Interrogation of the microstructure and residual stress of a nickel-base alloy subjected to surface severe plastic deformation

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Abstract

A low stacking-fault energy nickel-base, single-phase, face-centered-cubic (fcc) alloy has been subjected to surface severe plastic deformation (S^2PD) to introduce nano-grains and grain size gradients to the surface region of the alloy. The simultaneous microstructural and stress state changes induced by S^2PD have been investigated via the X-ray diffraction (XRD) analysis that includes evaluation of annealing and deformation twins, deformation faults, in-plane lattice parameters and elastic strains of the crystal lattice, macroscopic residual in-plane stresses, crystallite sizes, internal strains, dislocation densities, and crystallographic texture as a function of the depth measured from the processed surface. Microstructural changes have also been characterized using optical and electron microscopy in order to corroborate the findings from the XRD analysis. The results from the XRD analysis are in excellent agreement with those derived from the microscopy analysis. This is the first systematic and comprehensive study using XRD to quantify depth-profile changes in a wide range of microstructural features and stress states in a fcc material resulting from the S^2PD process.
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1. Introduction

Surface severe plastic deformation (S^2PD) processes have recently been developed to introduce nano-grains and grain size gradients into the surface region of bulk materials. There are several variants of such S^2PD-based techniques, including ultrasonic shot-peening (USSP) [1,2], high-energy shot-peening (HESP) [3], surface mechanical attrition treatment (SMAT) [4–6], surface nanocrystallization and hardening (SNH) [7–10], and particle impact processing (PIP) [11,12]. Several studies have demonstrated that S^2PD can improve mechanical properties of the material. An example of such improvements is a recent study on a nickel-base alloy [7], showing a 50%

improvement in the fatigue resistance after S^2PD processing. Improvements in the wear resistance of steels have also been demonstrated via S^2PD [13]. When the material being processed is in a plate form, a 35% improvement in the tensile yield strength of mild steels with minimum degradation in ductility and toughness has been obtained [3]. Recently, a 65–84% increase in the 0.2% offset yield strength of a nickel-base alloy has also been reported [14].

In spite of the aforementioned progress, the mechanisms responsible for the improved mechanical properties induced by S^2PD are far from being clear. Such a situation is due to the fact that the formation of nano-grains at the surface region induced by S^2PD is often accompanied by introduction of high density of dislocations, deformation-faulting and twinning, and in many cases macroscopic residual stresses [2,4–8,15,16]. These changes in densities of dislocations, faults and twins as well as the macroscopic

stress state together with the refined grain size collectively contribute to the improvement in the mechanical properties of the material. However, there has not been any study so far being devoted to quantifying these simultaneous changes in the microstructure and stress state in any single material. Also, there is a lack of efforts to establish experimental techniques with simple, well-defined procedures that can offer comprehensive depth-profile evaluation of the simultaneous microstructural and stress state changes induced by S^2PD.

X-ray diffractometry is specially suited to investigate the simultaneous microstructural and stress state changes induced by S^2PD. In perfect crystal and instrumental conditions XRD peaks are delta-like functions with positions in the diffraction pattern dictated by Bragg's law according to a well-defined unit cell and with intensities given by the crystal structure [17,18]. Apart from asymmetric peak-broadening attributed to instrumental causes, deviations from the ideal shape of the XRD peaks appear when the crystal lattice becomes finite and/or imperfect [19–22]. Due to the wavelength of the characteristic X-ray radiation, the crystal lattice can be considered finite if the crystallites are about a micrometer or lower in size [22], and thus peak-broadening is expected from ultra-fine-grained and nano-grained materials. In addition, any disruption of the regular crystal lattice causes an inhomogeneous state of strain that also results in peak-broadening, and sometimes in peak-shifting and asymmetry [19–22]. Among various crystal lattice imperfections, dislocations, faults, twins, solutes, vacancies, precipitates, and inclusions are the most frequent ones inducing peak-broadening, shifting or asymmetry. Furthermore, any modification of the unit cell dimensions leads to irremediably peak-shifting, making it possible to interrogate formation of solid solutions and introduction of macroscopic residual stresses [19–22]. Finally, systematic deviation from the theoretical intensity ratio emerges when crystallites exhibit preferred crystallographic orientations (i.e. texture) [19–21]. It is thus clear that XRD patterns contain substantial microstructural information and have the potential to be an effective tool for evaluating the simultaneous microstructural and stress state changes. Additionally, the information obtained from XRD analysis is very representative in statistical terms because the zone illuminated with the X-ray incident beam is very large (more than 10^8 times the grain size for a typical micrometer-sized material).

