Piecewise Model with Two Overlapped Stages for Structure Formation and Hardening upon High-Pressure Torsion

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The evolution of micro/nanostructure in metals subjected to high-pressure torsion (HPT) still need to be explained theoretically although experimental datasets are growing persistently. A major problem associated with the understanding of HPT is the synergetic effect of several competing processes that alter the material structure. In this study, we propose a piecewise model to analyze material hardness and true strain data during the HPT procedure. The model is built on two postulates: (a) the hardness vs true strain dependence is a sum of two piecewise power-law functions (each of these functions describes an unique micro/nanostructural stage of the deformation) and (b) each piecewise function has free-fitting strain breakpoints, which limit the strain range in which one mechanism predominantly determines the micro/nanostructure. The model was applied to analyze the HPT data for pure polycrystalline iron, AISI 1020 steel, and AISI 13B20 steel to reveal the distinctive strain breakpoints and power-law exponents. In the result, we found that deduced power-law exponents for AISI 1020 and AISI 13B20 steels are remarkably close to each other within full strain range.

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I. INTRODUCTION

SEVERE plastic deformation (SPD) is defined as a solid-state material treatment in which a very large strain is applied to a bulk sample to produce an ultrafine-grained material.\[1\] It should be noted that from the beginning, SPD was intended to be a part of industrial technology\[2,3\] that covers a variety of materials ranging from pure metals\[1\] to ceramics.\[4,5\] A historical review and the current development of SPD technology can be found in various studies.\[6–10\]

One of the main problems associated with the development of the quantitative theory of SPD is related to the fact that this material treatment technique is a synergetic product of several competing physical processes that alter the material micro/nanostructure. As a result, the material structure is neither crystal with long-range order nor amorphous solids with short-range order, but something in between, as pointed out by Birringer et al.\[11\] Despite the fact that these authors described nanocrystalline materials as gas-like solids, SPD materials can be considered as frozen quantum liquids because atoms/ions in SPD materials have a very strong interaction with each other (which is not a characteristic of gaseous media).

Remarkably, the first study of the physical properties of SPD materials was a study of SPD-processed quantum materials, i.e., superconducting niobium alloys reported by Fietz and Webb.\[12\] One of the milestones that results in the understanding of SPD are the experimental reports on the violation of the Hall–Petch relationship in ultrafine-grained materials.\[13–16\] This experimental result is in good agreement with our view that attempts to extrapolate physical laws established for microcrystalline materials on SPD materials (with minor modifications) should fail. However, because the theory of quantum materials is far from being fully developed, our current approach is to try to implement some empirical laws that have been discovered in the field of quantum materials in the last 50 years to SPD counterparts.

We chose SPD iron to conduct our study irrespective of the variety of materials available. Iron is a model metal\[14\] in which the properties of ultrafine-grained materials can be observed prominently. On the other hand, iron is a basic construction material in the modern world. Thus, any improvement in iron properties will have a massive impact on the global economy. To test
the applicability of the model for a wider class of materials, we analyzed AISI 1020 and AISI 13B20 steels.

In this study, we considered hardness as a property that can be measured conveniently in SPD materials. Thus, the establishment of a quantitative relationship for this property can be interesting for the entire research and development field.

The purpose of this study is to propose a model to reveal the quantitative relationship between hardness and true strain in a wide range of SPD materials.

II. EXPERIMENT

Deformation of pure iron (<0.03 wt pct impurities) and AISI 13B20 constructional low-carbon steel (composition (wt pct) 0.2C, 1.45Mn, 0.25Si, 0.003B, 0.012S, 0.011P) was carried out by high-pressure torsion in Bridgman anvils without lateral support (unconstrained HPT)\(^{[17]}\). The deformation pressure was 8 and 9 GPa, and the rate of anvil rotation was \(\omega = 1 \text{ rpm}\). The angle of anvil rotation was varied from 15 deg to 15 revolutions. Before deformation, the samples had the shape of a disk with a diameter of 5 mm and a thickness of 0.3 mm, and as a result of deformation they acquired the shape of a lens. With the deformation scheme used in this work, the maximum possible number of anvil revolutions is limited by a decreasing sample thickness. The true strain was calculated taking into account the angle of anvil rotation \(\omega\), the distance from the axis of rotation \((R_i)\), and the thickness of a sample before and after deformation \((h_0)\) and \(h_{IR}\), respectively:

\[
e = \ln \left(1 + \frac{[\varphi \cdot R_i]}{h_{IR}}\right)^{0.5} + \ln \frac{h_0}{h_{IR}}. \tag{1}\]

