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Determination of composition, residual stress and stacking fault depth profiles in expanded austenite with energy-dispersive diffraction

S. Jegou [a,*,1], T.L. Christiansen [a], M. Klaus [b], Ch. Genzel [b], M.A.J. Somers [a]

[a] Technical University of Denmark, Dept. Mechanical Engineerings, Kgs. Lyngby, Denmark
[b] Helmholtz-Zentrum Berlin für Materialien und Energie GmbH, Berlin, Germany

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ABSTRACT

A methodology is proposed combining the scattering vector method with energy dispersive diffraction for the non-destructive determination of stress- and composition-depth profiles. The advantage of the present method is a relatively short measurement time and avoidance of tedious sublayer removal; the disadvantage as compared to destructive methods is that depth profiles can only be obtained for depth shallower than half the layer thickness. The proposed method is applied to an expanded austenite layer on stainless steel and allows the separation of stress, composition and stacking fault density gradients.

1. Introduction

Residual stresses are widely and deliberately introduced within the near surface region of materials to locally modify the mechanical properties and enhance the component performance with respect to wear and/or fatigue. Surface engineering associated with tailoring of the surface properties and residual stress can be achieved by thermal, chemical or mechanical treatment [1] and yields a functionally graded material that changes its properties from surface to interior. The quantification of residual stress-depth profiles to investigate the effect of the surface engineering treatment can be performed by X-ray diffraction analysis [2]. This technique relies on the determination of *hkl* specific lattice strains for various orientations of the scattering vector with respect to the sample surface normal combined with an appropriate grain-interaction model [3]. Numerous factors affect the so-called X-ray diffraction stress analysis, e.g. grain size, triaxiality of the stress state and preferred orientation. The evaluation of stress-depth profiles in functionally graded materials can be influenced by the stress gradient itself, as well as by other gradients. Steep residual stress gradients can lead to the so-called ghost stresses, i.e. systematic errors inherent to the applied measurement and/or evaluation procedure, if no precautions are taken.

When superimposition of composition and stress gradients occurs, such as for a composition-induced stress gradient, stress evaluation over the information depth also depends on composition, because the reference spacing is composition dependent. This can lead to dramatic ghost stresses if not taken into account during data acquisition and evaluation [4,5].

Among the various techniques developed for non-destructive depth resolved stress determination [3,6–9], energy-dispersive diffraction methods, using white radiation, give some advantages associated with multiple reflections recorded in one energy spectrum and deeper information depths [10–13]. Stress-induced errors can effectively be avoided combining a modified multi-wavelength approach with the $\sin^2\psi$ method or the scattering vector method [14]. In [15] it was shown that the energy-dispersive method can be applied even to the detection of very steep in-plane residual stress gradients in surface treated hard coatings, if the information depth is adapted to the steepness of the gradient. However, for a composition-induced (self-induced) stress gradient, the 'optimisation procedure' developed for the scattering vector method cannot be applied straightforwardly, because the lattice spacing in the strain-free direction varies with the information depth. Instead a $\sin^2\psi$-based approach should be considered, where $\sin^2\psi$ dependencies at prechosen information depths are evaluated by interpolation among the experimental data. The reference lattice parameter for the appropriate information depth follows from interpolation among the data in the strain free direction or from independent spectroscopic analysis and knowledge of the relation between lattice parameter and composition.

This work deals with the evaluation of residual stress by means of non-destructive energy-dispersive diffraction under the influence of steep stress- and composition gradients. Steep superimposed multigradients arise after low temperature thermochemical surface treatments of stainless steel [16]. Such treatments (nitriding, carburising or nitrocarburising) give rise to the formation of a surface zone of

* Corresponding author at: Arts & Métiers ParisTech, MécaSurf Laboratory, 2 cours des Arts et Métiers, 13617 Aix-en-Provence, France. Tel.: +33 442938174; fax: +33 442938114.
E-mail addresses: sebastien.jegou@ensam.eu (S. Jegou), tch@mek.dtu.dk (T.L. Christiansen), klaus@helmholtz-berlin.de (M. Klaus), genzel@helmholtz-berlin.de (C. Genzel), somers@mek.dtu.dk (M.A.J. Somers).
[1] Now with: Arts & Métiers ParisTech, MécaSurf Laboratory, Aix-en-Provence, France.

