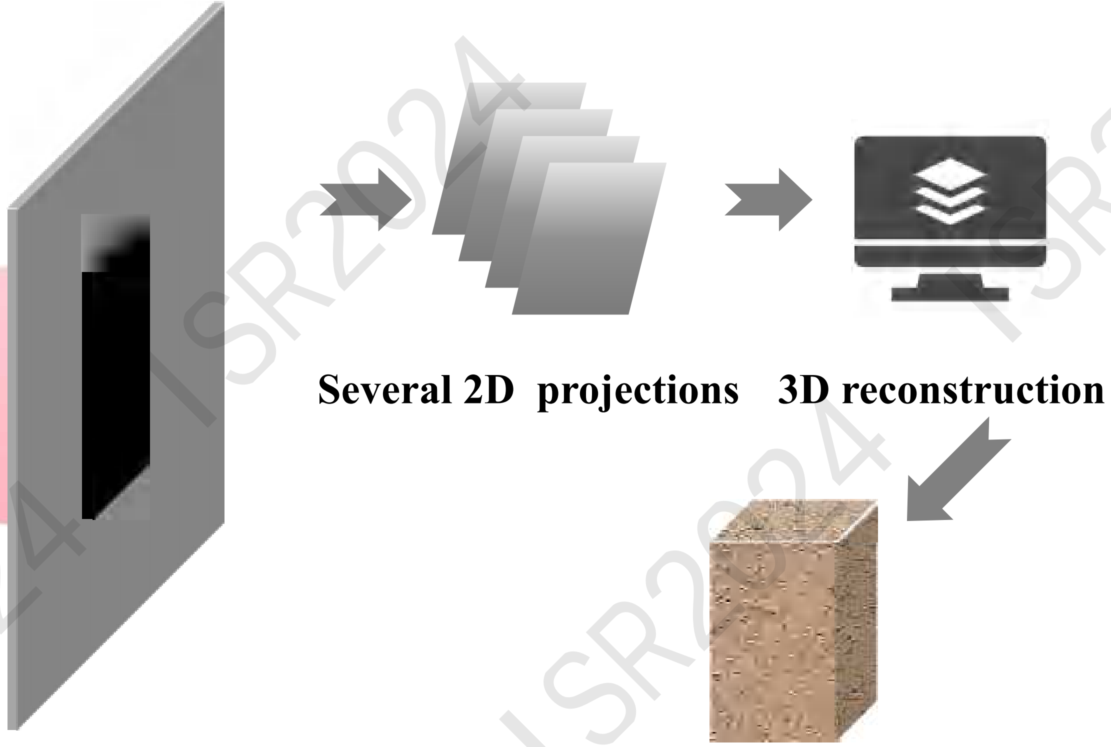


Electronic chip

Composite fiber

Working principle of CT





x-ray source

the X-ray source generates high-energy X-ray beams, these beams have strong penetrating power and can penetrate most materials, including steel, ore, composite materials and so on.

Sample stand / fixture

The X-ray beam interacts with the material when passing through it. Substances with different densities and compositions have varying absorption capabilities for X-rays.

The intensity attenuation of the X-ray in the transmission direction when it passes through a material is proportional to the distance in the matter through which it passes, and is proportional to the density of the material.

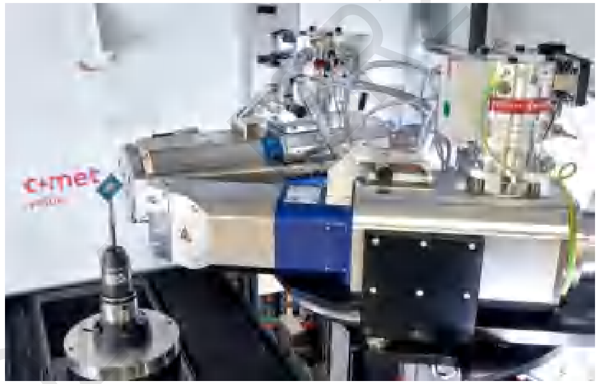
detector

After the X-ray penetrates the sample, the attenuated photon signal will be projected to the detector, which receives different photon signals and converts them into electrical signals. These electrical signals contain information about the internal structure of the sample.

3D image

The material rotates continuously at tiny angles with the sample stage, and the detector can continuously receive photon signals after X-ray transmission. After completing one rotation, all information inside the material can be obtained.

Industrial CT equipment in our laboratory



The CT equipment used in the laboratory in this paper is the **YXLONE FF35CT** industrial CT from Germany, with a focus mode of Micro focus. It is equipped with:

- **225 kV micro-focus refracted X-ray probe** (maximum power 320 W, spatial resolution ≤ 4 μm, minimum defect detection capability 3 μm);
- **190 kV micro focus transmitted X-ray probe** (maximum power 64 W, spatial resolution ≤ 0.6 μm, minimum defect detection capability 300 nm);
- high-resolution imaging panel Y.Panel 2530;
- 225 KV radiation protection lead room;
- Supporting analysis software: **VG Studio MAX 3.2** (VG), **Dragonfly;**
- high-resolution, high-power target material;
- CT reconstruction workstation;
- Large display screen operation console;
- various types of fixtures, etc.

