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Separating macro- (Type I) and micro- (Type II+III) residual stresses by ring-core FIB-DIC milling and eigenstrain modelling of a plastically bent titanium alloy bar

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ABSTRACT

A novel approach to separating macroscopic (Type I) from microscopic (Type II + III) residual stress is presented, based on Focused Ion Beam – Digital Image Correlation (FIB-DIC) ring-core stress evaluation and eigenstrain modelling. This approach was applied to study the residual stresses for a titanium alloy bar following plastic four-point bending. It was found that electrochemical polishing is a surface preparation technique that is very well suited for FIB-DIC ring-core measurements, in the sense that it removes the influence of prior sample grinding and polishing, leads to a stress profile that satisfies force and moment equilibrium, and thus enables the evaluation of absolute values of total residual stress. The obtained relief strain profile across the bar width is asymmetric, highlighting the difference in the alloy's response to tension and compression. Total experimental residual stress values were calculated using (i) the assumption of material elastic isotropy, with an average Young's modulus, and (ii) under the assumption of elastic anisotropy, taking into account the crystallographic orientation of each investigated grain. Based on the measured relief strain values, the eigenstrain distribution in the bar was reconstructed and used to obtain the macroscopic (Type I) residual stress profile. The differences in the residual stress between the eigenstrain reconstruction values and the individual experimental results were ascribed to the local microscopic (Type II + III) residual stresses. This conclusion was substantiated by revealing the correlation between the residual stress values in individual grains in the elastic zone and their respective Young's moduli in the loading direction, as well as the correlation between the residual stress values in grains located in the plastic zone and their respective Schmid factors for basal slip.

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1. Introduction

Residual stresses play an important role in the deformation and failure behaviour of engineering materials and for this reason have been studied extensively for many decades. The creation of residual stresses is unavoidable when a material is subjected to inelastic processes, because these processes generally lead to strain incompatibilities. Based on specific length scales in materials, residual stresses can be classified into macroscopic (Type I) and microscopic (Type II and Type III) stresses [1]. Type II stresses are intergranular stresses that represent the deviation of grain average

values from the macroscopic long range average caused by grain interactions. Type III stresses are intragranular stresses that represent the deviation of local stress from the sum of Type I (macro-) and Type II (intergranular micro-) stresses, and that arise due to nanoscale defects, such as vacancies, dislocation structures e.g. pile-ups, etc. It is worth emphasising that the three types of residual stress are additive, which means that at a specific location in the material the total residual stress is equal to the sum of Type I, II and III stresses [1].

One of the main challenges in this field of research is to obtain accurate and reliable values of local residual stress that are

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absolute, i.e. are not computed based on quantities such as lattice strains that are known to be dependent e.g. on solute concentration variation [2]. Currently available stress evaluation techniques achieve this with varying degrees of success. X-ray diffraction techniques, which can be used to measure changes in interplanar lattice spacing, are a powerful tool for residual stress analysis. However, in general only Type I stresses can be evaluated due to the fact that the interaction volume is relatively large and thus comprises a large number of grains. This limitation has to some extent been overcome by the development of micro-focus synchrotron X-rays [3,4]. Nevertheless, diffraction techniques require the knowledge of stress-free lattice parameters as reference. The evaluation of local residual stress, without reference, has been made possible by the development of the Focused Ion Beam – Digital Image Correlation (FIB-DIC) ring-core milling technique [5]. This semi-destructive technique is based on the principle of strain relief after material removal. A FIB is used for incremental milling of a circular trench, which leads to the formation of a central island, or micropillar. By ensuring that the top surface of this micropillar carries a stable contrast pattern, it is possible to measure the strain relief by means of Digital Image Correlation (DIC) analysis of scanning electron microscopy (SEM) images, while the material is experiencing gradual strain relief due to progressive milling. Careful analysis by simulation and modelling confirmed that the micropillar is fully relieved of residual stresses when the milling depth becomes approximately equal to its diameter. The relief strain can be used to calculate the local residual stress that was present prior to milling [6]. The great advantage of this technique lies in the fact that it can be performed within any FIB-SEM machine, and allows absolute values of residual stress to be evaluated at the micro-to nano-scale resolution without access to a synchrotron source, in materials that may be single or poly-crystalline, heavily deformed, or amorphous. On the other hand, FIB-DIC ring-core milling is very surface-sensitive, as it measures residual stress in a surface layer less than a few microns in depth [4]. Although this means that it can be used to obtain residual stress values in for example thin coatings [6], it also means that it is crucial to ensure good surface preparation which doesn't induce additional residual stresses. The diameter of the micropillar is typically smaller than the grain size of the material, so that the determined stress values represent the sum of Type I, II and III residual stress. However, the separate contributions of each residual stress type are not known, leading to the appearance of a significant degree of scatter in the results [7]. It is clear, however, that total residual stress oscillations may in fact reflect the short range stress variation, i.e. the Type II + III contributions. The task of decomposing the experimentally determined total residual stress into Type I, II and III stresses is an interesting challenge, in that the specific components of residual stress are associated with different physical mechanisms, and therefore exert different and distinct influence on structural integrity, particularly under complex loading conditions, such as fatigue, creep, crack propagation, etc.