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so-called expanded austenite which essentially is a solid solution of colossal amounts of interstitials (carbon and/or nitrogen) in the austenite lattice. This results in biaxial compressive residual stresses of several GPa's that find their origin in the lattice misfit between the expanded austenite “case” and the untreated core [16,17].

2. Non destructive depth profiling with energy-dispersive X-ray stress analysis

X-ray stress analysis is based on the lattice strain measurement $\varepsilon_{\varphi\psi}{}^{hkl}$ experienced by a set of lattice planes $\{hkl\}$ in a given direction defined by the azimuth, φ, and inclination, ψ, with respect to the sample surface normal (Fig. 1):

$$\varepsilon_{\varphi\psi}^{hkl} = \frac{d_{\varphi\psi}^{hkl}}{d_o^{hkl}} - 1 \tag{1}$$

where d_o^{hkl} is the unstrained lattice spacing.

In energy-dispersive diffraction using a white beam, measurements are carried out for fixed and predetermined diffraction and scattering angles. The Bragg equation then takes the following form:

$$d^{hkl} = \frac{hc}{2\sin\theta}\frac{1}{E^{hkl}} \tag{2}$$

where 2θ is the scattering angle, h is Planck's constant, c is the velocity of light and E^{hkl} is the energy for which diffraction of the hkl lattice planes occurs.

Introducing Eq. (2) in Eq. (1) gives the lattice strain $\varepsilon_{\varphi\psi}^{hkl}$ in the measuring direction defined by φ and ψ as:

$$\varepsilon_{\varphi\psi}^{hkl} = \frac{E_o^{hkl}}{E_{\varphi\psi}^{hkl}} - 1 \tag{3}$$

where E_o^{hkl} corresponds to the unstrained lattice spacing d_o^{hkl}.For surface engineered quasi-isotropic polycrystalline materials usually a state of rotationally symmetric biaxial stress ($\sigma_{13} = \sigma_{23} = \sigma_{33} = 0$ and $\sigma_{11} = \sigma_{22} = \sigma_{//}$) can be assumed, leading to:

$$\varepsilon_{\psi}^{hkl}(z) = 2S_1^{hkl}\sigma_{//}(z) + \frac{1}{2}S_2^{hkl}\sigma_{//}(z)\sin^2\psi \tag{4}$$

where S_1^{hkl} and $1/2S_2^{hkl}$ are diffraction elastic constants, depending on the crystal orientation hkl and elastic interaction among the crystals. The lattice spacing, $\langle d_\psi^{hkl}\rangle$ (or equivalently the energy at which diffraction occurs) determined in an X-ray diffraction experiment for a

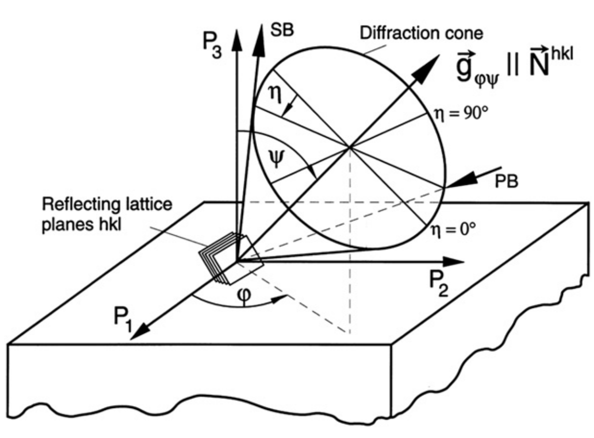

Fig. 1. Diffraction geometries in X-ray stress analysis from [17]. η denotes the rotation of the sample around the scattering vector $\vec{g}_{\varphi\psi}$ for a fixed measuring direction (φ,ψ) with respect to the sample system P. PB and SB denote primary and secondary (diffracted) beam.