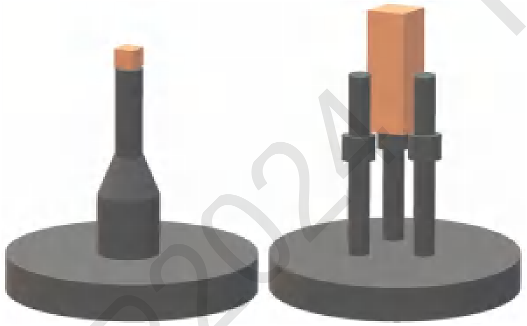
This industrial CT equipment can achieve **Quick Circular Scan**, **Circular Scan**, **HeliExtend Scan**, **HeliExtend Dual Scan** and other functions, which can basically meet the scanning and analysis of materials in various fields.

Industrial CT testing operation steps

① **Sample preparation:** The maximum thickness of the scanned sample should be within the range of 1 mm - 200 mm. If there are higher scanning resolution is required, the smaller the sample should be prepared. For refractory samples, generally **a 10 mm cylindrical sample** can meet the accuracy requirements for measurements.

② **System preparation:** Check the electrical system, mechanical system, radiation protection and other parts of the equipment, and deal with faults and hidden dangers in time. Subsequently, prepare for scanning such as micro focus alignment and device reset, and confirm that the self-test information interface of the main control software is normal.

samples

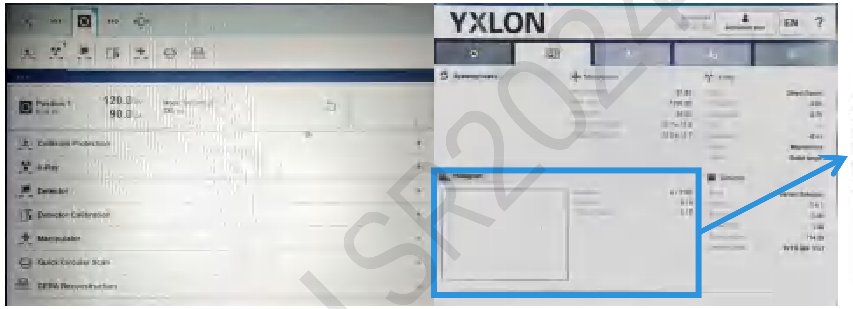


fixtures

③ **Place the sample:** Install the appropriate fixture on the sample table, fix the sample on the fixture, and manually rotate 360° after fixing the sample to ensure that the sample will not shake and fall off during the scanning process.

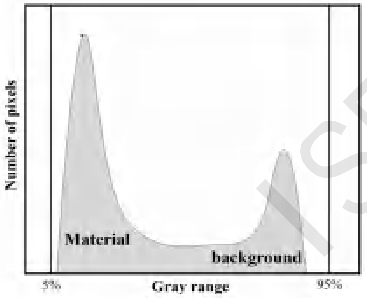
Industrial CT testing operation steps

Collision prevention



Operation panel

Data display panel



Grayscale histogram

④ **Parameter setting:** After fixing the sample, close the lead door and open the workstation equipped with YXLON software on the computer to set the scanning parameters. There are different scanning parameters are available for different materials.

View the grayscale histogram to ensure that the grayscale value curves of the sample and background vary within the upper and lower limits (3000-55000). After all parameter settings are completed, the X-ray source can be opened for scanning.

Quick Circular Scan → **10~20 minutes**

HeliExtend Scan → **1~4 hours**

Set collision prevention

↓

Radiation source parameters

tube voltage, tube current, filter

↓

Detector parameters

projection amplitude, exposure time, integration time, detector type, frame merging times

↓

Mechanical geometry parameters

focal length, object distance, magnification

↓

Scanning mode

Quick Circular Scan, Circular Scan, HeliExtend Scan

↓

Plate correction

↓

Reconstruction parameters

beam hardening parameters, median filter

Industrial CT testing operation steps

⑤ **Data Processing:** The images obtained from 3D reconstruction can be displayed in four views and analyzed using software such as VG, Dragonfly, Avizo, etc. **Taking VG**, which is widely used in the industrial field, as an example, a set of VG software can meet all the needs for measuring.By following these steps, we can obtain beautiful images and a large amount of data.

