Application News

Microfocus X-Ray CT System inspeXio SMX-225CT FPD HR Plus Phase-Contrast X-Ray CT System Xctal™ 5000

Observation of Rubber Fatigue Testing Specimens **Using Two Types of X-Ray CT Systems**

Kayo Migita and Wataru Sato

User Benefits

- ◆ The volume and shape of individual micro-defects in rubber can be observed in detail using a microfocus X-ray CT system.
- The approximate position and size of microcracks can be determined efficiently using a phase-contrast X-ray CT system.
- The progression status of defects and cracks in rubber can be compared at respective cycle counts of fatigue testing.

■ Introduction

Rubber products serve an important role in a wide range of fields, such as automotive tires, industrial products, and medical devices. However, functional losses can occur due to fatigue and aging after long periods of use. Therefore, evaluating the durability of rubber is an important issue.

In order to evaluate the progression of defects during usage, rubber must be inspected before it fails. It can be inspected by cutting it apart, but non-destructive inspections are preferable due to the disadvantages of destructive inspections, such as the inability to restore specimens after inspections and the limitation of only being able to evaluate cut surfaces.

Internal defects can be detected and three-dimensionally observed using an X-ray CT system. One X-ray imaging method typically used is absorption imaging, which generates images based on the quantity of X-rays absorbed. Another method is phase imaging, which is a new unconventional method that generates images based on variations in the phase of X-rays. This article describes using two types of instruments based on those different imaging methods to observe rubber during fatigue testing (Table 1).

Table 1 Features and Key Points of Instruments Used

Instrument	Instrument Features for this Example	Factors Observed for this Example	
inspeXio SMX- 225CT FPD HR Plus	Can vividly observe profiles of microdefects with higher magnification using absorption imaging	Volume, shape, and count ratio of defects	
Xctal 5000	Can efficiently inspect for microcracks in large exposure fields using phase imaging	Position and approximate size of cracks	

■ Target of Analysis

For this example, the four rubber fatigue testing samples shown in Fig. 1(a) were scanned. An overview of the samples and test parameter settings are indicated in Table 2. If high-cycle samples 1 and 2 are bent over, the external cracks are even visible with the naked eye (Fig. 1(b)). (Samples were provided by the Chemicals Evaluation and Research Institute, Japan.)





Fig. 1 Fatigue Testing Specimens (a) From the Left are Samples 1, 2, 3, and 4; (b) Cracks in Sample 1

Table 2 Overview of Fatigue Testing Specimens

		1	2	3	4
Material		Vulcanized acrylonitrile rubber containing carbon black			
Fatigue Test				oreak)	
Parameters	Cycle Count	1000	500	250	_

■ inspeXio SMX-225CT FPD HR Plus Imaging of **Rubber Fatigue Test Specimens**

The inspeXio SMX-225CT FPD HR Plus (Fig. 2) is a microfocus X-ray CT system. It can be used to visualize the internal structure of parts with high resolution using absorption imaging, which is the most common imaging technique. Because images are generated directly from the X-ray quantities acquired by the detector, high magnification is required to observe micro areas.

CT scan settings used in this case are indicated in Table 3. The sample must be bent over in order to see the external cracking, as shown in Fig. 1(b), so most of the defects in the sample were assumed to be closed shut. Due to the almost complete lack of air in closed defects, defects are difficult to detect in the closed state (Fig. 3(a)). Therefore, for this example, the jig shown in Fig. 3(b) was used to stretch the sample by 10 mm before imaging. (The jig was provided by the Chemicals Evaluation and Research Institute.)

Although magnified imaging is useful for observing microdefects, if the magnification is adjusted based on the width at the parallel portion of the sample, only about one-third of the sample will fit within the X-ray exposure field. However, by using the serial sectioning function, magnified images were acquired in a series of three sections and then recombined.



