Materials Science & Engineering A 636 (2015) 476–483

Contents lists available at ScienceDirect

Materials Science & Engineering A

journal homepage: www.elsevier.com/locate/msea

Local residual stress measurements on nitride layers

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ARTICLE INFO

Article history:
Received 21 December 2014
Received in revised form
5 April 2015
Accepted 8 April 2015
Available online 16 April 2015

Keywords:
Nitriding
Residual stress
Slit method
Focused ion beam
Gradient

ABSTRACT

In this work, local stresses in different nitrided maraging steel samples of high practical interest for industrial applications were studied through the so-called micro-slit milling method using a focused ion beam. The nitrogen concentration profiles were acquired by glow discharge optical emission spectroscopy. The residual stress state was measured on the surface and also in cross-section, i.e. examining effects of the nitrogen concentration gradient. It is shown that an enhanced lateral resolution can be achieved when a novel multiple fitting approach is employed. The results presented show an overall agreement with stress profiles obtained by X-ray diffraction. Finite Element Modeling is used to explain the apparent discrepancies. A clear correlation between the residual stress and nitriding profiles has been found and the applicability of this method is shown in particular when stress gradients are present.

1. Introduction

Residual stresses are defined as those remaining after the extrinsic cause of loading, e.g. external forces, heat gradients, etc., is removed. They appear in almost all materials, and arise whenever anelastic processes are present, like plastic deformation, thermal treatments or surface modification. The contribution of residual stresses can be either beneficial or harmful, depending on the actual sign and location of the residual stress. As a consequence, the precise knowledge and control of residual stresses are of high practical value in applications [1,2].

In general the traditional methods for measuring residual stress are divided into non-destructive and destructive. The most prominent non-destructive method is based on X-ray diffraction (XRD) [3]. X-ray can also be used to measure effects of external loading on the density and change in structure in amorphous materials as was recently shown in [4] but for stress/strain quantification a reference state is necessary and that is very much ill-defined for a glassy state. In fact it is fairly impossible to define precisely and in a positive way a reference of a glassy state (although a fully randomnized system would be a good starting point). For crystalline solids the situation is of course completely different because the *stress free lattice parameters* (which is not always a trivial quantity in alloys) can be taken as the reference state and the lattice acts as a strain gauge. Various relaxation methods have been developed for both general and specific specimens [5]. Despite the large differences in geometry and experimental techniques, all methods share the concept of measurement of displacements caused by local cutting of stressed material [5]. The most commonly used are: Hole Drilling and Ring Coring [4], Deep Hole Drilling [6] and the slit milling method [7,8]. The advent of dual beam microscopy (FIB-FEG-SEM) has opened with the Focused Ion Beam (FIB) new avenues so as to make microscale cuts [9], holes [10], rings [11] or slits [12]. Together with Digital Image Correlation (DIC) the displacements at the surface can be obtained [13,14]. In DIC a reference image is recorded and digitized prior to deformation. Facets of an image (typically a square of about 10–20 pixels on each side) are then defined. Displacements can be found by tracking the movement of corresponding facet centers between the reference image and the image recorded after deformation. In our case after the first SEM image is recorded, a slit is made, the second SEM image is collected and the resulting displacement field is determined by DIC. In-plane displacements normal to the slit can be related to the residual stress in the same direction by an analytical expression that is based on isotropic linear elasticity [15,16]. This method has been applied to a wide range of specimen and stress geometries [12,16,17]. Nevertheless, an imaging stability test is recommended prior to the measurements in order to find the best imaging conditions [18,19].

In this paper we concentrate on samples of maraging steel with practical applications, which have been nitrided with different thicknesses in order to improve the surface mechanical and chemical properties [20]. The results from different approaches have been compared, i.e. the traditional 'averaging' and the so-called 'multiple fitting' method [21]. As a consequence, the lateral resolution of the method is enhanced at the expense of certain loss of

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http://dx.doi.org/10.1016/j.msea.2015.04.023
0921-5093/

signal-to-noise ratio. The results of residual stress have been compared with values obtained by XRD and their evaluation was carried out through the use of finite element simulation methods.

2. Experimental details

Four different maraging steel (Phytime) components (loops) were studied. Each specimen was annealed prior to nitriding, so that the residual stresses are expected to be arising from the nitriding process only. The components, strips with a thickness of about 200 μm, were gas nitrided in the range of 450–500 °C, all in the same batch process. During nitriding, molybdenum nitrides form, which are responsible for a volumetric strain. Subsequently orientation imaging microscopy (OIM) was used to determine the grain size and texture at different locations at the cross section of the sample (center is not nitrided and surface is nitrided). Each specimen was cut and prepared by mechanical grinding and