Image preprocessing → Image segmentation → Threshold division → Rendering → Other operations

Application of X-CT in Shaped Refractory Materials

Internal structure

Structural observation, dimensional measurement, Phase distribution, material shape, etc

Pore analysis

Pore distribution, pore size, pore volume, sphericity of pores, etc

Crack analysis

Crack distribution, crack length, crack direction, fracture mechanism, etc

Failure analysis

Analysis of internal damage morphology, erosion mechanism, tensile mechanism, etc

In situ characterization

In situ mechanical testing, etc

Summary and Outlook

Summary:

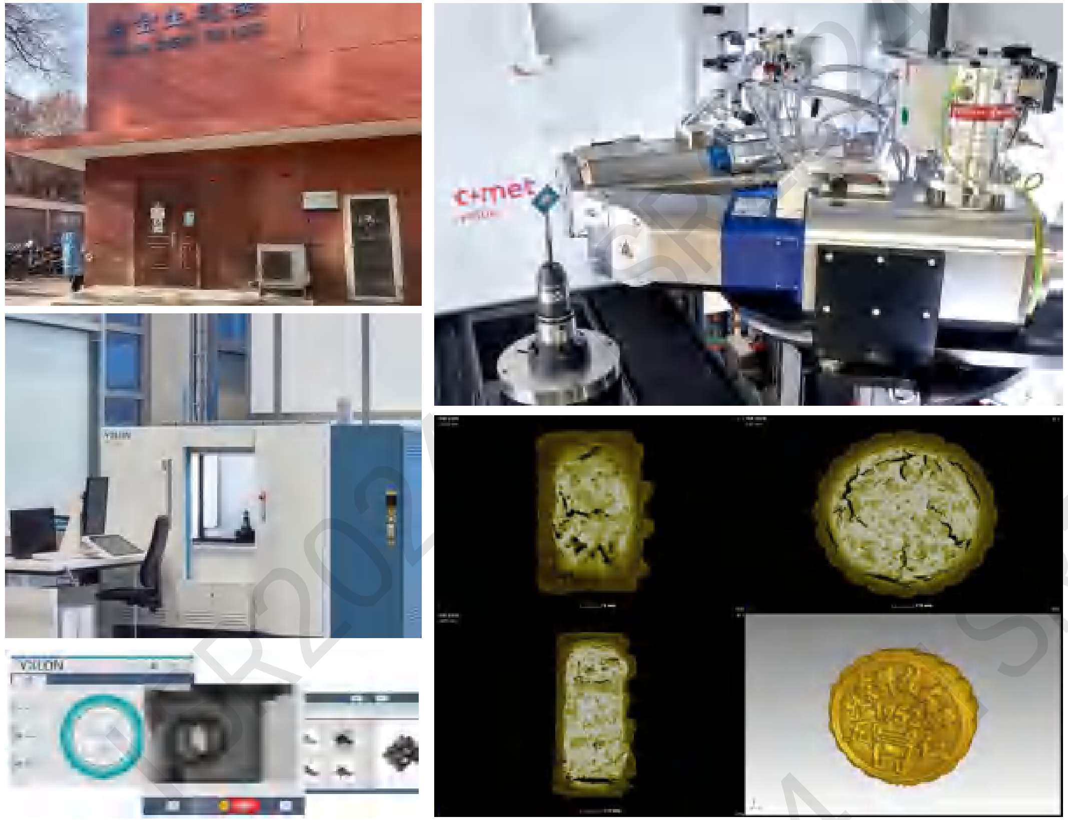
The characterization methods for the internal structure and pore characteristics of refractories are very limited, and it is necessary to develop more advanced detection methods. Industrial CT, as a non-destructive and three-dimensional display detection method, has a wide range of application prospects in the field of refractories. In addition, refractory material testing usually uses multiple characterization methods to understand its different characteristics and properties. Combining industrial CT with other characterization methods can display material information more clearly and accurately.

Outlook:

However, there are currently several issues with industrial CT in refractory material testing:

Subjective	Contingency	Standard
The phase composition of refractories is very complex, and the phases in the images obtained by CT scanning are mainly distinguished based on grayscale thresholds, which makes threshold division highly subjective and leads to significant errors in quantitative analysis of refractories.	Due to the need to produce smaller brick samples in CT testing, errors caused by different sampling positions are inevitable. To address this issue, researchers can take samples from different locations on the bricks and select the most representative samples for testing.	There are relatively few standards released for CT technology in refractory testing. It is necessary to promote the release of relevant standards, strengthen the use of CT scanning in the field of refractories, and unify the testing standards.

Our laboratory



If you are interested in this technology and equipment, contact me：

实验地点：北科大冶金生态楼S104室
负责人：高原
电话：010-62333014
15652779982（微信同号）

Thank

Look forward to
cooperating with you

北 京 科 技 大 学

University of science and technology Beijing

Reporter：职佳爽

Time:2024.10.17