Effect of Shock Loading on the Microstructure, Mechanical Properties and Grain Boundary Characteristics of HT-9 Ferritic/Martensitic Steels

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Abstract: The microstructural changes and mechanical response of an HT-9 sample shock loaded to a peak pressure of 11 GPa have been investigated by TEM, XRD, microhardness and EBSD techniques. Dislocation densities obtained by both direct measurements (via TEM) and indirect calculations (by XRD and hardness) indicate that shock loading results in ~2-3 fold increase in dislocation density. TEM analyses show that the shape, and density of the dislocations change after shock loading. In addition, shock loading causes local plastic deformation of the continuous parallel lath structure in some regions, together with an overall decrease in the aspect ratio of laths due to local plastic deformation and lath fragmentation. As a result of XRD analyses, the fraction of edge dislocations is determined to increase by ~24% after shock loading. Furthermore, hardness increases by ~40 HV after shock loading due to the increased dislocation density. EBSD analyses show that the fraction of CSL boundaries decreases by ~5-10 % as a result of shock loading.

1. Introduction:

The effect of shock-loading on the microstructural evolution and mechanical response of metals has been studied for over 50 years. By studying the capability of materials to withstand a shockwave created plastic wave, new knowledge can be obtained on defect generation and mobility mechanisms. The first response of materials to shock loading is elastic deformation within 100 picoseconds [1,2]. At the same time, if the pressure is high enough, second phase particles can precipitate due to instantaneous heating and activation of thermal processes [3]. If the pressure is not high enough for phase transformations, existing defects can be activated or new ones can be nucleated as slip continues along preferred planes. Following defect activation and slip processes, either twinning occurs if it is favorable, or strain hardening occurs owing to the interaction and entangling of dislocations which promotes the formation of new dislocations. Since BCC crystals lack the number of close packed planes compared to FCC crystals, in

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addition to having fewer slip planes, the interaction and entanglement of dislocations are more severe in BCC crystals [4]. On the other hand, shock generated deformation twinning in FCC metals is much more sensitive than BCC metals [5] to both material properties (e.g. grain size [6], crystallographic orientation with respect to the deviatoric stress state [7]) and experimental parameters (e.g. peak pressure [8] and pulse duration [9]).

In the literature, there are many studies on the shock response of iron-based materials. Together with the fully reversible phase transformation from alpha-iron (α) to epsilon-iron (ϵ) at 13 GPa [10–13], shock loading creates a number of defects [14]. Shock deformation in BCC iron alloys occurs by the competition between deformation twinning and slip mechanisms [15]. A critical twinning stress is postulated to be directly proportional to the stacking fault energy [16,17]. Therefore, under normal deformation conditions, pure BCC iron deforms by dislocation glide since it has a relatively high stacking fault energy [18,19]. On the other hand, low temperatures and high deformation rates facilitate twin formation in most crystal structures by retarding thermally-activated dislocation processes [15]. Recently, Gregory et al. [20] studied the effect of high strain rate explosive loading on the microstructure of plain carbon steels. It was discovered that shock loading leads to elongated grains in ferrite and pearlite phases and the formation of slip bands along with mechanical twins. Similarly, De Resseguier and Hallouin [21] observed a considerable amount of twin formation at high peak loading pressures up to 60 GPa in polycrystalline iron. They reported that twin volume fraction decreases with decreasing shock loading and predicted the elastic-plastic response of the materials and twin volume fractions as a function of pressure using a constitutive twinning model proposed by Johnson and Rohde [22].

Ferritic/martensitic HT-9 steel is a promising material with extensive irradiation experience for use in the nuclear materials community. Although the effect of shock loading on plain carbon steels, pure iron and FCC stainless steels has been extensively studied, there are no detailed studies on the effect of shock loading on microstructural and mechanical properties of BCC stainless steels. Therefore, in this study, the effect of shock loading on microstructure, dislocation density and mechanical properties, together with (sub)grain boundary characteristics has been investigated below the α -iron to ϵ -iron transformation pressure. As-received and shock deformed microstructures and mechanical properties were investigated using TEM, XRD, microhardness and EBSD techniques.