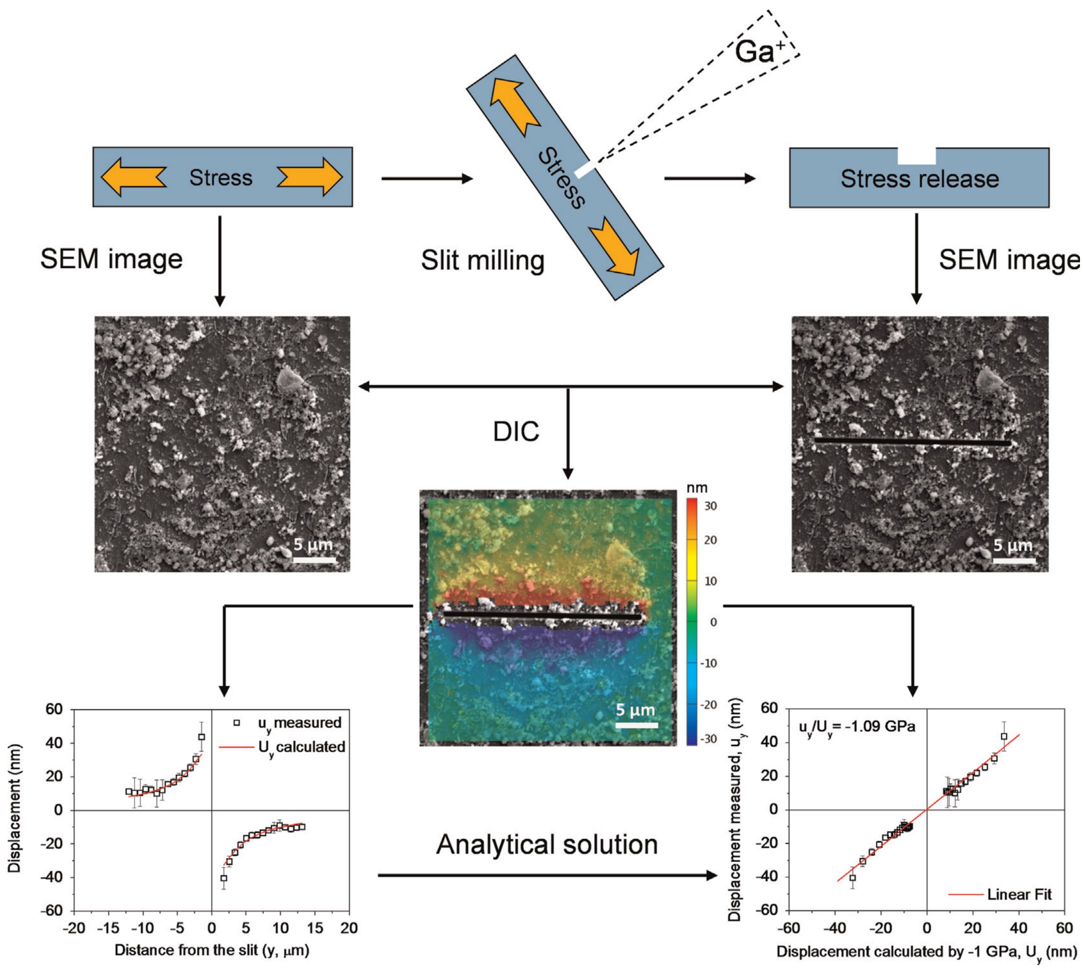

Fig. 1. Steps involved in the measurement of residual stress using a combination of SEM imaging, FIB machining and DIC image analysis.

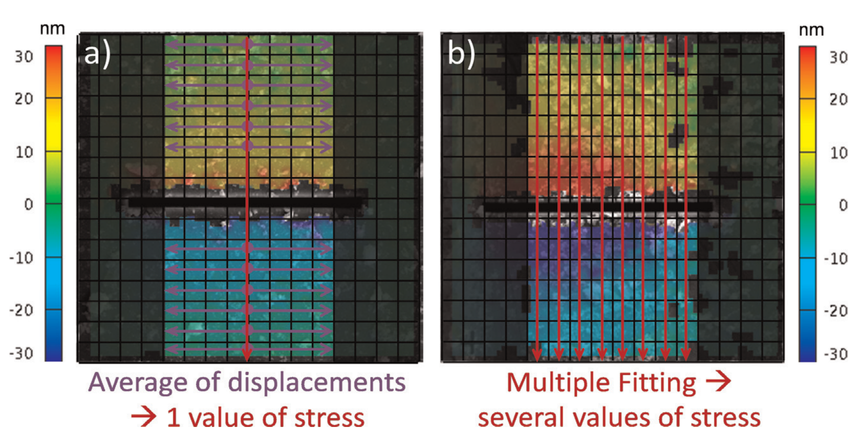

Fig. 2. Comparative scheme of the calculation methods of residual stress from DIC images. (a) Averaging: the measured displacements along the lines parallel to the slit are averaged prior fit to Eq. (1). (b) Multiple fitting, where displacements along the lines perpendicular to the slit are separately considered and analyzed using Eq. (1). The shaded areas represent the excluded regions near the ends of the slit.