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Disturbance induced by surface preparation on instrumented indentation test

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ABSTRACT

Surface preparation, which may induce considerable sample disturbance, plays an important role in instrumented indentation test (IIT). In this study, the sample disturbance (mainly divided into residual stresses and plastic strain) induced by the surface preparation process of instrumented indentation test specimens were investigated with both experimental tests and numerical simulations. Grazing incidence X-ray diffractions (GIXRD) and uniaxial tensile tests were conducted for characterizing the residual stresses and high plastic strain in the top surface layers of a carefully mechanically polished indentation sample, which, in the present work, is made of commercially pure titanium. Instrumented indentation tests and the corresponding finite element simulations were performed as well. For comparison, a reference sample (carefully mechanically polished & electrolytically polished) which represents the raw material was prepared and tested. Results showed that a careful mechanical polishing procedure can effectively reduce the level of residual stresses induced by this process. However, the high plastic strain in the surface region imposed by the polishing process is significant. The induced plastic strain can affect a depth up to 5 μm, which is deeper than the maximum penetration depth h_{max} (3 μm) used for the instrumented indentation tests. In the near surface layer (in the range of depth about 350 nm), the plastic strain levels are fairly high. In the very top layer, the plastic strain was even estimated to reach more than 60%. The simultaneous use of indentation tests and numerical simulations showed that the existence of high plastic strain in the surface region will make the load vs depth (P–h) curve shift upwards, the contact hardness (H) increase and the contact stiffness (S) decrease.

1. Introduction

In the past few decades, instrumented indentation test (IIT), also known as depth-sensing indentation or nanoindentation test, has absorbed much attention and been broadly used in many fields for the characterization and quality control of materials properties. Being different from conventional hardness tests, IIT can reach a much higher level of control, sensitivity and data acquisition, which enabled numerous advances in materials and mechanics field, especially in studying the mechanical behavior of materials on small length scales (micro and even sub-micrometer level) [1]. IIT has been used in many researching domains, such as dislocation behavior in metal [2–4], Young's modulus and hardness characterization [5,6], measurement of residual stress and accumulated plastic strain induced by shot peening [7–10], creep behavior of metals and polymers [11,12], fracture behavior in ceramics [13], mechanical properties of thin films, coatings, bone and cells [14–17]. Its attractiveness stems largely from the fact that properties of mechanically meaningful material phases can be identified in situ by performing large grids of indentations on highly heterogeneous samples, with a proper choice of the indentation depth or force to avoid the so-called indentation size effect [18].

Recently, much attention has been paid to the characterization of residual stress and accumulated plastic strain by IIT. One big challenge which occurs during this process is the preparation of a test surface that minimizes both sample disturbance (such as unwanted extra residual stresses and plastic deformations) and surface roughness. Another challenge is to understand how this disturbance can affect the results of IIT. Although surface preparation is of great importance to IIT process, only a few researches focused on this part of study. Furthermore, nearly all of them concentrated on the surface roughness and topography of testing samples. Little attention was paid to the mechanical origin

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of sample disturbance induced by surface preparation.

In this paper, with both experimental tests and numerical methods, close attention was paid to the residual stresses and plastic strain induced by the surface preparation process (careful mechanical polishing process) into the top surface layer of the IIT specimen. Grazing incidence X-ray diffractions (GIXRD) [19] and uniaxial tensile tests were conducted for characterizing the values of residual stresses and plastic strain. Both IIT and finite element method (FEM) simulations were performed for investigating the influences of plastic strain induced by the polishing process.

2. Theoretical background

2.1. Geometrical definition of X-ray diffraction system

The key for describing a diffraction system consistently is to split the useful angles, a set of eight angles which are usually found in the literature, into three groups corresponding to three different aspects of physical acquisitions. The first group is the 'sample angles', denoted Φ, Ψ, η, which are used to probe the mechanical state of the material at a given depth. The second group is the 'goniometric angles', denoted φ, χ, ω, which are used to describe the rotation of the goniometer system. The third group is called the 'diffractometric angles', denoted θ and γ, which are used to define the direction of the diffracted beam [20], as shown in Fig. 1. Similarly, three orthonormal reference systems need to be defined as well. The first is 'the specimen (S) reference system', denoted (S_1, S_2, S_3), as shown in Fig. 2. S_3 is normal to the specimen surface and directed towards outside. S_1 and S_2 stay in the plane of the surface and are freely chosen to make the system to be direct. The second reference system is called 'the goniometer (G) reference system', denoted (G_1, G_2, G_3) and is represented in Fig. 2 as well. G_1 is the propagation direction of the incident X-ray beam. G2 is the axial direction of the goniometer. G_3 is chosen so that the system (G_1, G_2, G_3) is direct. The third reference system is called 'the laboratory (L) reference system', denoted (L_1, L_2, L_3), as shown in Fig. 1(a). L_3 is normal to the diffracting plane {hkl}. L_2 is the intersection line of the diffracting plane {hkl} and the diffraction plane defined by the incident and diffracted beams. L_3 is chosen so that the system (L_1, L_2, L_3) is direct. One thing needs to be noticed is that the acquisition modes using a zero- (punctual) or a one-dimensional (position-sensitive) detector can be described by taking $\gamma=0$, which means that everything takes place in the equatorial plane of the diffractometer [20].