2. Experimental Procedure:

The composition of the HT-9 alloy studied is given in Table 1. The alloy was normalized at 1040 °C for 30 minutes and air cooled followed by tempering at 760° C for 1 hour and air cooling. During tempering, carbon released from martensite precipitated as carbides on the lath boundaries and primary austenite grain boundaries.

The present shock recovery experiments were performed using an 80-mm single-stage launcher. The shock recovery technique was presented previously elsewhere [23,24]. A 5.08-mm-thick, 25.4-mm-diameter sample was stacked behind a 38-mm-diameter, 1-mm-thick cover plate and slip fit into a precisely bored recess on top of a 7 degree tapered, 12.7-mm-thick, inner momentum disk. Sample spallation was suppressed by backing the sample assembly with a 3.8-mm-thick spall plate, which also served as a longitudinal momentum trap. The central disk and spall plate were further surrounded by two concentric rings with outside diameters of 69.8 and 82.5 mm which protected the center of the sample from large amplitude release tensions. All assembly components were fabricated from AISI 4340 steel to ensure good impedance matching with the HT-9 steel sample during shock loading. The sample assembly was placed inside a steel impact cylinder that permitted passage of the sample inner momentum trapping ring through a central hole but stopped the projectile. Samples were shock loaded to an ~11 GPa peak pressure and "soft" recovered and cooled by decelerating the sample and inner momentum trapping ring in a water catch chamber positioned immediately behind the impact area [24].

Standard metallographic techniques were used to prepare the samples for characterization. Jet electropolishing was performed using a solution of perchloric acid (5%) and methanol at -40°C with an applied voltage of 20 V. Samples were investigated using both TEM and SEM. The microstructure and dislocation morphology were characterized using an FEI Tecnai F30 TEM operating at 300kV. Dislocation densities from TEM images were calculated by using two different methods. A line-intercept method defined in Ref [25] was used for the TEM images taken at the same kinematic conditions, whereby six randomly placed lines of different angular orientation were drawn over the TEM images. The dislocation density ρ is simply the number of points N divided by the total line length of the random lines L_t , multiplied by foil thickness t [26]:

$$\rho = \frac{N}{L_t t} \dots\dots\dots(\text{Eq. 1})$$

A second technique was used to check the reliability of the dislocation density results obtained from the line-intercept method. This technique was employed simply by measuring the total length (L_t) of the dislocations divided by the volume of the investigated region (V). The thickness of the region of interest was measured by the energy filtered TEM technique which introduces ~10% error to the measurements. The total dislocation density ρ was calculated using the following relationship [27]:

$$\rho = \frac{L_t}{V} \dots\dots\dots(\text{Eq. 2})$$

In addition to the TEM investigations, dislocation densities were also calculated using X-ray diffraction and hardness methods. X-ray analysis of the specimens was conducted with Rigaku Ultima III. The wavelength of the source was Cu K_α containing both $K_{\alpha 1}=1.54056$ Å and $K_{\alpha 2}=1.5444$ Å. Instrumental broadening was determined by using NIST calibrated LaB_6 powders. Scans were operated from 10° to 140° with the rate of $0.02^\circ/\text{min}$ using a constant slit to reveal the diffraction planes of (110), (200), (211), (220), (310) and (222). Moreover, the effect of shock loading on the mechanical properties was tested by measuring microhardness using a Struers DuraScan-70 hardness tester with 1 kg load.

The grain boundary characteristics of the as-received and shock pretrained specimens were examined with electron backscatter diffraction (EBSD) using an FEI Inspect FEG SEM equipped with TSL EBSD equipment. Since the sample was tilted 70° from the horizontal, diffraction data were obtained from a very thin surface layer, meaning that the results were surface sensitive. During scans, an acceleration voltage of 20 kV and aperture size of 50 μm were used. The size of the scan region was determined according to the grain and substructure size as $50\mu\text{m} \times 145\mu\text{m}$. The step size showing the scan rate of the selected region was kept at 140 nm for all scans.