The Vickers hardness of iron and steel was measured at a load of 0.5 N and a loading time of 10 seconds. Measurements were performed along the radius of the samples at a step of 0.25 mm along two mutually perpendicular diameters. The results of the measurements made on various samples were averaged over the true strain range at a step of 0.2.

The structure of the deformed material was investigated by the thin foil method using a JEM 200CX transmission electron microscope at an accelerating voltage of 160 kV and a distance of 1.5 ± 0.2 mm from the center of a sample. The foils were prepared by mechanical thinning and subsequent electrolytic polishing in a chromic anhydride and phosphoric acid solution. The experiment is described in detail in Reference 19.

III. MODEL DESCRIPTION

Initially, we built a model to analyze the experimental data on HPT polycrystalline iron. In our previous report\(^{[19–21]}\) we showed that some SPD metals exhibit two distinctive microstructures and hardness vs. strain dependence, which is attributed to the relatively low and high true strain. In Figure 1, we present the hardness vs. true strain behavior of pure iron obtained from our previous studies\(^{[19,21]}\) along with TEM images of the material structure at minimal and maximal true strain achieved in our experiment. Each experimental data point in Figure 1 is the mean value of several measurements obtained from different samples. It should be noted that at small strain values, the microstructure of iron is mainly represented by dislocation cells that are disoriented by small angles within the original grains. This material structure was significantly different from the structure observed after large HPT true strains.\(^{[19,21]}\)

In the latter case, a new ensemble of strongly disoriented microcrystallites was observed, where the boundaries of initial grains were completely reconstructed. It can be observed from Figure 1 that despite distinctive differences in microstructures, it is difficult to reveal the characteristic dependence of hardness versus true strain for these two structural states of iron.

The existence of two stage structure was first demonstrated by Thompson\(^{[14]}\) in pure nickel. There are two distinctive grain size ranges, and the relationship between yield strength and grain size can be established for both of them. Later studies conducted on BCC metals (iron, niobium, and molybdenum) that exhibit stacking fault energies larger than 140 mJ/mol also showed two types of structures resulting from HPT treatment. The first type of structure consists of dislocation cells, whereas the second type consists of microcrystallites.\(^{[17,19,22–25]}\) It is well known that dislocation cells are formed due to the motion and interaction of individual dislocations, whereas microcrystallites are formed due to rotational deformation modes with the participation of collective disclination effects. Based on these findings, two physical mechanisms of structure formation were realized in BCC metals deformed at room temperature.

Thus, our model postulates the following:

1. There are two distinctive deformation mechanisms that determine material hardness.

![Fig. 1—Experimental data of hardness \(H(e)\) vs. true strain, and TEM images for structural Stage I and structural Stage II for pure iron.](image-url)
(2) There is a true strain range where two mechanisms coexist.
(3) In this coexisting range of true strain, the total hardness is the linear additive sum of each mechanism.

These three postulates are expressed in the general continuous piecewise fitting function proposed in this study:

\[ H(\varepsilon) = \theta(\varepsilon_2 - \varepsilon) \cdot H_1(\varepsilon) + \theta(\varepsilon - \varepsilon_1) \cdot H_2(\varepsilon) + \theta(\varepsilon - \varepsilon_2) \cdot \theta(\varepsilon_1 - \varepsilon) \cdot \left( \frac{|\varepsilon - \varepsilon_1|}{|\varepsilon_2 - \varepsilon_1|} \cdot H_1(\varepsilon) + \frac{|\varepsilon - \varepsilon_2|}{|\varepsilon_2 - \varepsilon_1|} \cdot H_2(\varepsilon) \right) \]

where \( H_1(\varepsilon) \) and \( H_2(\varepsilon) \) are hardness functions for structural states I and II, respectively \( [i.e., \text{within the strain range of} (0, \varepsilon_1) \text{and} (\varepsilon_2, \infty) \text{, respectively}] \), \( \varepsilon_1 \) is the free-fitting upper strain limit for structural state I, and \( \varepsilon_2 \) is the free-fitting nucleation breakpoint of structural state II.

