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Diffraction contrast tomography for the study of polycrystalline stainless steel microstructures and stress corrosion cracking

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Key words:
Diffraction contrast tomography; synchrotron radiation; stainless steel; stress corrosion cracking

Abstract – X-ray diffraction contrast tomography is a non-destructive technique for the 3D characterisation of polycrystalline microstructures containing up to a few 1000 grains. The sample is illuminated with a monochromatic beam of high energy synchrotron radiation. As the sample is rotated, and as grains pass through alignments for Bragg diffraction, diffraction spots are recorded on a 2D detector placed close behind the sample. The diffraction geometry is used to assign spots to the grains from which they arise, and to determine the crystallographic orientations of grains. The spots are used as projections of the grains to reconstruct the grain shapes. The technique has been applied to several materials science investigations in stainless steels, including the 3D characterisation of grain boundary networks, and in-situ studies of intergranular stress corrosion cracking.

Mots-clés :
Tomographie en contraste de diffraction ; synchrotron ; acier inoxydable ; corrosion inter granulaire sous contraintes

Résumé – La tomographie en contraste de diffraction pour les études des microstructures polycristallines d'acier inoxydable et des fissures de la corrosion inter granulaire sous constraintes. La tomographie en contraste de diffraction est une technique non destructive pour la caractérisation en trois dimensions des microstructures polycristallines comprenant jusqu'à plusieurs milliers de grains. L'échantillon est illuminé avec un faisceau synchrotron monochromatique, de haute énergie. Pendant la rotation de l'échantillon, les grains sont orientés de manière à diffracter le faisceau selon la loi de Bragg. Les tâches de diffraction sont détectées sur un détecteur 2D situé à quelques millimètres après l'échantillon. La géométrie de la diffraction est utilisée pour associer les tâches avec les grains d'origines et pour calculer l'orientation cristalline de ces grains. Les formes des tâches correspondent à la projection des grains et sont utilisées pour reconstruire leur forme respective. La technique a été utilisée pour quelques études sur des aciers inoxydables, par exemple pour la caractérisation 3D des réseaux des bords des grains, et des études in situ de la corrosion inter granulaire sous contraintes.

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Intergranular stress corrosion cracking (IGSCC) in stainless steels is a subject of great current interest due to being a possible damage mechanism in stainless steel components of the cooling circuits in light water reactors. Grain boundaries can become sensitised due to irradiation or thermal cycling. Certain combinations of loading and environmental conditions can then result in crack propagation along the sensitised boundaries, eventually leading to failure [1].

IGSCC is a complex, three dimensional problem, affected by the polycrystalline microstructure of the material. Failure results from the propagation of cracks along sensitised grain boundaries, often at relatively low stresses. Non-sensitised grain boundaries have been shown to retard crack propagation by forming ligaments bridging the advancing crack [2]. The geometry of a grain boundary determines whether or not it will be susceptible to sensitisation. The crystallographic orientations of the adjoining

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grains and the crystallographic plane of the grain boundary are both important aspects of this geometry. Standard two dimensional microstructural characterisation techniques such as EBSD reveal the orientation of surface grains, but not the plane of the grain boundaries, as only intersection of the boundary plane with the surface is visible. Furthermore, only surface information is available, so if the technique is extended to 3D grain mapping by serial sectioning it is necessarily destructive, making in-situ studies impossible. There is therefore a clear motivation for non-destructive, 3D characterisation techniques.

1 Three dimensional grain mapping techniques

X-ray diffraction contrast tomography (DCT) and 3DXRD are non-destructive, 3D orientation mapping techniques using synchrotron radiation [3–5]. Grain shapes and orientations can be determined in samples containing several thousand grains. From the grain shapes and orientations, full microscopic descriptions of all grain boundaries can be calculated. The nondestructive grain mapping can be combined with in-situ observations of intergranular stress corrosion cracking by absorption or phase contrast synchrotron X-ray microtomography. By combining the datasets, the grain boundaries along which cracks propagate can be identified, and the characteristics of boundaries susceptible or resistant to IGSCC can be identified, providing valuable input to efforts to develop more resistant microstructures [6]. Such tools for 3D, non-destructive characterisation of polycrystalline materials are therefore of the utmost interest to the study of IGSCC in stainless steels.

The basic experimental apparatus used for DCT is shown in Figure 1. The sample is illuminated by a beam of parallel, monochromatic synchrotron radiation. A 2D area detector is placed close behind the sample. This central part of this detector captures an absorption contrast image, or radiograph. In addition, when the sample is rotated grains pass through alignments which fulfil the conditions for Bragg diffraction. When this occurs, a diffraction spot is formed which will be recorded by the detector. An additional extinction contrast is observed superimposed on the absorption image. The diffraction spot can be treated as a projection of the grain volume. By recording images as the sample is rotated through 360°, many such spots are recorded for each grain. Spatial and crystallographic criteria allow the spots can be associated with the grains from which they arise, and for the crystallographic orientations of the grains and their positions in the sample to be determined. Finally, the 3D grain shapes are reconstructed using the diffraction spots as grain projections [4, 7]. Figure 2 shows a 3D computer rendered image of such a grain map, combined with the path of a IGSCC measured using synchrotron microtomography.