3. Results and Discussion:

3.1. Effect of Shock Loading on the Microstructure and Mechanical Properties

The effect of shock loading on the microstructure was investigated by TEM and XRD methods while microhardness and compression tests were utilized to assess the shock loading effect on the mechanical properties. Dislocation densities of the as-received and as-shocked samples were

obtained by using TEM, XRD and hardness methods. Details of those methods are explained in the following sections.

3.1.1. Effect of Shock Loading on the Microstructure

Figure 1 shows the TEM bright field images of the HT-9 samples in the as-received and as-shocked conditions. In the as-received condition, carbides precipitate along primary austenite grain boundaries together with parallel lath boundaries as a result of tempering. After shock loading, carbides remain at the lath and prior austenite boundaries; however, the dislocation density increases considerably (~2-3 fold). The dislocation density of the as-received sample is calculated as $3.3 \pm 0.7 \times 10^{14} \text{ m}^{-2}$ while it is calculated as $7.8 \pm 1.6 \times 10^{14} \text{ m}^{-2}$ for the as-shocked sample. The calculated errors occur during thickness and dislocation counting measurements.

Detailed investigations performed under the same kinematic conditions (g-vector of (110)) indicate that the dislocation structures in both cases are quite different. Initially, dislocations are continuous and distributed homogeneously within the laths (Figure 2.a). On the other hand, dislocations are observed to become entangled as a result of shock loading. Moreover, the dislocation density at the lath boundaries is considerably higher compared to the inner parts of the laths (Figure 2.b). As a result of the high dislocation density at the lath boundaries, contrast of the carbide particles increases leading to a coarsened shape.

Yasunaga et al. observed that at high strain rates exceeding $\sim 10^3 \text{ s}^{-1}$, dislocation cell structures do not tend to form and small dislocation loop formation occurs in BCC metals of V, Nb and Mo [28]. Similarly, HT-9 F/M steel shows dislocation loops without dislocation cell formation after high rate shock loading as shown in Figure 3. As an example, some of the dislocation loops are circled with red dashed lines.

Together with subtle changes in the dislocation structure, shock loading is observed to reflect local plastic deformation of the continuous parallel lath structure in some regions, as well as an overall decrease in the aspect ratio of laths due to local plastic deformation and lath fragmentation. Figure 4.a and 4.b show bright field images of different regions where several of the continuous laths are observed to be deformed and fragmented. Lath boundaries are denoted by the dashed red lines for better visualization in Figure 4.b. Fragmentation of the lath structure is consistent with previous observations in the literature. Zel'dovich et al. [29] studied the effect

of shock loading on the cementite laths in a pearlite structure and observed that depending on the macroscopic orientation of the pearlite structure relative to the shock-wave propagation direction, the cementite laths were observed to be either deformed or locally fragmented. In addition, Figure 4.c shows that the overall aspect ratio of the laths is reduced in some regions. This decrease in the aspect ratio of the laths is attributed to the local plastic deformation of the laths compared to the surrounding microstructure during the plastic compression and rarefaction portions of the shock pulse. Since shock loading subjects a sample to principally 1-D uniaxial strain compression [30], plastic flow predominantly occurs in the loading direction. Accordingly, if the lath structure lies parallel with the direction of shock loading, plastic flow is mostly along the laths resulting in preservation in the parallel structure. Conversely, if the packets are oriented at an angle to the loading direction, plastic deformation and localized shear deformation occur across lath boundaries and inside of laths rather than along laths and lath boundaries. Consequently, fragmentation of the laths leads to an overall reduction in the lath aspect ratio.

X-ray diffraction was another method implemented to investigate the effect of shock loading on the microstructure of HT-9 steels. Dislocation densities were calculated by X-ray diffraction line profile analyses. The diffraction angles and FWHM values for the as-received and as-shocked samples before subtraction of instrumental broadening are given in Table 2. There are no clear changes in the diffraction angles as a result of the peak shift. On the other hand, the peak broadening as a result of shock loading is quite pronounced especially at high diffraction angles which is consistent with the significant increase in dislocation density.