For clarity, Figure 2 shows the weight function of Eq. [2]:

\[ y = \theta(\varepsilon_2 - \varepsilon) + \theta(\varepsilon - \varepsilon_1) + \theta(\varepsilon - \varepsilon_2) \cdot \theta(\varepsilon_1 - \varepsilon) \cdot \left( \frac{|\varepsilon - \varepsilon_1|}{|\varepsilon_2 - \varepsilon_1|} + \frac{|\varepsilon - \varepsilon_2|}{|\varepsilon_2 - \varepsilon_1|} \right) \]

which is split into two parts, \( i.e., \) structural state I and structural state II.

A continuous piecewise function approach is a feasible way to describe complicated systems. An approach that is extensively used in quantum materials and superconductivity is to find the scaling law of a key property vs. a major parameter. As mentioned by Fietz and Webb, the authors proposed the use of power-law scaling for the pinning force \( F_p \) vs. reduced magnetic field \( B/B_c2 \). Further development of this approach has been reported since then along with the decomposition of complicated temperature dependences of physical properties in superconductors and normal conductors as a sum of reduced polynomial values.

Thus, it is very convenient to use an analytical form for the hardness vs. true strain function in the following form:

\[ H_1(\varepsilon) = H_1(0) \cdot \left( 1 + \left| \frac{\varepsilon}{\varepsilon_2} \right|^\alpha \right), \text{stage I,} \]
\[ H_2(\varepsilon) = H_2(0) \cdot \left( 1 + \left| \frac{\varepsilon}{\varepsilon_2} \right|^\beta \right), \text{stage II,} \]

where \( H_1, H_2, P, Q, \alpha, \) and \( \beta \) are the free-fitting parameters of the model (Eq. [2]). \( P \) and \( Q \) describe the physical characteristic true strain within the two deformation stages, while \( \alpha \) and \( \beta \) are parameters that describe the degree of nonlinearity of the system at a given deformation stage.

IV. RESULTS AND DISCUSSION

A. Pure Polycrystalline Iron

The experimental \( H(\varepsilon) \) data and fit for Eqs. [2] and [4] for HPT iron are shown in Figure 3. The deduced values for \( \varepsilon_1 \) breakpoint, \( i.e., \) the point at which the cellular structure designated by Stage I disappears (indicated as A in Figure 3), and the nucleation breakpoint for Stage II \( \varepsilon_2 \), are shown in Eqs. [5] and [6].

\[ \varepsilon_1 = 5.4 \pm 1.0, \]

Fig. 3—Experimental data of hardness \( H(\varepsilon) \) vs. true strain and fit to Eqs. [1] and [3] for pure iron. (a) Total fit, breakpoints \( \varepsilon_1 \) and \( \varepsilon_2 \), and fit quality \( R = 0.994 \). (b) Fit, where contributions from both stages in overlapped strain range and power-law exponent are shown.
The most interesting result revealed by the fit is that the extrapolated $H_2(\varepsilon)$ curve (Eq. [4]) (indicated as B in Figure 3(b)) has a starting (nucleation) breakpoint at $\varepsilon_2 = 3.0 \pm 0.1$, which has the absolute value for hardness, i.e., $H_2(\varepsilon = 3.0 \pm 0.1) = 4.4 \pm 0.1\text{GPa}$. This value corresponds to the hardness of iron after significantly greater true strain ($\varepsilon = 8$). This means that the newly nucleated microcrystals in SPD iron in Stage II exhibit maximal strength properties. Perhaps, these first microcrystallites are analogous to ideal (defect-free) crystals. The latter demonstrate theoretical strength, and the appearance of defects upon deformation decreases the strength.[38]

