Visualization of dual-phase structure in duplex stainless steel

Since the discovery of X-rays by Röntgen [1] in 1896, absorption has been a dominant principle of X-ray imaging techniques. Conventional imaging techniques such as radiography and computed tomography (CT) rely on the decrease in X-ray beam intensity (attenuation) when traversing an object, which can be measured directly using an X-ray detector. As well known, however, this mechanism of image formation often provides poor contrast when imaging light-element materials such as polymers and biological tissues, as well as when imaging multiphase objects with a small difference in intensity between multi-composed structures such as the ferrite-austenite duplex stainless steel considered in this study. The difference in theoretical linear attenuation coefficient between two phases was only about 1.1 cm^{-1} (3.1% difference) at an X-ray energy of 37.7 keV owing to their quite similar chemical compositions. Various alternative methodologies have been developed to overcome such poor contrast [2]. Around the time of the development of laboratory-based micro-CT systems in the mid-1960s, in addition to absorption, creating X-ray images using the refraction of X-rays by matter was attempted [3]. This type of phase-contrast imaging (PCI) greatly enhances the visibility of weakly absorbing features in low density objects that barely absorb X-rays. PCI is different from the absorption contrast technique: It uses information concerning changes in the phase of an X-ray beam that passes through the object in order to create its images. Moreover, it enables the different phases to be distinguished even if their attenuation coefficients are very similar, which would be impossible with the absorption contrast technique alone.

Over the last several years, a variety of PCI techniques have been developed, all of which are based on the observation of interference patterns between diffracted and undiffracted waves. The most commonly used PCI techniques are propagation-based imaging, crystal interferometry, analyzer-based imaging, edge illumination and grating-based imaging. The propagation-based imaging is the most common name of the first technique and is also called in-line phase-contrast, in-line holography, refraction-enhanced imaging or phase-contrast radiography. A simple but powerful approach to realizing a higher imaging sensitivity when using synchrotron light sources is the propagation-based imaging method used in this study. Its experimental setup is basically the same as that of conventional radiography. It consists of an in-line arrangement of an X-ray source, a sample and an X-ray detector and no other optical elements are required (Fig. 1). By leaving an appropriate drift space between the sample and the imaging detector, interfaces within the probed sample can be visualized (Fig. 2(a)). If the X-ray wavefront distorted by the sample has a sufficient degree of transverse coherence, Fresnel diffraction on microscale structures will lead to interference fringes that enhance the edges and interfaces of the sample in the recorded radiograph. Compared with the conventional absorption contrast technique, PCI allows us to study samples either with negligible absorption or with many different components that show too similar degrees of absorption to be discriminated such as duplex metals.

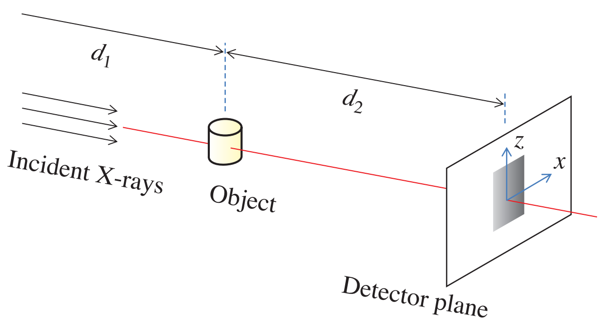

Fig. 1. Experimental setup of propagation-based imaging with single distance d_2 for duplex steel.

Owing to the weak refraction of X-rays compared with the light, specialized conditions are required to make use of X-ray refraction in the imaging. The X-ray beam should have a high spatial coherence and a significant distance between the sample and the detector d_2 is required. Experimental Hutch 2 of beamline **BL20XU**, which is used in this study, provides highly coherent beams sufficient for meeting the above first requirement, and the distance of the beam source from the sample, d_1, is 245 m. In the case of the parallel beam source used in this study, PCI fringe intensity increases with the propagation distance d_2, but the fringes broaden out. Therefore, a wide d_2 range from 8 to 1200 mm was considered to compare the visibility of fringes at the interfaces of duplex structures with the change in X-ray energy from 37.7 to 78 keV.

