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On the determination of stress profiles in expanded austenite by grazing incidence X-ray diffraction and successive layer removal

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Abstract—Surface layers of expanded austenite resulting from nitriding typically exhibit large gradients in residual stress and composition. Evaluation of residual-stress profiles is explored by means of grazing incidence X-ray diffraction (GI-XRD), probing shallow depths, combined with successive layer removal. Several factors complicating the stress determination are analysed and discussed: (1) ghost stresses arising from a small variation in the shallow information depths probed with GI-XRD, (2) selection of the grain interaction model used to calculate the X-ray elastic constants for conversion of lattice strains into residual stress and (3) the composition dependence of these elastic constants.

Keywords: Low temperature surface hardening; Gas nitriding; Expanded austenite; X-ray diffraction; Stress analysis

1. Introduction

Although the first exploitation of expanded austenite dates back about 30 years [1,2], it continues to be a topic of research and discussion in the current literature. While plasma-based processes for the surface hardening of stainless steel have dominated the first 15 years of intensive investigation, later years have seen the advent of and a growing interest in gaseous processing [3,4]. The transformation of the surface region of austenitic stainless steel into a case of expanded austenite is associated with a spectacular improvement of the wear and fatigue performance, while the corrosion performance remains unaffected, or is even improved [5].

Expanded austenite is obtained by interstitially dissolving colossal amounts of nitrogen and/or carbon into austenite [6,7] at a temperature that is too low to allow long range diffusion of substitutionally dissolved components in the alloy. Accordingly, the depth of the hard case brought about is entirely the result of (stress-assisted) interstitial diffusion of carbon/nitrogen atoms in austenite. No new phase develops and expanded austenite should be considered as a diffusion zone in austenite. As a consequence of the high content of interstitially dissolved nitrogen/carbon in existing austenite grains huge compressive residual stress is built up along with the interstitial concentration profile. Compressive residual stress values of 7.5 GPa in the plane parallel to the surface have been reported for probing the 200 reflection of expanded austenite [3]. Moreover, plastic accommodation and associated relaxation of the enormous composition induced stresses were observed as grain push-out [7,8], lattice rotations [9–11] and enhanced stacking fault densities [12].

Depth-resolved quantification of composition-induced stress profiles in expanded austenite with X-ray diffraction techniques is far from trivial, as apart from an influence of the stress gradient on the local lattice spacing, also gradients in composition and stacking-fault density affect the lattice spacing [6]. Both destructive and non-destructive measurement strategies and data correction procedures have been published in the latter years to unravel the contributions of stress, composition and stacking fault gradients on lattice spacing profiles [13,14]. The application of an asymmetric path by grazing incidence allows X-rays to probe only a very shallow depth range under the exposed surface and thus minimises the effect of gradients. In the present article this technique is combined with successive layer removal to analyse the effects of steep gradients in stress and composition on the determined stress profiles in an expanded austenite case obtained by gaseous nitriding.

1.1. X-ray diffraction stress analysis in expanded austenite zones

Surface layers obtained by thermochemical surface engineering can usually be assumed to experience a rotationally symmetric biaxial state of macroscopic (Type 1)

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stress, implying that $\sigma_{11} = \sigma_{22} = \sigma_{\parallel}$, which leads to a simplification of the dependence of the lattice strain, ε_{ψ}^{hkl}, of the family of lattice planes $\{hkl\}$ on the tilt angle ψ:

$$\varepsilon_{\psi}^{hkl} = \frac{d_{\psi}^{hkl} - d_{\varepsilon=0}^{hkl}}{d_{\varepsilon=0}^{hkl}} = 1/2S_2^{hkl}\sigma_{\parallel}\sin^2\psi + 2S_1^{hkl}\sigma_{\parallel} \tag{1}$$

where d_{ψ}^{hkl} is the lattice spacing for the same $\{hkl\}$ planes in the direction defined by ψ, $d_{\varepsilon=0}^{hkl}$ is the strain-free lattice spacing and S_1^{hkl} and $1/2S_2^{hkl}$ are X-ray elastic constants (XECs) depending on the material and on the $\{hkl\}$ indices. The strain-free lattice spacing, $d_{\varepsilon=0}^{hkl}$, in Eq. (1) is probed for the so-called strain-free measurement direction, $\psi_{\varepsilon=0}$, which is obtained from equating Eq. (1) to zero and rearranging terms:

$$\sin^2\psi_{\varepsilon=0} = \frac{-2S_1^{hkl}}{1/2S_2^{hkl}}. \tag{2}$$

After determination of $d_{\varepsilon=0}^{hkl}$ it is straightforward to obtain the stress from Eq. (1).