It should also be noted that the deduced power-law exponents, $\alpha$ and $\beta$, as shown in Eqs. [7] and [8]:

$$\alpha = 0.63 \pm 0.13 \cong \frac{2}{3},$$  \hspace{1cm} [7]

$$\beta = 2.1 \pm 0.3 \cong 2 \hspace{1cm} [8]$$

are in good agreement with the theoretically predicted values for SPD body-centered cubic metals.[39,40]

Our previous approach[19] to study the structural stages in HPT-processed iron was based on transmission electron microscopy data and the analysis of hardness vs. true strain dependence, for which we employed a technique proposed in Reference 39. The technique involved plotting hardness data against the square root of true strain in which two or three linear parts depending on the material can be distinguished. The data shown in Figures 1 and 3(a) are represented in this manner in Figure 4. It can be observed from Figure 4 that SPD iron has three stages. The linear part in the low strain region ($\varepsilon \leq 4.0$) represents the first stage of the HPT treatment at which the cellular structure is formed. The strain boundaries for this stage were determined as $\varepsilon \leq 4.0$.[19] The highest true strain value for this region ($\varepsilon = 4.0$) was designated as $\varepsilon_{1-2}$ in Reference 19 and Figure 4.

The second linear part (Figure 4), which is called the mixed-type structure, covers the range of true strain of $4.0 \leq \varepsilon \leq 6.0$.[19] During this deformation, the iron microstructure is represented by dislocation cells and microcrystallites. At the largest true strain, with $\varepsilon \geq 6.0$, the iron microstructure was formed only by microcrystallites. The breakpoint between the second and third linear parts, i.e., $\varepsilon = 6.0$, was designated as $\varepsilon_{2-3}$ in Reference 19 and Figure 4.

It is interesting to compare the breakpoints deduced in our previous papers[19,21] with those deduced in this study. For instance, there is a reasonable agreement between $\varepsilon_{2-3} = 6.0 \pm 0.2$[19] (Figure 4) and the corresponding value of $\varepsilon_1 = 5.4 \pm 1.0$ (Figure 3) deduced in this study. It should be mentioned that this breakpoint corresponds to the HPT treatment at which dislocation cells vanish and the iron structure is transformed into a pure submicrocrystalline structure.[19,21]

The breakpoints of $\varepsilon_{1-2} = 4.0 \pm 0.2$ in Figure 4 and Reference 19, and $\varepsilon_{2-3} = 3.0 \pm 0.1$ (Figure 3) deduced in this study, did not agree well. We performed detailed TEM studies of iron deformed with the true strain of $\varepsilon = 3.2 \pm 0.2$ to confirm/disprove the position of $\varepsilon_{2-3} = 3.0 \pm 0.1$ for the breakpoint. We found that the iron structure deformed at $\varepsilon = 3.2$, as shown in Figure 5, has small, but detectable amounts of predicted microcrystallites. It is obvious that experimental technique given in Reference 19 (i.e., hardness vs. square root of true strain), owing to the integrated nature of hardness, cannot directly detect a small number of new elements in the structure, i.e., microcrystallites. Additionally, a disagreement between $\varepsilon_2$ and the $\varepsilon_{1-2}$ breakpoints (Figure 4) is also related to the fact that the model utilized in Reference 19 uses a fixed power-law exponent of 2. However, it does not use the overlapping idea of stages, which was implemented in the proposed model. Thus, our model is a new instructive research tool for determining strain breakpoints with better accuracy.

The deduced $\varepsilon_1, \varepsilon_2, \alpha,$ and $\beta$ parameters are listed in Table I. We did not show $H_1, H_2, P,$ and $Q$ parameters in Table I because different research groups used different indentation procedures. $H(\varepsilon)$ data can be represented in the hardness number units HV, however, we used GPa units in this study. As unitless $\varepsilon_1, \varepsilon_2, \alpha,$ and $\beta$ parameters describe the $H(\varepsilon)$ shape, these parameters should be comparable in any chosen experimental conditions and $H(\varepsilon)$ units.

It should be noted that the power-law exponent $\beta$, which characterizes the hardening rate at Stage II, is reasonably high. In our previous studies,[19] it was found that at the Stage II there is a strong refinement of the structure: the average size of microcrystallites in iron decreases from 0.19 $\mu$m at the beginning of the stage (at $\varepsilon_{2-3}$, Figure 4) to 0.06 $\mu$m at the maximum achieved deformation of $\varepsilon = 9.3$. We believe that the high $\beta$ value is associated with a decrease in the size of the microcrystallites.

