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A new methodology for the near-surface strain measurement on Pd–Ag–Sn alloy

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ABSTRACT

With the development of modern synchrotron sources, high-energy X-ray diffraction plays an important role in the residual stresses analysis of materials. This paper deals with the development of a new high-energy synchrotron X-ray diffraction (HESXRD) stress evaluation method for improving the near-surface strain measurement. For this purpose a new Monte Carlo simulation program has been developed to modelize any synchrotron radiation instrument. Futhermore conventional X-ray diffraction measurements have also been carried out after chemical etching, to define the surface and in-depth stresses of the sample, thus giving a reference to test the synchrotron radiation measurements. It has been shown that the reliability of this method is better than 5 μm. This method has been applied to a machined palladium alloy (Pd–Ag–Sn) plate substrate.

1. Introduction

In the 80s [1,2], alloys featuring high palladium content have been developed for biomedical applications. Furthermore, porcelain-fused-to-metal (PFM) technique was developed as strengthening mechanism for porcelain. For dental application, palladium content is usually in the range of 75–78 wt.% and the alloys are Pd-based ternary systems such as Pd–Ag–Sn or Pd–Cu–Ga or Pd–Co–Ga. Palladium has a high melting point (1829 K) and its thermal expansion is too low to be used with most commercial porcelains. So silver has been introduced to alloys for, primarily to raise the thermal expansion of palladium, lowering the melting range of palladium. Additionally silver is claimed to add fluidity to casting alloys. Tin is used as a strengthener and hardener in palladium PFM alloys lowering the melting range of palladium, too, and raising coefficient of thermal expansion. Moreover it contributes to bonding oxide formation. Gallium is used exclusively in palladium based PFM alloys as a powerful strengthener, and it lowers the melting range of palladium. Ruthenium and indium are used primarily as grain refiner [3].

High-energy synchrotron radiation is a widely used and extremely powerful tool allowing non-destructive measurements and a precise way to determine residual strain at the surface, in the bulk and at interface of different materials. It is particularly well suited for investigation in the near-surface regions of engineered components [4]. High intensity and low divergence of the beam allows small gauge volumes to be defined in order to study stress fields over a range of 100 μm [5].

Synchrotron radiation with high flux and high photon energy (20–300 keV) is penetrating typically two or three order of magnitude more than conventional Cu-K_α radiation. Using the crystal lattice as internal strain gauge, the high penetration of synchrotron radiation lets stress to be evaluated throughout the surface and in the bulk of the samples by means of Bragg diffraction.

Due to the short wavelengths using high-energy X-ray large elongation of the gauge volume results from low Bragg angles to be used. Usually gauge volume has a ratio of about 10:1 between the diagonals (Fig. 1), giving a spatial resolution typically ten times better in one direction than in the other one. Therefore it is necessary to develop methods to overcome this effect.

High resolution residual stress information can be obtained from a surface treated specimen in both, the normal and in-plane directions. A complete review has been presented by Edwards [6]. The strain in the crystal lattice is measured and the residual stress is calculated, assuming a linear elastic distortion of the crystal lattice. This is particularly important if the specimen contains a strong gradient of the stress near its surface [7].

In this paper, analysing method for evaluating the distribution of strain in the surface layer of a machined palladium alloy (Pd–Ag–Sn) plate substrate using a new Monte Carlo simulation program has been developed to modelize any synchrotron radiation instrument.

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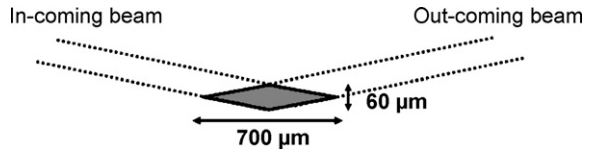

Fig. 1. Shape of gauge volume in high-energy synchrotron X-ray diffraction (HES-RXD) due to the small Bragg angle.

2. Method

2.1. Modelling of the synchrotron spectrometer and introduction to simulation software

To perform near-surface strain scanning using synchrotron radiation, the surface of the component must be accurately established. E.g. at the surface of a powerful absorbent, such as palladium alloy (Pd–Ag–Sn) materials, is required as well as a careful acquisition and precise processing of the experimental data. In particular, surface layers are very difficult to analyse due to their topography, to the X-rays absorption in the material and also because of geometrical divergences. To overcome these problems, a new approach based on Monte Carlo simulation program has been developed to model any synchrotron radiation diffractometer, been proposed in Refs. [8,9].