Table 1. X-ray elastic constants for the {111} and {200} family planes according to different models. The values are given in 10^{-6} MPa^{-1}. The single crystal elastic constants from which the Voigt and Reuss constants are calculated for a randomly textured polycrystal are $s_{11} = 10.7$ MPa^{-1}, $s_{44} = 8.60$ MPa^{-1}and $s_{12} = -4.25$ MPa^{-1} [30]. The Kröner–Eshelby constants are from [31].

	Kröner–Eshelby		Voigt		Reuss	
	111	200	111	200	111	200
S_1^{hkl}	−1.1	−2.3	−1.3	−1.3	−0.7	−4.3
$1/2S_2^{hkl}$	5.1	8.83	6.01	6.01	4.3	15.0

The standard method known as the "$\sin^2\psi$" method employs the symmetric Bragg–Brentano geometry and leads to a significant variation of the information depth for different tilt angles. For the case of lattice spacing gradients these variations in information depth with tilt angle lead to ghost or fictitious stresses [15]. Avoiding such artefacts requires an effective correction procedure of the obtained lattice spacing results [13–15]. Successful application of the correction method firstly proposed in Ref. [15] for unravelling stress- and composition–depth profiles in γ'-Fe$_4$N$_{1-x}$ surface layers, was demonstrated for carbon- [16] and nitrogen-expanded austenite [3]. In these attempts a symmetrical diffraction method was applied, associated with relatively large (variations in) information depth, leading to broad asymmetric X-ray line profiles as a consequence of the very broad composition range for, in particular, nitrogen-expanded austenite. If a grazing incidence angle is applied, only a shallow depth is probed and the error made in the evaluated lattice spacing is much smaller, because narrower X-ray line profiles are obtained, while the variation of the information depth with ψ tilting is reduced importantly. In principle, an appropriate choice of the combination of grazing incidence angle with tilt angle allows probing the material at the same information depth for a range of tilt angles [17]. In this way several information depths can be probed non-destructively, as was demonstrated experimentally for vapour deposited Ni-layers [18] and ground Al$_2$O$_3$ [19]. In both these applications the samples investigated had only a stress–depth profile and were uniform in composition within the investigated depth range. Provided that the surface layer (or case) investigated diffracts independently from the bulk (or core) the maximum information depth that can be probed by this non-destructive technique corresponds to half the thickness of the surface layer, which for expanded austenite is associated with broad X-ray line profiles as a consequence of the composition–depth profile. Therefore, in this investigation, successive layer removal was applied combined with grazing incidence X-ray diffraction. Instead of keeping the information depth constant by varying the grazing incidence angle with ψ, in the present work the grazing incidence was kept fixed for all applied ψ tilts. Applying grazing incidence the lattice planes are actually probed in a direction that is tilted with respect to the surface normal even when no actual rotation, χ, over the Ψ-axis perpendicular to the Ω ($\omega/2\theta$)-axis is applied in the goniometer [16]. The effective tilt angle ψ that should be accounted for in the calculation of the stress is therefore:

$$\cos\psi = \cos\chi \cdot \cos(\theta - \alpha) \tag{3}$$

where α is the fixed grazing incidence angle, χ is the rotation angle around the Ψ-axis and 2θ is the Bragg angle.

The applicability of this method for the present case will be further explored in Section 4.1.

1.2. X-ray elastic constants and grain interaction models

The XECs can be calculated from the single crystal elastic constants, adopting an appropriate model for the elastic interaction between the grains in a polycrystal. The two extreme interaction models are the Voigt [20] assumption that all grains experience the same strain and the Reuss [21] model based on all grains having equal stress. Other interaction models have been devised, including self-consistent approaches by Eshelby [22] and Kröner [23], where the (anisotropic) grains probed interact with a matrix with isotropic properties averaging over all grain orientations. The Voigt and Reuss approaches were proven by Hill [24] to be the upper and lower bounds for the elastic modulus of a bulk polycrystal. This has suggested averaging of the results of the two models [25]. For the special conditions at free surfaces and two-dimensional grain interaction in the plane of the surface, the Vook–Witt model [26,27] assumes equal strains in the surface plane and a zero stress perpendicular to the surface, whilst the inverse Vook–Witt model [28] assumes equal stresses in the surface plane and equal strains perpendicular to the surface.

Irrespective of the grain interaction model (GIM) adopted, the single crystal elastic constants of the material considered are essential components in the calculation of the XECs for the polycrystal. Single crystal constants have not yet been determined for expanded austenite and the values reported for alloys with Cr and Ni contents in the range 12–18%, e.g. [29], have therefore often been used [30]. XECs derived from such single crystal values are listed in Table 1. XECs from the Kröner–Eshelby model (as given in [31]) are employed to discuss the effects of gradients in composition and stress, while the upper and lower bounds by Voigt and Reuss are the basis for discussion of GIM selection. Finally, the effects of nitrogen content on the XECs are analysed.

2. Experimental

2.1. Sample preparation

Discs with a diameter of 20 mm and a thickness of 3 mm were cut from a solution treated bar of AISI 316L with the