![Fig. 4—The hardness vs. the square root of the true strain; the boundaries of stages from Ref. [20] are given in the figure.](image-url)
B. AISI 1020 Low-Carbon Steel

Low-carbon steels are the closest construction materials to pure polycrystalline iron. Thus, the analysis of experimental data for HPT-processed low-carbon steels can be a good test for model validity.

Based on several published $H(e)$ datasets on HPT-processed low-carbon steels, we analyzed the microhardness data reported by Cardona et al.\cite{39} for commercial AISI 1020 low-carbon steel. Cardona et al.\cite{41} performed microhardness measurements using an Essequay model 600 hardness tester at a load of 50 g F and a dwell time of 25 seconds. The hardness was reported in HV units. It should be noted that Cardona et al.\cite{41} reported the strain in their samples using the number of anvil rotations $N$ and rotation radius $R$.

In this study, we recalculated the values reported by Cardona et al.\cite{41} in true strain $\varepsilon$ using Eq. [1]. In these calculations, we used the sample thickness reported in Reference 42, where HPT treatment was performed under the same conditions and in the same apparatus as in Reference 41. Other details of the experiment can be found in Reference 41.

In Figure 6, we show the fit of $H(\varepsilon)$ data to Eqs. [2] and [4]. It can be observed from Figures 3 and 6 and Table I that carbon doping, even at a low level, causes a significant reduction in both breakpoint values:

$$e_1 = 3.4 \pm 0.1\text{ (designated as A in Fig. 6(b))},$$

$$e_2 = 2.07 \pm 0.05\text{ (designated as B in Fig. 6(b))}.$$ 

### Table I. Deduced $\varepsilon_1, \varepsilon_2, \alpha$, and $\beta$ Parameters for HPT Processed Polycrystalline Iron and Commercially Available Steels

| Material        | $\varepsilon_1$ | $\varepsilon_2$ | $\alpha$ | $\beta$ |
|-----------------|-----------------|-----------------|----------|---------|
| Pure Fe         | 5.4 ± 1.0       | 3.0 ± 0.1       | 0.63 ± 0.13 | 2.1 ± 0.3 |
| AISI 1020 Steel | 3.4 ± 0.1       | 2.07 ± 0.05     | 0.27 ± 0.05 | 1.5 ± 0.7 |
| AISI 13B20 Steel| 4.78 ± 0.25     | 1.85 ± 0.28     | 0.25 ± 0.07 | 1.7 ± 0.6 |

Fig. 5—Iron microstructure after deformation with $\varepsilon = 3.2 \pm 0.2$; (a) bright-field image, and (b) dark-field image taken in the reflection $\{110\}$. Micro-crystallites are marked with an arrow in panel (a). Scale bars are 250 nm.

Fig. 6—Experimental data of hardness $H(\varepsilon)$ vs. true strain and fit to Eqs. [2] and [4] for AISI 1020 low-carbon steel. (a) Total fit, breakpoints $\varepsilon_1$ and $\varepsilon_2$, and fit quality $R = 0.995$. (b) Fit where contributions from both stages in overlapped strain range and power-law exponent are shown.
There is also significant suppression in both power-law exponents, \( a \) and \( b \), which originates from the same reason, i.e., carbon doping.

C. AISI 13B20 Steel

AISI 13B20 steel has a carbon content similar to that of AISI 1020 steel. Boron doping improves the chemical and structural homogeneity of steel but does not alter the steel hardness.\(^\text{[19]}\) However, AISI 1020 and AISI 13B20 steels have a significant difference in the manganese content (0.3 to 0.6 pct in contrast to 1.4 pct, respectively). Thus, the difference in the deduced parameters for these HPT-processed steels can be attributed to magnesium doping.\(^\text{[19]}\)

In Figure 7, the \( H(e) \) data and fit to our model (Eqs. \([2]\) and \([4]\)) are shown. The deduced breakpoints are shown in Figure 7 and Table I.

\[
\begin{align*}
    e_1 &= 4.78 \pm 0.25 \text{ (designated as A in Fig. 7(b))}, \quad [11] \\
    e_2 &= 1.85 \pm 0.28 \text{ (designated as B in Fig. 7(b))}. \quad [12]
\end{align*}
\]

These values imply that Stage I in the AISI 13B20 steel exhibits a much wider strain range than that in the AISI 1020 steel. At the same time, the nucleation strain \( e_2 \) for Stage II, and power-law exponents \( a \) and \( b \) in AISI 1020 and AISI 13B20 steels are practically identical (Table I).