sample (or layer) of thickness, t, is the diffracted intensity-weighted average over depth, z, i.e.:

$$\left\langle d_\psi^{hkl}\right\rangle = \frac{\int_0^t d_\psi^{hkl}(z)\exp\{-\mu(E)kz\}dz}{\int_0^t \exp\{-\mu(E)kz\}dz} \tag{5a}$$

where, for measurement in reflection geometry (as practised in the present work)

$$k = \frac{2\sin\theta\cos\psi}{\sin^2\theta - \sin^2\psi + \cos^2\theta\sin^2\psi\sin^2\eta} \tag{5b}$$

describes the diffraction geometry, $\mu(E)$ is the linear absorption coefficient which, for a homogeneous layer, depends on the photon energy and η denotes the rotation angle around the scattering vector, $\vec{g}_{\varphi\psi}$ (Fig. 1). For completeness it is mentioned that $\mu(E)$ depends on composition. This second order effect is not considered here.[2] Hence, it is obtained for the lattice strain, averaged over the diffracting volume, $\langle\varepsilon_\psi^{hkl}\rangle$:

$$\left\langle \varepsilon_\psi^{hkl}\right\rangle = \frac{\int_0^t d_\psi^{hkl}(z)\exp\{-\mu(E)kz\}dz}{\int_0^t d_o^{hkl}(z)\exp\{-\mu(E)kz\}dz} - 1 \tag{6}$$

Note that these equations are only valid for the case where the studied layers are well within the gauge volume. From Eq.(6) it is observed that the lattice strain evaluated for experimental lattice spacings has to be evaluated from strained and unconstrained lattice spacings weighted over the same depth range. This lattice strain can be assigned to the information depth, τ:

$$\tau(E) = \langle z\rangle = \frac{\int_0^t z\cdot\exp\{-\mu(E)kz\}dz}{\int_0^t \exp\{-\mu(E)kz\}dz} = \frac{1}{\mu(E)k} + t\frac{\exp\{-\mu(E)kt\}}{\exp\{-\mu(E)kt\}-1} \tag{7}$$

Note that the information depth in a layer is maximally $t/2$ for the case where the layer can be considered infinitely thin as compared to the penetration of the X-rays.[3] For an infinitely thick layer the information depth equals $1/[\mu(E)k]$, which for the present case amounts to 27 µm. It is important to realise that, in general, $\langle d_\psi^{hkl}\rangle$ and $\langle d_o^{hkl}\rangle$ are not experimentally determined at the same information depth, because the strain-free lattice spacing applies only for one specific value for ψ (and thus τ), the so-called strain-free direction, ψ_o, defined by $\sin^2\psi_o = -\frac{2S_1^{hkl}}{\frac{1}{2}S_2^{hkl}}$ (as obtained by equating Eq. (4) to zero). Consequently, application of Eq. (6) requires that a value for $\langle d_o^{hkl}\rangle$ at τ_ψ is obtained by interpolation among the experimentally determined strain-free lattice spacing-depth profile $\langle d_o^{hkl}(z)\rangle$.

In the case of stress-depth profiling, various methods have been developed, based on either successive layer removal (destructive methods) or assigning the evaluated data to a depth below the surface (non-destructive methods). According to Eqs. (7) and (5b) for a fixed value of θ the information depth can be varied by variation of the angles ψ and η or, for energy dispersive analysis, by selecting another energy E where diffraction occurs. In the present work the scattering vector method (varying η and ψ) and the multi-wavelength method (varying E and ψ) are combined for non-destructive depth profiling of the composition, stress and stacking fault probability in low temperature hardened stainless steel.

[2] For the present case where a layer of expanded austenite on stainless steel is considered, the error in assuming $\mu(E)$ independent of depth (i.e. a homogeneous layer) lies in the range 3.94 to 4.39% for a composition ranging from $y_N = 0.30$ to $y_N = 0.50$ (cf. Fig. 4).

[3] For ‘Real space’ method, the measuring depths are not limited to $t/2$ since the gauge volume is used to define the observed volume.