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Effect of Processing on Nix-Gao Bilinear Indentation Results Obtained for High Purity Iron

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ABSTRACT

Instrumented indentation of a high purity Fe surface with unresolved surface deformation due to mechanical polishing is compared to the same grain surface annealed at increasing time and temperature. The differences in indentation size effect behavior with annealing are correlated with hardness and electron backscatter diffraction measurements as independent measures of surface layer deformation. It is found that the Nix Gao plot evolves from non-linear (bilinear) towards the predicted linear relationship as the surface deformation is removed. The experimental observations are rationalized by inclusion of a depth dependent, polishing induced forest dislocation density within the Nix-Gao model.

INTRODUCTION

The indentation size effect has been studied for variety of materials reporting significant variation in hardness as a function of depth [1-3]. To explain mechanism responsible for the indentation size effect, various strain gradient plasticity [4-5] and mechanistic models [6-8] have been proposed. Nix and Gao [6] proposed, arguably the most widely accepted mechanistic model, based on geometrically necessary dislocations (GND) required for the indentation plastic shape change and statistically stored dislocations (SSD) due to the indentation characteristic strain [6]. To explain decreasing hardness with increasing depth, this model proposes that GND have an increase in spacing as the indentation depth increases. The Nix Gao model for indentation of initially defect free material relates the hardness to the indentation induced dislocation density as

$$H = H_0 \sqrt{1 + \frac{h^*}{h}} \quad (1)$$

$$H_0 = 3\sqrt{3}\,\alpha G b \sqrt{\rho_{SSD}} \quad , h^* = \frac{3\,tan^2\theta}{2b\rho_s}, \rho_{GND} = \frac{3\,tan^2\theta}{2fbh} \text{ , where}$$

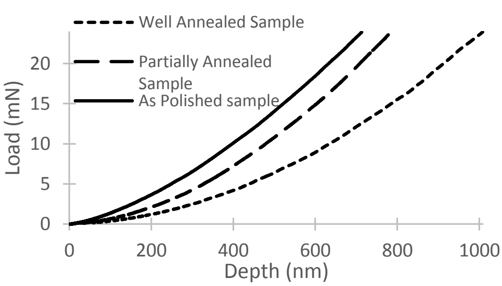
α- constant=0.5, G- Shear modulus, b- Burger's vector, ρ_{GND}- GND density, ρ_{SSD}- SSD density, θ- angle between indenter and un-deformed surface, h^*- Characteristic depth, f - material dependent factor for volume correction [9]. According to the simplest interpretation of the Nix-Gao indentation size effect theory, the slope of hardness squared (H^2) plotted against inverse depth 1/h should be determined by the geometry of the indenter and the volume of the plastic zone under the indenter. For a Berkovich indenter, this slope is predicted approximately constant for hardness at indentation depths that exceed depths effected by tip defect contributions and contributions due to energy barriers for dislocation nucleation. This type of linear behavior has been observed in a number carefully prepared materials such as Cu [2] and Ag [3]. However, there have been multiple reports of so called 'bilinear' behavior, reviewed in [8], in which the Nix-Gao plots show measurable deviation from linearity. That is, the slopes of H^2 vs 1/h at small indentation depth are observed to be significantly different (less) than the slopes at large indentation depths. This paper examines the effect of surface deformation induced by polishing can have on Nix-Gao non-linearity (bilinear behavior), noting that mechanical polishing introduces 'forest dislocations' (FD), which will be in addition to the SSD and GND's required for accommodation of indentation plasticity. This approach leads to the rather simple, but previously unexplored, concept that the spatial variation of FD (due to mechanical polishing) with depth can contribute to bilinear Nix-Gao behavior.

EXPERIMENTAL PROCEDURE

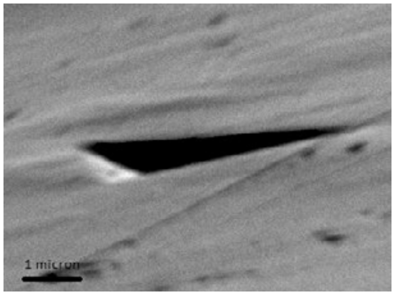
A high purity (99.999+) α- Iron specimen 10 mm diameter by 4 mm thickness was cut from as-drawn rod purchased from Goodfellow USA. The specimen was annealed and then mechanically polished followed by multiple annealing steps in reducing atmosphere with continuous flow of high purity hydrogen gas through the furnace chamber and then furnace cooled with the last stage in high purity Ar to reverse any hydrogen adsorption. After each processing step, 20 indentations were carried out within the same millimeter diameter grain, orientation~<110>. The nanoindentation experiments were performed using a diamond Berkovich indenter tip and the Nano Flip (Nanomechanics Inc, Oak Ridge, TN). The loading rate, $\dot{P}$, was controlled such that $\dot{P}/P$ was held constant at 0.2 1/s. The maximum load was 24 mN. Justified by Fe's high ratio of elastic modulus to yield strength, the contact area was taken to be in the original plane of the surface (Fig 1b). This definition allows the hardness to be calculated as a continuous function of depth without utilizing dynamic testing methods that can obfuscate experimental data at small indentation depths. Electron backscatter diffraction (EBSD) experiments were performed to support the hypotheses derived from the nano-indentation experiments. These EBSD results were obtained using an FEI XL40 ESEM interfaced with the Oxford EBSD system.