D. Comparison of the Parameters of the Piecewise Model for Steels AISI 1020 and AISI 13B20

As mentioned above, AISI 1020 and AISI 13B20 steels have practically identical \( e_2, a, \) and \( b \) values. At the same time, AISI 13B20 has a larger \( e_1 \) value than AISI 1020. We can hypothesize that doping with manganese increases the strain range where Stage I is exhibited. However, Zambrano\(^\text{[43]}\) calculated that Mn doping up to 10 wt pct decreases the stacking fault energy in Fe-Mn-0.5C steel. This implies that the dislocation mobility is an inverse function of manganese concentration in the aforementioned range.

A lower dislocation mobility causes the formation of a misoriented structure at much higher strain values. As a result, in steel containing more manganese, the Stage I can exist at much higher strain ranges in comparison with manganese-poor steel, and this is exactly what our analysis suggests.

Transmission electron microscopy examination of AISI 13B20 steel showed that a low-misoriented dislocation cellular structure was formed in Stage I (Figure 8(a)). Wide-dislocation cell boundaries do not create a discrete misorientation. Some high-angle discrete misorientations were detected in the steel structure after the true strain \( e = 2.1 \) (Figure 8(b)), which is slightly higher than \( e_2 \). However, there are still low-misoriented dislocation cells along with high-angle misorientations. The dislocation cellular structure is no longer observed after true strain \( e = 5.0 \) (Figure 8(c)), which is close to \( e_1 \). Thus, the calculated \( e_1 \) and \( e_2 \) values are in good agreement with the TEM-detected boundaries of the structural stages.

High values of the exponent for steels at Stage II (Table I), as in iron, can be associated with a strong refinement of the structure. It was found in Reference 19 that at this stage in AISI 13B20 steel the average size of microcrystallites reduces from 0.25 to 0.08 \( \mu \text{m} \) while the deformation is changing within the Stage II up to \( e = 8.2 \).\(^\text{[19]}\)

Cardona et al.\(^\text{[41]}\) did not report microstructural studies on the strain of HPT-processed AISI 1020. However, the deduced values for the breakpoints for AISI 1020 and AISI 13B20 steel are in good agreement with each other. This indicates that the model can be used to describe a material structure even without conducting detailed structural examination and determine the strain of the transition from slightly misoriented deformation structures to severely misoriented ones.
V. CONCLUSION

In this study, we propose a continuous piecewise model with two overlapping stages for the analysis of experimental hardness vs. true strain data of materials processed using the HPT method. The novelty of this approach lies in its ability to deduce free-fitting parameters and strain breakpoints that separate different micro/nanostructure modes generated during SPD processes. We applied the proposed model to analyze the experimental data for pure polycrystalline iron and two commercial steels, AISI 1020 and AISI 13B20.

For pure iron, our analysis revealed a distinctive strain boundary of \( \varepsilon = 3.0 \pm 0.1 \) at which a new submicrocrystalline structure starts to form in iron. Precise transmission electron microscopy study confirmed the accuracy of this deduced value.

The application of the proposed model for studying steels with similar compositions, AISI 1020 and AISI 13B20, revealed that these steels have practically identical \( \varepsilon_2 \), \( \varepsilon \), and \( \beta \) parameters. Thus, the proposed model adequately reflects the structural transformations in iron and iron alloys processed using HPT.

The proposed model can characterize the structural state of HPT-processed materials even in cases where microstructural examination was not performed. Therefore, the model can be used as an optimization tool to concentrate the true strain ranges at which microscopic studies can be performed.

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DATA AVAILABILITY

The data that support the findings of this study are available from the corresponding author upon reasonable request.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

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