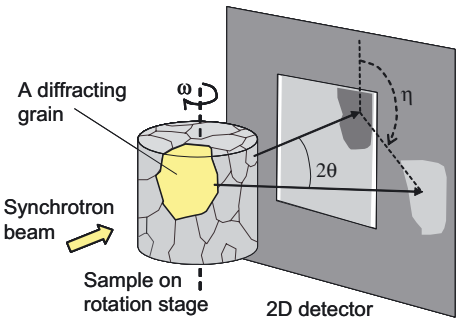


Fig. 1. Diffraction contrast tomography experiment.
Fig. 1. Tomographie en contraste de diffraction.



Fig. 2. Rendering of a grain map produced by DCT (colours) combined with an IGSCC (white) (colour online at www.revue-metallurgie.org).
Fig. 2. Représentation de plan trois dimensionnel des grains fait par la tomographie en contraste de diffraction (en couleur) combinée avec une fissure de corrosion inter granulaire sous contraintes (blanc) (en couleurs dans la version électronique, www.revue-metallurgie.org).

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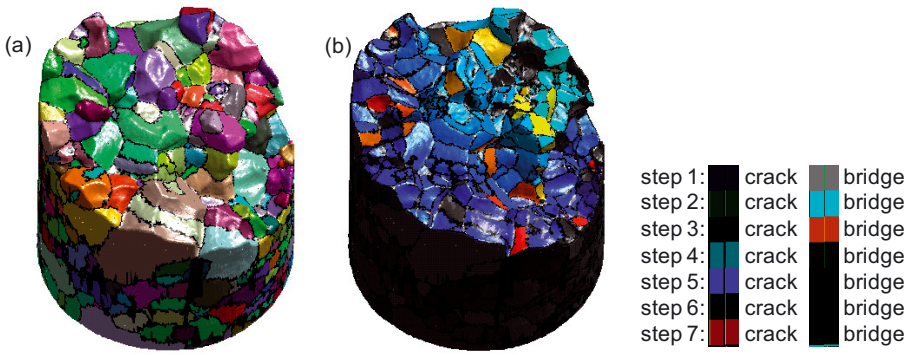


Fig. 3. (a) Rendering of the grains in a stainless steel fracture surface. (b) Coloured according to the propagation of the crack and the bridging ligaments formed.
Fig. 3. (a) Représentation des grains dans la surface d'une rupture d'acier inoxydable. (b) Représentation colorée en fonction de la propagation de la fissure et des « pontages ».

The non-destructive nature of the techniques allows the temporal evolution of polycrystalline microstructures to be studied. DCT can reveal changes in grain shapes and orientations during annealing, or during limited plastic deformation. In combination with synchrotron microtomography, the temporal evolution of IGSCC has been studied, revealing the locations of grain boundaries which are resistant to sensitisation and so form bridges across the crack. These bridges may be temporary features, forming and then failing as the crack advances further, so post-mortem techniques are of limited use in their characterisation. Figure 3 shows a 3D rendered image of the grains present in one half of a sample fractured by IGSCC, and the same rendering coloured according to the evolution of the crack front [8]. Grain boundaries forming bridges during the crack propagation are identified by a different colour scale. Crystallographic properties of these boundaries may be determined from the DCT grain map [6].

2 The HEMS beamline, Petra III

The High Energy Materials Science beamline at the new Petra III synchrotron radiation source at DESY research centre in Hamburg, Germany, will feature an instrument dedicated to such DCT and 3DXRD grain mapping experiments. Other instruments will be available for diffraction based measurements of stress, strain and texture in engineering components and for microtomography. The beamline is currently in commissioning, and the grain mapping instrument is expected to be constructed in 2011. An overview of the grain mapping instrument is shown in Figure 4. This is designed to allow different beam optics and detectors to be quickly and easily interchanged, so complementary techniques can be combined, and new methodologies tested [9].

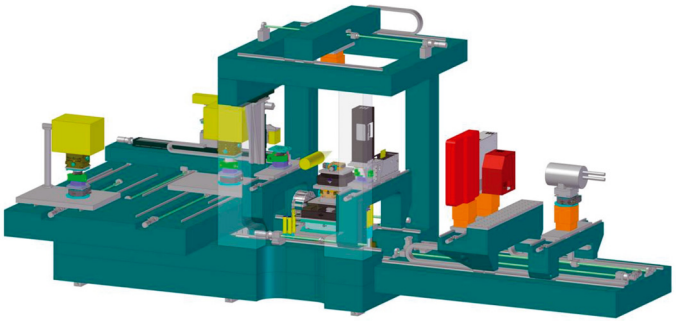


Fig. 4. The grain mapping instrument that will be constructed at the HEMS beamline.
Fig. 4. L'instrument de cartographie des grains qui va être construit sur la ligne de lumière de l'HEMS.

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