Using the modified Williamson-Hall method [31-35], dislocation densities of the as-received and as-shocked HT-9 steel samples were calculated by using the broadening values in Table 2 subtracted by the instrumental broadening as in the following equation.

$$\Delta K \cong \frac{0.9}{D} + \sqrt{\frac{\pi M^2 b^2}{2}} \rho^{1/2} K \bar{C}^{1/2} + O(K^2 \bar{C}) \dots \dots \dots (\text{Eq. 3})$$

where $K = 2 \sin \theta / \lambda$ and $\Delta K = \cos \theta [\Delta(2\theta)] / \lambda$. Here θ is diffraction angle, $\Delta(2\theta)$ is the full width at half maximum (in radians), and λ is the wavelength of the x-rays which is 1.54056 Å for Cu radiation. In Eq. 3, D is the average substructure size, M is a constant that depends on both the effective outer cut-off radius and the density of dislocations, b is Burger's vector and ρ

is dislocation density. O represents the higher order terms in $K^2\bar{C}$ where $\bar{C}$ is the dislocation contrast factor and represented by Eq. 4.

$$\bar{C} = \bar{C}_{h00}(1 - qH^2) \quad \text{.....(Eq. 4)}$$

$\bar{C}_{h00}$ is the average dislocation contrast factor on $(h00)$ and is determined by crystal elasticity. It is calculated as 0.285 for pure iron [36]. Moreover, H^2 is a plane indices-dependent variable which can be represented as $H^2 = \frac{(h^2k^2 + h^2l^2 + k^2l^2)}{(h^2 + k^2 + l^2)^2}$. The q value is a parameter dependent on elastic constants of the crystal and edge or screw character of dislocations.

Eq. 3 can be converted to the linear relationship of $((\Delta K)^2 - \alpha) / K^2$ vs. H^2 as;

$$[(\Delta K)^2 - \alpha] / K^2 \cong \beta \bar{C}_{h00}(1 - qH^2) \quad \text{.....(Eq. 5)}$$

where $\alpha = (0.9/D)^2$ and $\beta = \pi M^2 b^2 \rho / 2$. In order to be able to calculate α values, average lath sizes of 698 ± 33 nm and 870 ± 51 nm were measured by EBSD analyses for as-received and as-shocked HT-9 samples, respectively. The slope of the $((\Delta K)^2 - \alpha) / K^2$ vs. H^2 plot shown in Figure 5 gives $-\beta \bar{C}_{h00} q$. Moreover, the q values of 2.10 ± 0.08 and 1.77 ± 0.09 were obtained for the as-received and as-shocked samples, respectively from the intercept of linear fit to the x-axis.

To calculate dislocation density from the slope of the plots, M is required. M is defined as $R_e \sqrt{\rho}$ where R_e is the effective outer cut-off radius of dislocations which is $R_e \approx 0.78R$. R is the radius

of the circular sub-area and it is defined as $\frac{Gb}{2\tau}$ where G is shear modulus, b is Burger's vector

and τ is shear stress. Consequently, R_e values for the as-received and as-shocked samples are calculated as 20.1 nm and 16.5 nm, respectively. By inserting the R_e values into the slope

formula of $-\beta \bar{C}_{h00} q = -\frac{\pi R_e^2 b^2 \rho^2}{2} \bar{C}_{h00} q$, the dislocation densities of as-received and as-shocked

specimens are calculated as $4.7 \pm 0.4 \times 10^{14} \text{ m}^{-2}$ and $10.3 \pm 0.6 \times 10^{14} \text{ m}^{-2}$, respectively.

Theoretical values of q for edge and screw dislocations in pure iron are given as 1.28 and 2.67, respectively [37]. By using Eq. 6 the dislocation character can be determined as;

$$f^{edge} = \frac{q_{th}^{screw} - q_{exp}}{q_{th}^{screw} - q_{th}^{edge}} = 1 - f^{screw} \dots\dots\dots (Eq. 6)$$

