Note

Effect of the Surface Layer Strained by Mechanical Grinding on X-ray Diffraction Analysis

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X-ray diffraction is a powerful tool for characterizing the microstructure of steels. However, the strained surface by mechanical grinding could cause some errors in X-ray diffraction analysis. In this work, the strained layer was found to affect peak intensity, peak positions and enlarge the full width at half maximum. A quantitative relation between the depth of damaged layer and particle size of sander papers was established.

KEY WORDS: X-ray diffraction; mechanical grinding; lattice parameter; line broadening; steels.

1. Introduction

The X-ray diffraction (XRD) analysis is a powerful tool for the investigation of fine structures in metals which includes crystal structure, phase constituent, lattice parameter, dislocations density and residual stress, *et al.* The XRD is considered to be economic, effective, precise microanalysis in macro areas and environmentally friendly.^{1,2)} However, such advantages could only be truly achieved with precise diffraction information which directly linked to the quality of sample preparation. For iron and steel specimens, the surface is usually ground to remove oxides, elemental segregation, heterogeneous strain and extraneous matters nearby the surface layer.²⁾ **Table 1** shows the typical sander papers (P-scale) and the corresponding particle size, which cover typical papers for final step grinding in the fields of

Table 1. Typical sander papers (ISO, P-scale) and the corresponding particle size.

P-scale number	P 120	P 240	P 600	P 1000	P 2000
Particle size (μm)	125	58.5	25.8 $\pm$ 1	18.3 $\pm$ 1	10.3 $\pm$ 0.8

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academic and industry. Accordingly, a remarkable strained layer (tens or hundreds of microns) might be introduced during mechanical grinding.

In crystallography analysis by XRD, the common copper target owns a wavelength ($K\alpha$) of 0.15418 nm (molybdenum target: 0.0711 nm). The lower scale of wavelength than lattice parameter of steels (0.2–0.4 nm) leads to its atomic scale resolution. Because steels can strongly absorb X-rays, the intensity of the incident beam is reduced almost to zero in a very short distance below the surface. Cullity and Stock¹⁾ showed that 95% of the information refers to a depth of approximately 25 μm , and almost 50% of that originates only in the first depth of 5 μm . In general, qualitative illustrations on removing grinded surface by mechanical or/and electrolytic polish were given.^{3–6)} But less quantitative results were shown.⁴⁾ In the present work, main attentions will be firstly paid on the strained layer on XRD analyses in ferritic steels. The depth of damaged layer is to be quantitatively decided after grinding by various sander papers. This work will give significant reference for researchers when they implement XRD experiments.

2. Experimental Procedure

The interstitial free (IF) steel sheet (thickness: 1 mm) with 90% cold rolling was firstly adopted in this work.⁷⁾ In order to eliminate the dislocations and small crystalline size introduced by cold rolling, the samples (size: 15^l×15^w×1^t mm) were fully recrystallized at 750°C (Argon atmosphere), and then slowly cooled ($\sim$ 1.5°C/min) to room temperature. The yield stress (σ_y) was about 73 MPa with average grain size of 39 μm . For comparing the effect of strained surface by grinding on X-ray diffraction analysis, the samples were ground by P120, P240, P1000 and P2000 sander papers, respectively. The pressure force was about 9 N and the last time of grinding was about 90 s. Then, the samples were electrolytic polished in mixed acid ($\text{H}_3\text{PO}_4\text{:CrO}_3=2\text{:}1$) with constant current of 0.9 A (Voltage: $\sim$ 5 V) to remove the strained layers gradually. The present authors confirmed that this electrolytic polishing process could obtain constant thickness reduction rate and clear surface. The reference sample (Ref.) was directly electrolytic polished (without grinding) for about 120 min before XRD. The XRD measurements were carried out on an X-ray diffractometer (RINT2100, Rigaku Co. Ltd.) equipped with a Cu- $K\alpha$ radiation source (40 kV, 40 mA).⁶⁾ The diffraction information was recorded for 2θ range of 40°–140° with step size of 0.02°. Correspondingly, the thickness variation and surface condition were checked by micrometer, optical microstructure (OM) and electron backscatter diffraction (EBSD). Otherwise, to explore the influence from yield strength (or hardness), a well annealed Fe-3 mass% (3% Si) steel sheet ($\sigma_y \approx$ 350 MPa) was also performed with the same procedure.

3. Experimental Results and Discussions

Figure 1 shows the microstructures of IF steel after grinding by P1000 sander paper under OM (normal direction, ND) and EBSD (transverse direction, TD). Massive scratches are shown just after grinding. By combining the

Fig. 1. Microstructures of the ND surface observed by optical microscopy (OM) and the TD surface observed by electron back scattering diffraction (EBSD) for IF steel, which was grinded by P1000 sander paper. (Online version in color.)

image quality (IQ) map, inverse pole figure (IPF) map and Kernel average misorientation (KAM) map from EBSD, two strained layers are observed apparently which contains a severely strained layer (red frame) and an indirectly strained layer (blue frame). The thickness of such strained layers is estimated to be about $5\ \mu\text{m}$ and $4.5\ \mu\text{m}$, respectively. Because of the limited resolution of EBSD (tens of nanometers) and the surface etching of EBSD sample by colloidal silica solution could remove some less strained layers, the practical thickness should be larger.