Fig. 2 inspeXio SMX-225CT FPD HR Plus

Table 3 Scan Settings		
Setting		
200 kV		
70 μA		
2400		
2		
15.4 mm		
0.015 mm		
60 min (20 min 3 times)		

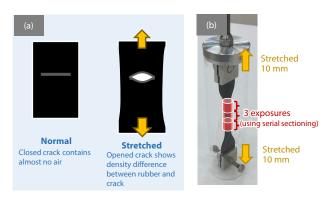


Fig. 3 Precautions for Imaging Cracks in Rubber (a) Illustration of Visualizing Closed Cracks; (b) Jig

■ inspeXio SMX-225CT FPD HR Plus Imaging Results

The defects were analyzed using the optional software VGSTUDIO MAX. Cross-sectional images from near the largest defect in each sample are shown in Fig. 4. However, sample 4 was not tested because no defects were detected. A comparison of cross-sections from samples 1 to 3 shows that the defects were thin and circular regardless of the number of cycles.

Next, the defects were colored yellow in 3D imaging as shown in Fig. 5. With each increase in the cycle count, the cracks, which are indicated with arrows, became more prominent, but smaller cracks are difficult to identify in Fig. 5.

The numerical results from defect analysis are indicated in Table 4, and the results from analyzing the count ratio by volume are indicated in Fig. 6. They show that the number of internal defects, the total volume of internal defects, and the volume of the largest defect all increased as the cycle count increased. Meanwhile, the ratio of relatively small defects (colored blue in Fig. 6) decreased as the cycle count increased. This suggests that the small internal defects gradually grew larger as the cycle count increased.

Thus, data acquired with the inspeXio SMX-225CT FPD HR Plus can be used to analyze each individual microdefect in test specimens and compare the defect count or defect volumes at different cycle counts. In this case, multiple specimens with different cycle counts were prepared, but the progression of defects or cracks can also be compared by CT scanning the same specimen each time the cycle count is increased.

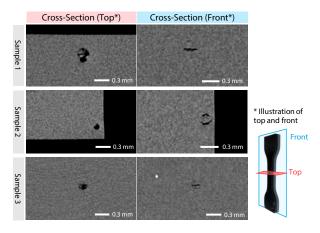


Fig. 4 Cross-Sectional Images (Near Largest Defect)

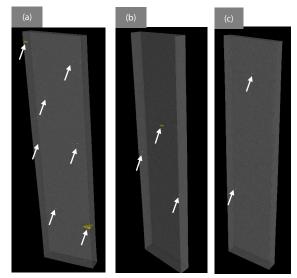
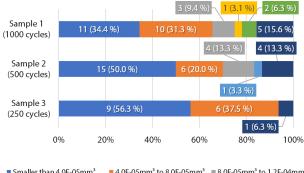


Fig. 5 3D Images (with Colored Defects)
(a) Sample 1 (1000 cycles); (b) Sample 2 (500 cycles); (c) Sample 3 (250 cycles)

Table 4 Defect Analysis Results

Sample	1	2	3	4
Cycle Count	1000	500	250	
Number of External Cracks	10	4		ı
Number of Internal Defects	32	30	16	
Total Volume of Internal Defects	5.6 × 10 ⁻³ mm ³	$3.1 \times 10^{-3} \text{mm}^3$	$1.1 \times 10^{-3} \text{mm}^3$	_
Smallest Defect Volume	$2.6 \times 10^{-5} \text{mm}^3$	2.6 × 10 ⁻⁵ mm ³	$2.6 \times 10^{-5} \text{mm}^3$	-
Largest Defect Volume	1.5 × 10 ⁻³ mm ³	6.1 × 10 ⁻⁴ mm ³	5.0 × 10 ⁻⁴ mm ³	_



- Smaller than 4.0E-05mm³ 4.0E-05mm³ to 8.0E-05mm³ to 1.2E-04mm³ 8.0E-05mm³ to 1.2E-04mm³ 1.2E-04mm³ to 1.6E-04mm³ 1.6E-04mm³ 2.0E-04mm³ to 2.4E-04mm³ 2.0E-04mm³ 2.0E-04
- Larger than 2.4E-04mm³

Fig. 6 Ratios of Defect Volumes

Xctal 5000 Imaging of Rubber Fatigue Test Specimens

Next, an example of imaging samples with an Xctal 5000 phase contrast X-ray CT system (Fig. 7) is described. The Xctal 5000 can visualize variations in the X-ray phase shift. It can be used to observe groups of microstructures over a large field of view and perform high-sensitivity scans of light element materials. These actions are not possible with typical X-ray inspection systems.