From the modelling point of view, residual stress fields within a solid can be conveniently described by use of the eigenstrain theory [8]. The term *eigenstrain* refers to the inelastic strain (misfit strain) stored in the material due to plasticity or other inelastic deformation mechanisms. The total strain in a material is given by the sum of elastic strain and inelastic strain (eigenstrain). If an eigenstrain distribution is present, for example after plastic deformation, it gives rise to a residual elastic strain distribution in order to maintain strain compatibility. This residual elastic strain distribution is directly linked via Hooke's law to the residual stress in the material, which must be in a state of self-equilibrium if no external load is present [9]. If the eigenstrain distribution can be modelled at the macro-scale, the resulting elastic strain distribution corresponds to

the macroscopic residual stress (Type I).

The purpose of this paper is to combine the FIB-DIC ring-core milling technique and eigenstrain modelling, with the objective of separating total residual stress into macroscopic (Type I) and microscopic (Type II + III) stresses. This can be achieved by using FIB-DIC ring-core data that gives the total (Type I + II + III) stress sum, as a basis for reconstructing the macroscopic eigenstrain distribution that describes only the macro-scale (Type I) residual stress. The difference between the residual stress reconstructed by the eigenstrain model, and the total local residual stress at each individual FIB-DIC ring-core point provides the estimate of Type II + III residual stress at this point.

In the present study, the well-known case of a plastically bent bar made of a titanium alloy is chosen to verify this approach. During bending of the bar, as the applied bending moment increases, two plastic deformation zones are formed: a tensile zone at the outside (convex) side of the bar, and a compressive zone at the inside (concave) side of the bar. Between the two zones the deformation remains purely elastic, with this elastic zone becoming progressively smaller as the applied bending moment further increases. After unloading, elastic springback leads to the formation of residual compressive stress at the outside (convex) side of the bar, and residual tensile stress at the inside (concave) side of the bar. This means that the relief strain, as determined by ring-core FIB-DIC, should be positive at the convex side of the bar, because the residually compressed material is allowed to expand, and negative at the concave side of the bar, because the material under residual tension is allowed to contract [9].

Electrochemical polishing is used to ensure that the surface of the titanium bar is free of additional residual stress that otherwise can be induced by mechanical grinding or polishing. As will be shown, this means of surface preparation is effective, in the sense that it results in the force and moment balance of the macroscopic residual stress profile across the bar width.

Finally, due to the high anisotropy of the hexagonal close-packed α phase of titanium [10,11], it is plausible that there should be a correlation between the orientation of a grain and the local Type II + III residual stress in that grain. This consideration is used to test whether the approach to separating Type I and Type II + III residual stresses is valid. For this purpose, a number of FIB-DIC ring-core measurements were performed inside individual grains along an axis parallel to the neutral axis. Because the macroscopic residual stress is constant along the axis, it is hypothesized that the variation between measurements can be related to microscopic residual stress and thus, in the first instance, to the grain orientation.

2. Material and methods

The dimensions of the Ti-6Al-4V bar and the width of the support and loading spans that were used for four-point bending are shown in Fig. 1. Prior to bending the bar was ground with grit size 2500 sandpaper. The maximum deflection at the centre of the bar was approximately 3 mm after unloading. Fig. 1 also shows the definition of the coordinate system and highlights the central region of interest. After bending, this area was electrochemically polished during 10 min with a current density of 2 kA/m² in an electrolyte containing 700 ml/l ethanol, 300 ml/l isopropanol, 60 g/l aluminium chloride and 250 g/l zinc chloride [12], which removed a surface layer of approximately 0.1 mm.

All scanning electron microscopy and FIB milling was performed using a Tescan LYRA3 FIB-SEM microscope. An Oxford Instruments NordlysNano detector was used for electron backscatter diffraction (EBSD) measurements. Ring-core FIB-DIC measurements were carried out inside single α grains with a pillar diameter of 5 μ m and

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