Monte Carlo simulation method allows:

- to predict the evolution of the theoretical diffracted intensity versus the adjusted depth;
- to simulate theoretical intensity curves, then being fitted to the experimental ones to define the precise position of the gauge volume as well as
- to acquire peaks, being processed together to achieve the in-depth stress profiles. A non-linear least squares optimisation method has been employed for that purpose.

In order to study a surface or an interface, a very accurate scanning has to be regularly performed in measurements across the studied area for reliable evaluation of the in-depth stress profile. This precise examination makes it possible to plot the evolution of the diffracted intensities versus the adjustment depth of the instrument (Fig. 2). The diffracted intensities grow when the synchrotron radiation probe enters inside the analysed material and then decrease quickly as a consequence of the strong absorption of the X-rays (Fig. 2(a)).

Traditional procedures would lead to considerable errors in the determination of the true measurement depth due to absorption dependence on the photon energy starting from the diffracted intensities in function of the adjustment depth of the instrument

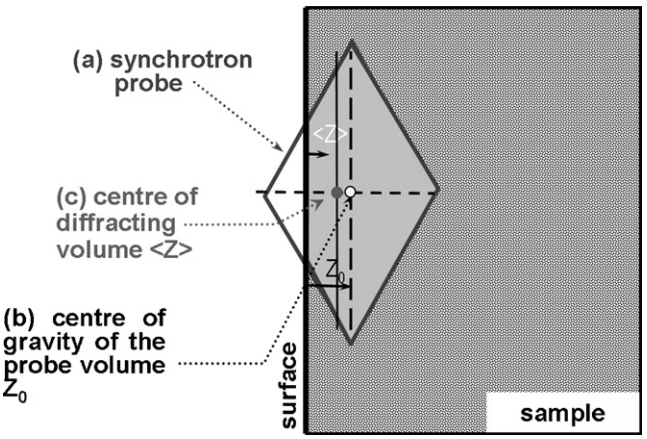

Fig. 3. Synchrotron probe partially immersed in the sample.

(Fig. 2(a)). In reality for standard adjustment procedures, the geometrical centre of the probe volume can match the studied surface (Fig. 2(b)) or interface at half maximum of the intensity graphs (Fig. 2(c), right, middle sketch) or when the probe volume is completely immerse (Fig. 2(d)) in the studied area of sample. This approach is not valid for high-density materials such as palladium or when the sample is partially immerse in the sample to be analysed. Fig. 3(a) shows the synchrotron probe partially immersed in the sample. The effect of X-rays absorption affects the position of the maximum in-depth intensity curves: infact the centre of gravity of probe volume (Fig. 3(b)) and centre of diffracting volume (Fig. 3(c)) are not located in the middle of synchrotron probe as reported in Fig. 2(c). Whatever the position of the X-ray gauge inside the material is, it is also usually assumed that the measurement is carried out in a position Z_0 located at the centre of the probe volume (Fig. 2(c)). However, this assumption is no longer valid in the case of palladium, which is a strong absorbent material.

In fact, for each position of the probe volume, it is necessary to define a true mean depth $\langle Z \rangle$ (Fig. 3(c) in the diffracting volume (immersed part of the gauge volume) which takes into account absorption of the X-rays by the material and changes of the local conditions of diffraction.

This new simulation method developed in some work of Carradó et al. [8,10] is based on the Monte Carlo method. It is possible to simulate some diffraction experiments carried out with X-rays and permits to predict the diffracted intensity, as well as the shape and position of the gauge volume in the matter under the conditions of the true measurement. A whole diffraction pattern can also be simulated. For that purpose, the different elements of the instrument are first to be defined:

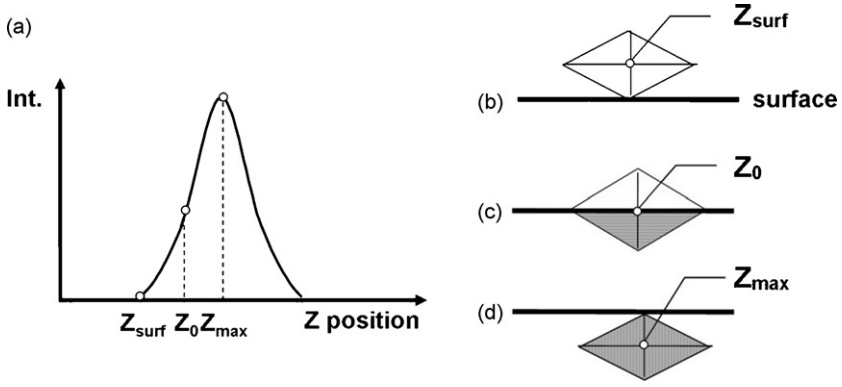

Fig. 2. Evolution of the diffracted intensities versus the adjustment depth of the instrument.