RESULTS AND DISCUSSION

It is observed from the load-depth (P-h) curves in Fig. 1 and averaged hardness vs depth (H-h) profile plots in figure 2 that the hardness for the as polished sample is significantly greater than that for the partially or fully annealed samples. This is expected on the basis of FD strengthening (work hardening) of the surface due to mechanical polishing [10]. The degree of the indentation size effect (ISE) can be determined by noting the decrease in hardness with



(a)



(b)

Figure 1. (a) Representative P-h curves, (b) SEM indent image tilted to ~80 degrees.

depth. As seen in figure 2, the ISE is most obvious for the un-annealed, mechanically polished specimen.

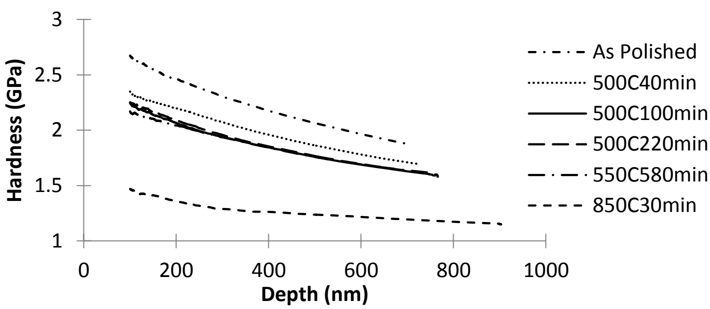


Figure 2: Plot between hardness and depth (averaged from 20 H vs h curves at each condition) for all the processing conditions, indicating presence of indentation size effect (ISE).

The data in figure 2 is converted to Nix-Gao curves (H^2 vs 1/h) in figure 3. According to the Nix Gao model, H^2 vs 1/h plot should be linear, with the slope corresponding to the decrease in GND density with depth. Only the 850°C anneal sample approaches linear behavior. A qualitative inspection of figure 3 shows that the nonlinear (bilinear) behavior of the Nix-Gao plots is reduced when the sample is annealed and the hardness is reduced. The reduction in hardness due to annealing is expected as the FD density is decreased during the annealing process. Adding the hardening effect of the FD density, ρ_{FD}, to the indentation GND and SSD density in equation 1 leads to

$$H^2 = {H_o}^2\left(1 + \frac{\rho_{FD}}{\rho_{SSD}} + \frac{h^*}{h}\right) \qquad (2)$$

A simple model for the reduction in FD density, $\rho_{FD,}$ during the annealing recovery process has been proposed by Nes [11], written in the form which exposes the diffusion coefficient as

$$\sqrt{\rho_{FD}(t)} = \sqrt{\rho_{FD(0)}}(-[1/A][\ln\{[1/L^2][L^2\exp(-A) + Dt\}]) \tag{3}$$

Where D is the self diffusion coefficient, t is annealing time, Q is the activation energy for self-diffusion, $L^2 = bA/\sqrt{\rho_{FD(0)}}$ and $A{\sim}Gb^3/kT$. Substituting equation 3 into equation 2 and differentiating with respect to 1/h suggests the behavior of the Nix Gao slope for a 1/h dependent polishing induced forest dislocation distribution as

$$\frac{\partial}{\partial(\frac{1}{h})}H^2 = \frac{{H_o}^2}{\rho_{SSD}}\left[\frac{\partial}{\partial\left(\frac{1}{h}\right)}\rho_{FD(0)}\right]\left[\frac{\rho_{FD}(t)}{\rho_{FD}(0)}\right] + [\frac{\partial}{\partial(\frac{1}{h})}\frac{\rho_{FD(t)}}{\rho_{FD}(0)}][\rho_{FD}(0)] + {H_o}^2 h^* \tag{4}$$