With this in mind, this study is undertaken with the objective to analyze the simultaneous microstructural and stress state changes induced by S^2PD in a fcc material using XRD patterns. The analysis includes evaluation of annealing twins, deformation faults and twins, in-plane lattice parameters and elastic strains of the crystal lattice, macroscopic residual in-plane stresses, crystallite sizes, internal strains, dislocation densities, and crystallographic texture as a function of the position measured from the processed surface. The contributions from the factors listed above to the XRD patterns have been separated and used to quantify the microstructural and stress state changes. The findings obtained from XRD analysis have been corroborated using optical and electron microscopy. This is the first systematic and comprehensive study using XRD to quantify depth-profile changes in key microstructural features and stress states induced by S^2PD in a fcc material.

2. Theoretical aspects of the XRD analysis

To facilitate the XRD analysis carried out in this study, the key theoretical aspects of the XRD interpretation applied to fcc metals and alloys are summarized in this section. The description of body-centered-cubic (bcc) and hexagonal-close-packed (hcp) cases is excluded from presentation because it exceeds the scope of the present study. Furthermore, the summary does not aspire to be complete, but only contains those aspects that have been utilized in this study and can be used on the base of no more than two XRD peaks collected using a conventional laboratory diffractometer in a single run.

It is well established that twins broaden the XRD peaks of fcc materials asymmetrically [23,24]. Thus, in principle it is possible to calculate twin probabilities (γ) from either the peak-broadening or asymmetry. However, the peak-broadening approach has met with limited popularity because, apart from twins, the peak-broadening may also be induced by faults, grain size refinement, dislocation polygonization, crystal lattice microstrains, and instrumental causes [20,21]. This makes it very difficult to evaluate γ from the peak-broadening, especially for materials processed via S^2PD. This problem becomes more tractable in the peak asymmetry approach, since the contribution from twins can be isolated more easily. In this context, γ can be readily evaluated from the lower limit of the Fourier sine coefficients (B_n with n being the ordinal of the Fourier coefficients) of the XRD peaks after excluding the instrumental broadening, that is, through the following relation [25]:

$$\gamma = C_t\sqrt{3}(B_n)_{n\to 0} \tag{1}$$

where the constant C_t adopts the value 4/3, −1, 2, 0, −4/3, and 1 for the 111, 200, 220, 311, 222, and 400 peaks, respectively. However, to avoid the calculation of B_n in practice, γ is simply estimated directly from the residual asymmetry of the 200 peak through the following relation [25]:

$$\gamma = \frac{\sqrt{3}\pi 2\theta^r (I^{2\theta_l} - I^{2\theta_r})}{2\beta}\left(1 + \left[\frac{\lambda}{4\pi D_{\text{eff},200}(\sin\theta^r - \sin\theta_{B,200})}\right]^2\right) \tag{2}$$

where λ is the radiation wavelength, β and $\theta_{B,200}$ are the integrated intensity and the Bragg angle of the 200 peak, respectively, $D_{\text{eff},200}$ is the effective crystallite size along the 200 crystallographic direction, $2\theta^r$ is any diffraction angle on the high-angle tail of the 200 peak, $I^{2\theta_r}$ is the intensity at $2\theta^r$, and $I^{2\theta_l}$ is the intensity at the equidistant position of $2\theta_{B,200}$ with respect to $2\theta^r$ at the low-angle tail of the 200 peak.