Although raw information on PCI is often useful for visual inspection with the naked eye like biomedical inspection, any further quantitative analysis, which is necessary for the segmentation of the tomographic

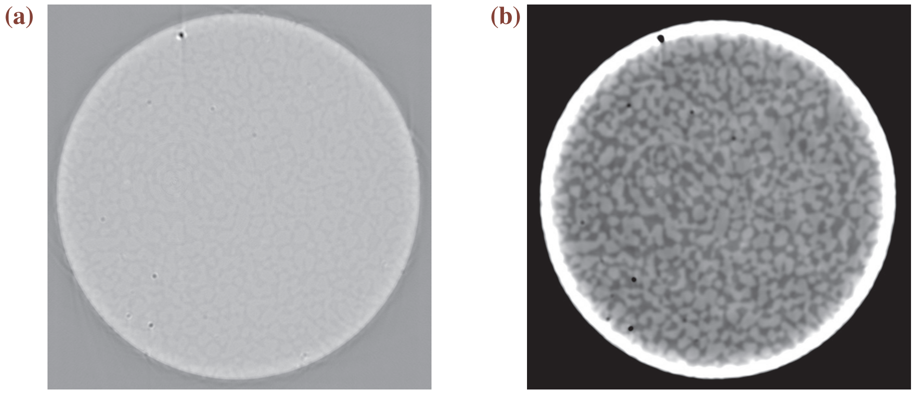

Fig. 2. Cross-sectional images of duplex steel **(a)** before and **(b)** after phase retrieval process at d_2 = 800 mm.

volume, is not easily possible using the raw information on PCI. This is because the gray levels in the multiple material regions are not too different to be divided into different segments; they only vary at the interfaces. However, if transmission radiographs are sent through an appropriate phase retrieval process, tomographic reconstruction can be performed and the tomograms will exhibit area contrast rather than edge-enhancing contrast (Fig. 2(b)). With subsequent post-processing such as noise filtering, a virtual 3D structure consisting of a complex dual-phase microstructure was successfully reconstructed, as shown in Fig. 3.

A variety of phase retrieval methods for PCI data have been developed since the mid-1990s. Although their algorithms are iterative or use analytical approaches, many methods require a large number of raw images obtained at different distances d_2 as well as numerous restrictions [3]. A single-distance phase-retrieval method developed by Paganin *et al.* [4] was chosen in this study, which is relatively simple and widely used in practical imaging. This particular algorithm retrieves the phase from radiographs obtained at a single distance d_2. Through a well-established multistage process of PCI mentioned above, 3D volumetric analysis for many types of duplex steels, such as ferrite-martensite, ferrite-cementite, and ferrite-austenite steels, has entered a new phase. The CT technique as an analytical tool for engineering takes a step forward once more via the success of this application, but many challenges still lay ahead, for example, PCI of heterogeneous metals consisting of more than two phases or improving the phase retrieval algorithm to fix dummy artifacts occurring at interfaces of defects inside samples.

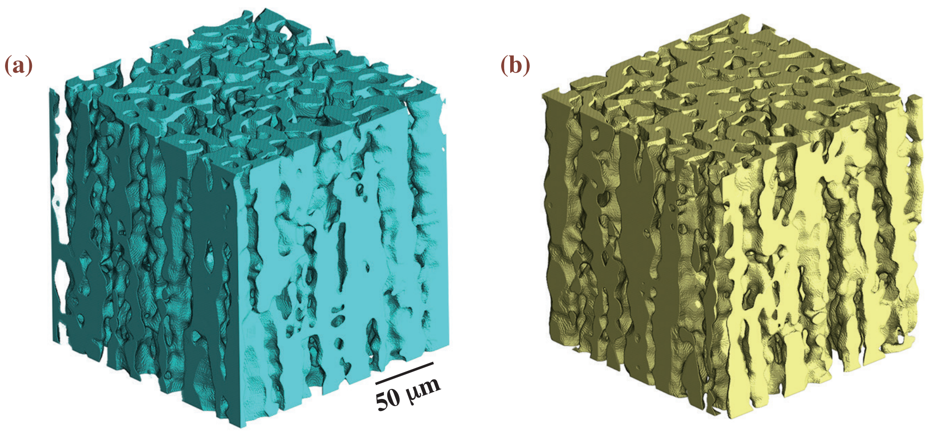

Fig. 3. Virtual 3D structures of duplex steel consisting of **(a)** ferrite and **(b)** austenite phases were reconstructed by a single-distance phase retrieval process.

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References

[1] W.C. Röntgen: Nature **53** (1896) 274.
[2] H. Toda *et al*.: Metall. Mater. Trans. **37A** (2006) 1211.
[3] P. Cloetens *et al*.: J. Appl. Phys. **81** (1997) 5878.
[4] D. Paganin *et al*.: J. Microsc. **206** (2002) 33.