Figure 2 shows the XRD patterns of IF steel after grinding (P2000) and various thickness reductions by electrolytic polishing. The results show clear effect of electrolytic polishing on the intensity for various diffraction planes. For instance, the peak intensity of 200, 211, and 222 reflections gradually increase with the processing of electrolytic polishing (increased t). The intensity variations correspond to the related ‘volume’ of each diffraction plane.^{1,8)} Such influences may affect the quantitative measurement result based on intensity analyses, such as phase volume fraction and texture analysis.¹⁾ The peak positions for various diffraction planes, which could be generally responded by the integral increase of lattice parameter (a), are found to shift slightly by strained surface because of heterogeneous plastic deformation distribution.¹⁾ According to the Nelson-Riley refine-

Fig. 2. X-ray diffraction patterns of IF steel polished by P2000 sander papers, showing the effect of electrolytic polishing. Thickness reduction (t) was measured by micrometer. The lattice parameter (a) was decided by the Nelson-Reilly refinement [1]. The magnified figure of 110 reflection shows the variations of peak position and peak intensity under various polished conditions. (Online version in color.)

ment, the peak center (2θ) of XRD reflections plays the key role in determining the lattice parameter.¹⁾ Therefore, the heterogeneous strained layers by grinding would lead to shift of peak positions and then result in increased values of a in Fig. 2 determining by XRD reflections. The peak positions are the keys for precise residual stress measurement.

XRD line broadening, which is always represented by full width at half maximum (FWHM) or integral breadth (β), mainly includes instrumental effect, crystallite size and dislocations in cold worked steels.^{1,4,9)} For the same X-ray diffractometer, instrumental effect is constant at critical diffraction angle. Therefore, the surface strain introduced by mechanical grinding could increase FWHM levels by enhancing both crystallite size and dislocations effects. **Figure 3** gives the variations of FWHM of 222 reflection with thickness reduction in IF steel and 3% Si steel. The immediate ground samples show remarkable increase in FWHM in both steels. The FWHM values of IF steel increase with particle size of papers (Table 1). After electrolytic polishing, the FWHM values initially decrease rapidly with thickness reduction, and then reduce gently. The harder 3% Si steel shows slightly higher FWHM than IF steel. By carefully comparing to the peak information of Ref. sample, the thickness of strained layer (t) could be estimated, which are approximately $20.5\ \mu\text{m}$ (P2000), $28.5\ \mu\text{m}$ (P1000), $40\ \mu\text{m}$ (P240) and $51\ \mu\text{m}$ (P120) for IF steel, about $17\ \mu\text{m}$ (P2000) and $43\ \mu\text{m}$ (P120) for 3% Si steel.

On the basis of particle size (d) in Table 1 and present evaluation of full strain layers thickness (t) from Fig. 3, a quantitative formula could be obtained:

$$t\ [\mu\text{m}] = (d/4 + 18.5) \pm 7.5 \dots\dots\dots (1)$$

Accordingly, an integrated sketch (**Fig. 4**) gives the full strained layer thickness after mechanical grinding by various sander papers. In practical processes, the depth of strained layer should also relate to the coupled effects from the

Fig. 3. Variations of full width at half maximum (FWHM) for 222 reflection in IF and 3% Si steels as a function of thickness reduction by electrolytic polishing. The line profiles of as-grinded and Ref. specimens are also shown in the figure (the slight shift of 2θ was normalized to that of Ref.). (Online version in color.)

hardness of samples, duration of grinding, holding pressure and possibly initial grinding paper. A general assessment on residual strained layer induced by mechanical grinding and polishing are needed by referencing Fig. 4 simultaneously. For softer metals, such as copper⁴⁾ and aluminum, the depth might be enlarged. On the contrary, the depth should be reduced for harder samples (such as 3%Si steel). As proposed in,⁵⁾ a combination of mechanical polish and full electrolytic polish would be the preferred procedure after grinding. Careful electrolytic polish could also be feasible individually. Chemical etching by special solutions would also work as suggested in.⁴⁾ Generally, electrolytic polishing or chemical etching is necessary for the last operation steps.

4. Conclusions

The strained surface by mechanical grinding, apparently

Fig. 4. Relation between the particle size (d) of sand paper and the thickness (t) of strained surface layer based on Eq. (1) in this work. The grey region represents the error bars area. The experimental results of IF steel and 3% Si steel specimens are also presented.

shown by EBSD, was found to influence the diffraction intensity, peak positions and FWHM levels in XRD analysis. The strained layer thickness (t) was found to dependent on the particle size (d) of various sander papers, and the quantitative relation was established: $t [\mu\text{m}] = (d / 4 + 18.5) \pm 7.5$.

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