In this case, fluoroscopy and cross-sectional images were acquired from sample 1 (1000 cycles). Fig. 8 shows the sample being scanned. With the Xctal 5000, samples were scanned in the vertical position without stretching.



Fig. 7 Xctal[™] 5000 Phase-Contrast X-Ray CT System

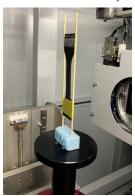


Fig. 8 Imaging Sample with the Xctal 5000

As mentioned above, the imaging method (phase imaging) used by the Xctal 5000 is different to the method typically used for imaging (absorption imaging). With absorption imaging, X-ray quantity values acquired with a detector are directly visualized as an absorption image, whereas with phase imaging, a diffraction grating is moved to acquire multiple fluoroscopic images that are used to calculate a dark-field image or a phase image.

The contrast level of absorption images is determined by the quantity of X-rays absorbed by the object. Therefore, microcracks that are smaller than the available resolution are difficult to visualize because they are hidden within the background contrast level (Fig. 9(a)). But the contrast level of X-ray dark-field images acquired by phase imaging is determined by the quantity of X-ray scattering. Given that microcracks are prone to X-ray scattering, even cracks smaller than the resolution level can be visualized due to the difference in contrast between the crack and base material (Fig. 9(b)). That means both microcracks and large overview images can be observed at the same time.

Consequently, samples can be scanned with a simple setup and no stretching, such as being held between two rods as shown in Fig. 8. Because microcracks can be observed even in overview images, magnified imaging is not necessary.

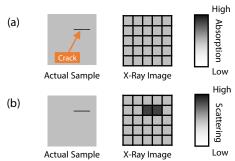


Fig. 9 Differences between Absorption Imaging and Phase Imaging (Dark-Field) (a) Absorption Imaging (b) Phase Imaging (Dark-Field)

■ Xctal 5000 Fluoroscopy

Scan settings for fluoroscopy are indicated in Table 5. In addition, a fluoroscopic image (X-ray absorption) of sample 1 is shown in Fig. 10(a); a fluoroscopic image (dark-field) is shown in Fig. 10(b); and magnified views of the crack areas are shown in Fig. 11.

In the absorption image, the defects appear white, and inclusions with higher density than the base material appear black. Consequently, in Fig. 11(a), inclusions, which are indicated with blue arrows, are visible, with dark-field images but defects are difficult to identify. In contrast, small cracks and inclusions prone to X-ray scattering both appear black, so it is not possible to distinguish between inclusions (blue arrows) and cracks (black arrows) based on Fig. 11(b) alone. So inclusions can be distinguished from cracks in absorption images, while the position and approximate size of cracks can be observed in dark-field images.

Thus, even microcracks that are much smaller than the exposure field can be easily and clearly detected in dark-field images. Although long narrow samples were scanned in this example, cracks can be detected in the same way regardless of the sample size, which is especially useful for imaging large samples when magnified imaging is not possible.

Table 5 Fluoroscopic Scan Settings

Parameter	Setting	
rarameter	setting	
Tube Voltage	40 kV	
Tube Current	800 μΑ	
Grating Angle	0°	
Number of Scans	10	
FOV (Exposure Field)	40.9 mm	
Resolution	0.018 mm	
Exposure Time	7 min	

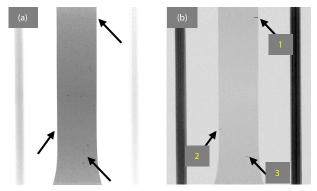


Fig. 10 Fluoroscopic Images of Sample 1 (a) Absorption Image (b) Dark-Field Image

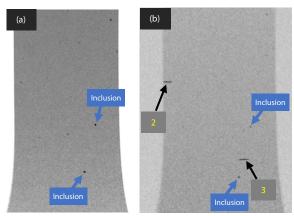


Fig. 11 Magnified (Digital Zoom) Fluoroscopic Images of Sample 1 (a) Absorption Image (b) Dark-Field Image

■ Xctal 5000 X-Ray CT Imaging

Lastly, the Xctal 5000 was used for X-ray CT imaging. The scan settings are indicated in Table 6.