where f^{edge} and f^{screw} are the fractions of edge and screw dislocations, respectively. By inserting the theoretical and experimental values of q into Eq. 6, the fractions of edge dislocations are determined to be 0.41 ± 0.06 for the as-received sample; and 0.65 ± 0.06 for the as-shocked sample showing an increase in fraction of edge dislocations from shock loading. Tang and Marian [38] studied the effect of high strain rate loading on single crystal iron by using dislocation dynamics simulations. At low temperatures, the mobility of edge dislocations is 2-3 times higher than that of screw dislocations resulting in plastic flow occurring mainly by screw dislocations. On the other hand, at higher temperatures and varying strain rates, it was discovered that edge dislocations move slower and plastic flow occurs mainly by entanglement of edge dislocations. They also reported that the fraction of the screw dislocations asymptotically converges to a rate-independent but temperature dependent value, f_{eq} . Consequently, f_{eq} values at 100, 300, and 600 K are calculated as 0.95, 0.73, and 0.55. These calculations show that with the increase in temperature, the mobility of screw dislocations increases. Therefore, the contribution of edge dislocations to the plastic deformation increases. Adiabatic heating during shock loading leads to an increase in the temperature resulting in increased mobility of screw dislocations. Consequently, the density of screw dislocations decreases and the fraction of entangled edge dislocations rises.

3.1.2. Effect of Shock Loading on the Mechanical Properties

To understand the effect of shock loading on the mechanical properties, microhardness was measured. Hardness tests show that while the average hardness of the HT-9 sample is 246 ± 3 HV before shock loading, it increases to 282 ± 2 HV after shock loading.

The relationship between the Vickers hardness and the yield stress, which was originally described by Tabor [39] with a proportionality constant of ~ 3 has been investigated extensively. Busby et al. [40] showed that the proportionality constant between the hardness change (ΔH_v) and yield stress change ($\Delta \sigma_y$) is 3.03 for austenitic steels and 3.06 for ferritic steels. The yield stresses of as-received and as-shocked samples were measured in compression as 692 ± 14 MPa and 842 ± 17 MPa, respectively and reported in our previous study [5]. As a result, the

proportionality constant between the hardness change and the yield stress change is derived as 2.35 ± 0.81 which is close to the results previously documented in the literature within error.

According to the Taylor's formula [41], yield stress can be related to the square root of dislocation density using the following formula;

$$\sigma_y = \sigma_0 + M\alpha Gb\sqrt{\rho} \dots\dots\dots(\text{Eq.7})$$

where σ_y is the yield stress, M is Taylor factor which is ~ 3 for polycrystalline metals [41,42], G is the shear stress (86.95 GPa for HT-9 steel), b is the length of the Burger's vector, 2.466 Å for the $\langle 111 \rangle \{110\}$ slip system, ρ is the dislocation density, and α is a factor describing the strength of the obstacles that a moving dislocation has to overcome during plastic deformation. Moreover, σ_0 is the stress required to move a dislocation in the absence of other dislocations that can arise as a result of solutes, Peierls-type stresses, grain-size strengthening, etc. The values of α being 0.22 and σ_0 being 386 MPa for HT-9 steel are obtained experimentally from the slope and y-intersection point of yield stress vs. dislocation density in Ref [43]. Hence, by using the above stated parameters, the dislocation densities of the as-received and as-shocked samples are calculated as $4.7 \pm 0.4 \times 10^{14}$ and $10.4 \pm 0.8 \times 10^{14}$, respectively. This is a rudimentary calculation assuming that only dislocations contribute to the hardening without considering the effect of lath size and second phase particles; nevertheless, the results appear reasonable and consistent.

In summary, dislocation density measurements and calculations by TEM, XRD and hardness methods conclude the same result that dislocation density increases ~ 2 -3 fold after shock loading. Table 3 shows the compiled dislocation densities obtained by the above stated methods. It should be noted that while dislocation densities calculated by XRD and hardness methods are almost the same, dislocation density measurements from TEM images give lower values. The reason can simply be attributed to the 2-D imaging of dislocations under TEM. Dislocations lying perpendicular to the image plane are not accounted into the measurements. On the other hand, XRD and hardness methods provide 3-D information from larger sampling regions. However, the dislocation density increase as a result of shock loading is ~ 2 -3 fold for all three cases.