Equation 4 considered together with equation 3 indicates the possibility that the Nix Gao slope obtained by indentation within a deformed, annealed surface layer can be a function of the product (**Dt**). The curves in figure 3 are fitted to 3rd order polynomials (R^2>0.99) allowing the Nix Gao slope at 1/(150 nm) and 1/(500 nm) to be determined via derivative. A plot of the Hardness vs $\log(Dt + L^2)$ and the (Nix-Gao slope) against $\log(Dt + L^2)$ at 150 nm indentation depth and 500 nm indentation depth is shown in figure 4. The value of L^2 for the 150 nm indentation depth is 10^{-19} m^2 while the value of L^2 for the 500 nm indentation depth is 10^{-18} m^2. The result in figure 4a reproduces the logarithmic decay in hardness value for increasing Dt often observed for the dislocation recovery process on annealing, as reviewed in [11]

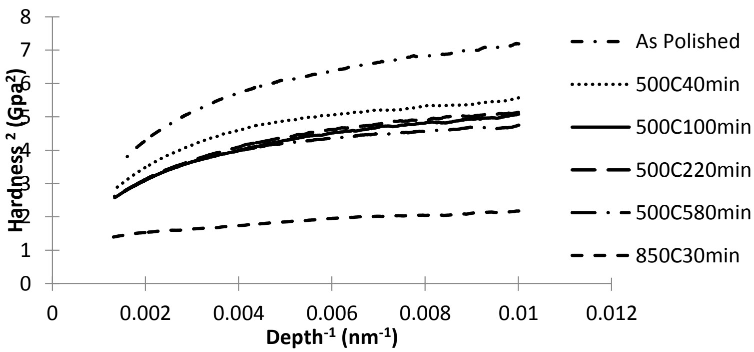
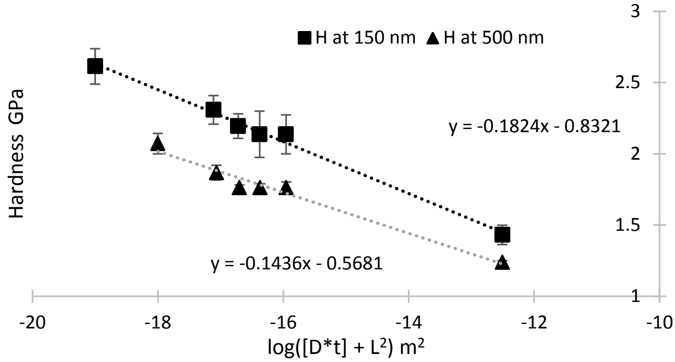
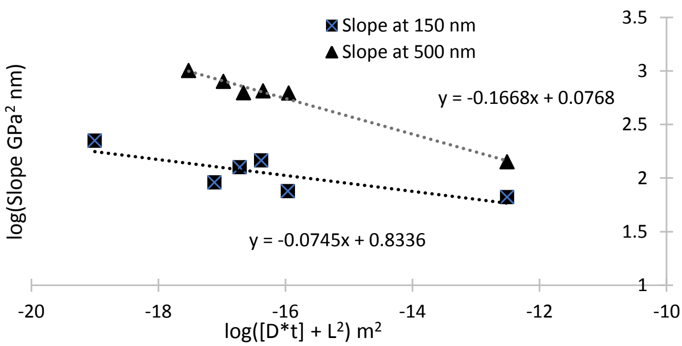


Figure 3: Nix-Gao plots for the various processing conditions

Figure 4b shows the Nix Gao slopes at the 150 nm and 500 nm indentation depths approach one another as annealing Dt increases and provides a quantitative measure of how the bilinear behavior as the polishing surface damage is removed with increasing Dt. To the authors' knowledge, figure 4 is the first quantitative correlation between Nix-Gao bilinear behavior and the cold worked condition via mechanical polishing of the sample surface layer. However, it is noted that previous work has observed the Nix Gao plot for mechanically polished Ni has a slope in the 400-800 nm indentation depth range much larger than the Nix Gao plot for mechanically polished Ni that has been electro-polished or annealed [12]. It is also noted that indentation studies on 10% pre-strained Cu compared with well-annealed Cu [13] do not report the effect observed here and in [12], perhaps because of a more homogeneous distribution of forest dislocations in[13].



(a)



(b)

Figure 4: (a) The hardness-log diffusion length plot of hardness measured at 150nm and 500nm showing a typical logarithmic decay in flow stress with heat treat recovery time. (b)The reduction in bilinear behavior with annealing of surface polishing deformation is indicated as the 150 nm and 500 nm slopes approach one another.

To extend this analysis further, we note the Nix-Gao theory can be used to rationalize the result in figure 4 with very little modification by invoking a depth dependence in the forest dislocation density induced by mechanical polishing and subsequently altered by annealing. Assuming equation 2 is 'correct' in the sense that the Nix Gao theory in equation (1) requires no modification other than the addition of the depth dependent FD density, the depth dependence of the FD density may be calculated, where Ho = 1.15 GPa and h* = 70.58 nm, are determined from the linear behavior of the 850^{o}C annealed material. The result is plotted in figure 5.