Front and side cross-sectional images obtained by X-ray CT imaging are shown in Fig. 12 and 13, respectively. The ability of the Xctal 5000 to simultaneously acquire both absorption and dark-field images allows the same cross-sections to be displayed side-by-side. The crosssectional images that are circled in Fig. 12 and 13 are of the crack that is labeled "3" in the fluoroscopic image in Fig. 10. Cracks in absorption images have similarly low contrast levels as fluoroscopic images, but cracks can be observed with high contrast in dark-field images. In cross-sectional images, only cracks present in that particular crosssection can be observed, so not all cracks can be detected at the same time. However, the ability to acquire side-view cross-sectional images enables the depth of cracks to be observed.

This article only describes the scanning of one sample (sample 1), but it is also possible to observe and compare the number, depth, approximate position, and other information about cracks in the same specimen by acquiring both fluoroscopic and CT images after successive cycle count increases.

Table 6 X-Ray CT Scan Settings

Table 6 A hay of Scall Settings		
Parameter	Setting	
Tube Voltage	40 kV	
Tube Current	800 μΑ	
Grating Angle	0°	
Number of Views	1500	
Average Count	4	
FOV (Exposure Field)	40.9 mm	
Voxel Size	0.040 mm	
Exposure Time	258 min	



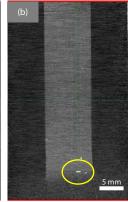
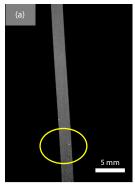




Fig. 12 Front-View Imaging (a) Absorption Image (b) Dark-Field Image



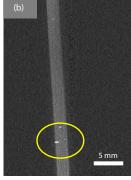




Fig. 13 Side Cross-Sectional Image (a) Absorption Image (b) Dark-Field Image

Xctal is a trademark of Shimadzu Corporation or its affiliated companies in Japan and/or other countries.



Shimadzu Corporation

www.shimadzu.com/an/

■ Conclusion

Specimens for testing rubber fatigue were scanned using two types of X-ray imaging systems, a microfocus X-ray CT system and a phase-contrast X-ray CT system. By stretching samples, magnified images were acquired by the inspeXio SMX-225CT FPD HR Plus X-ray CT inspection system, and even small defects could be observed in detail and defect volume and count values compared at respective cycle count levels. The Xctal 5000 can determine the approximate orientation, size, and depth of microcracks without stretching samples or using magnified imaging. Both instruments can inspect items non-destructively, and the same specimen can be used to understand the progression of defects and cracks at any number of cycles. The characteristics of these instruments for scanning and analyzing data from specimens tested for rubber fatigue are indicated in

Table 7 Characteristics of the Instruments

	inspeXio SMX-225CT FPD HR Plus	Xctal 5000
Advantages	Enables detailed volume and count analysis of 10 ⁻⁵ mm ³ or larger microdefects	Does not require stretching samples during imaging Does not require magnified imaging Enables efficient observation of the approximate position and size of microcracks
Points that Require Improvement	Requires stretching samples during imaging Magnification is required to analyze small defects	 Not suitable for detailed analysis of crack shapes, volumes, etc. Scans take a relatively long time

Acknowledgments

We are deeply grateful to the Chemicals Evaluation and Research Institute, Japan for their generous help with preparing this article, such as by providing samples, jigs, and advice about fatigue testing parameters.

01-00724-EN

First Edition: Nov. 2024

For Research Use Only. Not for use in diagnostic procedures.
This publication may contain references to products that are not available in your country. Please contact us to check the availability of these

products in your country.
The content of this publication shall not be reproduced, altered or sold for any commercial purpose without the written approval of Shimadzu.
See http://www.shimadzu.com/about/trademarks/index.html for details.
Third party trademarks and trade names may be used in this publication to refer to either the entities or their products/services, whether or not

they are used with trademark symbol "TM" or "®". Shimadzu disclaims any proprietary interest in trademarks and trade names other than its own

The information contained herein is provided to you "as is" without warranty of any kind including without limitation warranties as to its accuracy or completeness. Shimadzu does not assume any responsibility or liability for any damage, whether direct or indirect, relating to the use of this publication. This publication is based upon the information available to Shimadzu on or before the date of publication, and subject to change without notice.

Related Products Some products may be updated to newer models.



Related Solutions