3.2. Effect of shock loading on the substructure boundaries: EBSD study

Grain boundaries are vulnerable sites for corrosion, cracking, segregation and precipitation. Since the composition and misorientation across the boundaries vary within a sample, grain boundary character can also play an important role during degradation processes [44]. In this study, the effect of shock loading on the characteristics and amount of the boundaries has been investigated using EBSD technique. Figure 6 shows the distribution of phase boundary misorientations in the as-received and shock loaded HT-9 samples. Figure 6.b and 6.e indicate that primary austenite grain boundaries have the misorientation angle between 15° and 50° which corresponds to random grain boundaries (RGBs). On the other hand, the misorientation angle between the laths, within and between the packets generally lies between 2° - 15° (Figure 6.a and 6.d) and 50° - 65° (Figure 6.c and 6.f) corresponding to low angle boundaries (LABs) and special boundaries, respectively.

Figure 7 shows the misorientation angle distribution of both the as-received and as-shocked HT-9 samples within single grains. For each condition, ~ 20 grains from at least three different regions were studied. The most obvious common feature of both conditions is that there is almost no boundary with a misorientation angle between 20° and 47° [45]. Although the fraction of the low angle grain boundaries ($2^\circ < \theta < 15^\circ$) and high angle grain boundaries between 45° and 57° remain constant after shock loading, the type of the boundaries having the misorientation angle of $\sim 60^\circ$ decreases by ~ 5 - 10% . The postulated reason for this change is discussed in detail in the following section and can simply be attributed to the atomic displacements as a result of a high dislocation density at the lath boundaries after shock loading.

While the misorientation between the laths within the packets is primarily between 50° and 60° , the packet boundaries generally have misorientation angles of 10° - 20° and 47° - 50° as shown in Figure 8.

EBSD analyses show that there are five different types of CSL (coincidence site lattice) boundaries in the investigated materials, namely $\Sigma 3$, $\Sigma 11$, $\Sigma 25b$, $\Sigma 33c$ and $\Sigma 41c$ whose misorientation angles are tabulated in Table 4. The boundaries having $\Sigma > 29$ are not special boundaries; however the deviation angle, $\Delta\theta$, from the ideal conditions should be taken into account while determining the special boundaries. $\Delta\theta$ for the $\Sigma 3$ can be calculated using the Palumbo-Aust criterion which is much more restrictive than the widely used Brandon's criterion [46]. By using the Palumbo-Aust criterion that is given as $\Delta\theta \leq 15^\circ \Sigma^{-5/6}$, $\Delta\theta$ can be calculated

as 6° for $\Sigma 3$. According to this calculation, the misorientation angle for $\Sigma 3$ is $60^\circ \pm 6$ which includes the $\Sigma 33c$ and $\Sigma 41c$.

Table 5 shows the fraction of special boundaries in the as-received and as-shocked conditions. It should be noted that the error introduced by the OIM (orientation imaging microscopy) analysis of EBSD results is less than 5%. In both conditions, the amount of $\Sigma 3$ boundaries is higher compared to the other types of boundaries which is expected for the martensite structures. As a result of shock loading, the amount of the special boundaries decreases from $45.6 \pm 2.3\%$ to $37.6 \pm 1.9\%$. This is attributed to the pile up of dislocations at the lath boundaries (Figure 2), which is the main origin of increasing the average deviation degree and decreasing the fraction of low- Σ boundaries. Low- Σ boundaries, especially $\Sigma 3$ boundaries have the lowest grain boundary energy. During plastic deformation in one direction, the generated dislocations pile up at the boundaries and the grain boundary energy increases. In order to reduce the energy, atoms near the lath boundaries should move to lower energy sites [47]. Besides these displacements, a number of atoms at the boundaries are removed in order to reduce the grain boundary energy. After the displacements and removals, the deviation degrees are beyond the maximal deviation; therefore, CSL structures are lost. This is postulated to be the reason why especially the fraction of $\Sigma 3$ boundaries decreases with shock deformation. It was also found that even though shock loading to 11 GPa results in a decrease in the amount of special boundaries, it does not affect the crystallographic orientation relationship between austenite and martensite.