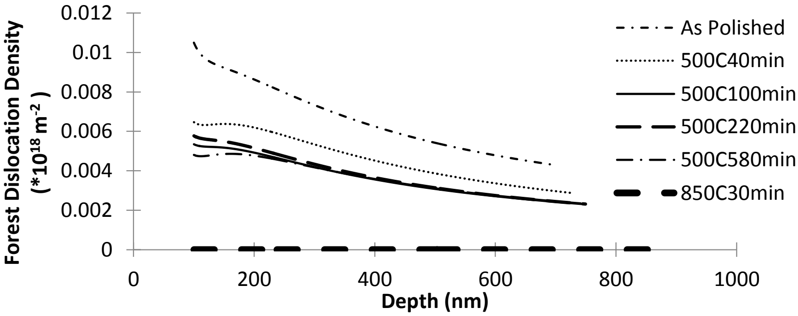


Figure 5: Plot of calculated forest dislocation density as a function of depth. The density is calculated assuming linear Nix Gao ISE for the various processing conditions.

The concept that the observed large changes in hardness and Nix Gao slope at low indentation depth (Fig.4) are correlated with annealing induced changes in ρ_{FD} (Fig. 5) is qualitatively tested using EBSD Kernel Average Misorientation (KAM) and Pattern Quality (PQ) measurements of surface layer deformation [14-15]. Although PQ is generally considered a qualitative measure of surface deformation, we do consider an adaptation of the pattern quality metric of [15], where the parameter proportional to the stored deformation energy (S_{FD}) introduced by mechanical polishing is given as

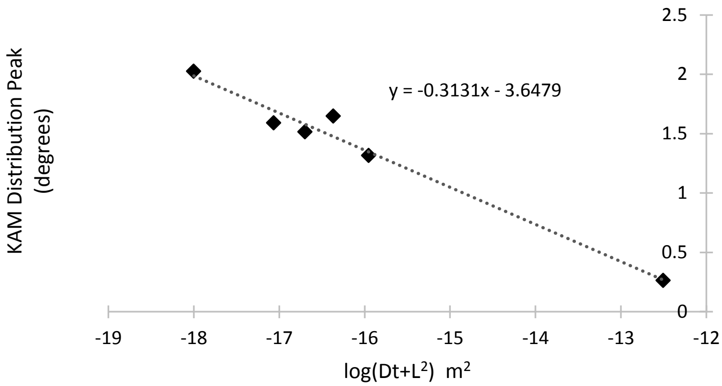
$$S_{FD} = \left[1 - \frac{Q_{FD(Ave)} - Q_{FD(min)}}{Q_{FD(max)} - Q_{FD(\min)}}\right] \quad (5)$$

The Q in equation 5 are the average, minimum and maximum pattern quality values for the distribution in the deformed (via polishing) or deformed (via polishing) and annealed samples.

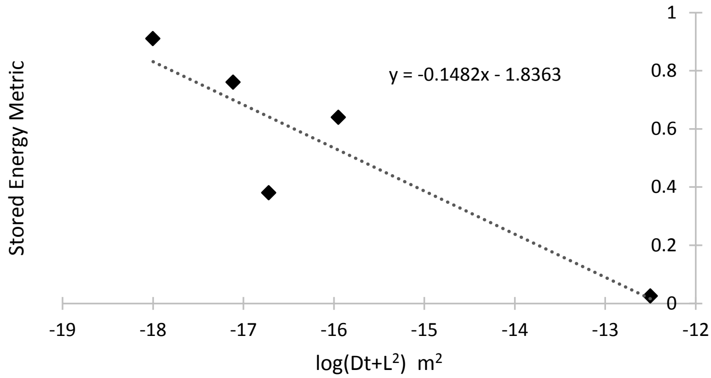
The uncorrelated average KAM value [14] and a pattern quality metric [15] are plotted against the logarithm of the annealing time figure 6. Both representations in figure 6 support the argument of the paper, with the caveat that these surface deformation measurements by EBSD are from 20-50 nm in depth while the indenter tip is sensing material behavior well below 150 nm.

CONCLUSIONS

The effect of processing conditions on indentation behavior, particularly on indentation size effect, were studied for high purity α-Fe. The transition from bilinear Nix-Gao behavior exhibited by cold worked (by mechanical polishing) and partially annealed samples to the predicted characteristic linear behavior for a well annealed surface has been correlated to the annealing diffusion length. Moreover, it is possible to recover the linear Nix Gao model from bilinear data if the forest dislocation density due to surface polishing is considered a function of indentation depth.



(a)



(b)

Figure 6: (a)KAM and (b) PQ metrics plotted against the logarithm of the anneal time.

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