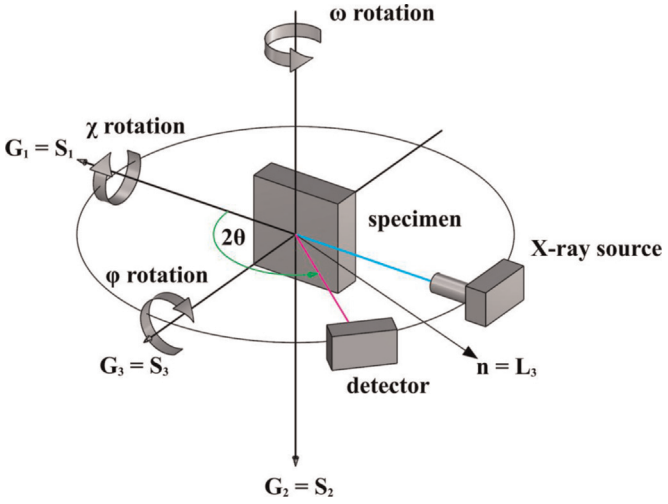

Fig. 2. Reference position ($\varphi=\chi=\omega=0$), for which the axes S_1 and G_1, S_2 and G_2, S_3 and G_3 are superposed pair wisely [20].

2.2. Standard $\sin^2\Psi$ method

The reference position for defining the origin of the angles is shown in Fig. 2. In order to determine the residual stresses in the material through X-ray diffraction, the specimen should have two kinds of rotations. One rotation is characterized by the tilt angle Ψ between S_3 and L_3. This kind of tilt can be accomplished by a rotation about S_1 (χ-mode) or S_2 (ω-mode) respectively, or by the combined rotation of S_1 and S_2 (combined tilt mode (mixed mode)). The other rotation is characterized by the azimuth angle Φ and is executed by rotating around the specimen normal S_3.

The residual or applied strain $\varepsilon_{\Phi\Psi}$ ({hkl},τ) can be determined from a change in the interplanar spacing $d_{\Phi\Psi}$ of the diffracting planes {hkl} as follows:

$$\varepsilon_{\Phi\Psi}(\{hkl\},\tau) = \ln\left(\frac{d_{\Phi\Psi}\{hkl\}}{d_0}\right) \approx \frac{d_{\Phi\Psi}\{hkl\} - d_0}{d_0} \quad (1)$$

where d_0 is the interplanar spacing of the unstrained material and θ_0 is the corresponding Bragg angle. τ is the average penetration depth. Strains calculated from X-ray diffraction data acquired at crystallographic scale $\varepsilon_{\Phi\Psi}$ ({hkl},τ) can be correlated to the components of macrostress tensor $\sigma_{ij}(\tau)$ in the sample volume by:

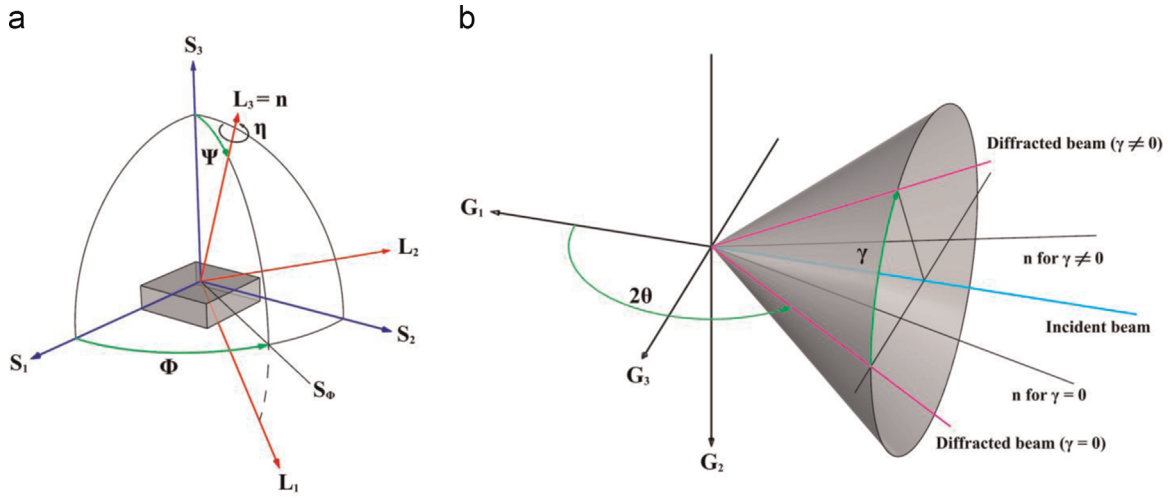

Fig. 1. (a) Definition of the specimen reference system (S_1, S_2, S_3), of the laboratory reference system (L_1, L_2, L_3) and of the sample angles Φ, Ψ, η; (b) Definition of the goniometer reference system (G_1, G_2, G_3) and of the diffractometric angles θ and γ [20].