Conclusions:

The microstructural changes and the mechanical response of an HT-9 steel shock loaded to a peak pressure of 11 GPa in the normalized and tempered condition have been investigated by TEM, XRD, microhardness and EBSD techniques. Results from TEM, XRD and microhardness methods are shown to be quite consistent and lead to the conclusion that a ~2-3 fold increase in dislocation density occurs as a result of shock loading. TEM analyses indicate that after shock loading the dislocations become entangled and concentrated mostly near or at the lath boundaries. Shock loading also causes local plastic deformation of the continuous parallel lath structure in some regions as well as an overall decrease in the aspect ratio of laths due to local plastic deformation and lath fragmentation. XRD analyses show that the amount of edge dislocations increases from 0.41 ± 0.06 to 0.65 ± 0.06 after shock loading. Moreover, the hardness

is seen to increase by ~40 HV after shock loading compared to the as-received condition. In addition, EBSD analyses indicate that shock loading causes a reduction in the fraction of CSL boundaries from $45.6 \pm 2.3\%$ to $37.6 \pm 1.9\%$.

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Figure 1: TEM bright field images of the a) as-received sample b) shocked loaded sample with the peak pressure of 11 GPa at room temperature.

Figure 2: TEM bright field images of a) as-received b) as-shocked conditions along (110) direction showing the structure of dislocations.

Figure 3: TEM bright field image of the as-shocked sample showing the dislocation structure and dislocation loops (red dashed circles) on $g=(110)$.

Figure 4: TEM bright field images of shocked loaded HT-9 sample showing a&b) the destruction of continuous parallel lath structure at different locations and magnifications c) the large aspect ratio as a result of shock loading.

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406 Figure 5: The modified Williamson–Hall plots of the FWHM for a) as-received b) as-shocked

407 specimens. The Miller indices of the reflections are also indicated.

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410 Figure 6: EBSD images of the as-received HT-9 steel showing the misorientation angles of a) 2°-

411 15° (LAB), b) 15°-50° (RGB) and c) 50°-65° (special boundaries); as-shocked HT-9 steel

412 showing the misorientation angles of d) 2°-15° (LAB), e) 15°-50° (RGB) and f) 50°-65° (special

413 boundaries).

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417 Figure 7: Histogram of misorientation fraction inside single primary austenite grains for the as-
418 received and as-shocked HT-9 samples.

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421 Figure 8: Misorientation angles between the packets of a) as-received b) as-shocked HT-9
422 samples (— 10°-20°; — 47°-50°)

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Table 1: Composition of the HT-9 samples (wt %).

C	Cr	Mo	Si	Mn	Ni	V	W
0.21	12.5	1.10	0.29	0.41	0.60	0.30	0.51

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429 Table 2: The XRD data of each reflection of the as-received and as-shocked HT-9 steels.

Bragg Reflection	2 θ /deg (as-received)	FWHM ($\Delta 2\theta$)/deg (as-received)	2 θ /deg (as-shocked)	FWHM ($\Delta 2\theta$)/deg (as-shocked)
(110)	44.7	0.3	44.7	0.4
(200)	64.9	0.4	64.9	0.6
(211)	82.2	0.5	82.2	0.6
(220)	98.7	0.6	98.7	0.7
(310)	116.0	0.8	116.0	1.1
(222)	136.5	1.0	136.5	1.5

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431 Table 3: Dislocation densities of the as-received and as-shocked HT-9 samples obtained by
432 different methods.

	Dislocation density (m ⁻²) TEM	Dislocation density (m ⁻²) XRD	Dislocation density (m ⁻²) Hardness
As-received	3.3 $\pm$ 0.7x10 ¹⁴	4.7 $\pm$ 0.4x10 ¹⁴	4.7 $\pm$ 0.4x10 ¹⁴
As-shocked	7.8 $\pm$ 1.6x10 ¹⁴	10.3 $\pm$ 0.6x10 ¹⁴	10.4 $\pm$ 0.8x10 ¹⁴

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436 Table 4: CSL boundaries existing in the HT-9 as-received and as-shocked samples and
437 corresponding misorientation angles.

Σ	θ (degrees)	uvw
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3	60.0	111
11	50.5	110
25b	51.7	331
33c	59.0	110
41c	55.9	110

Table 5: CSL boundary fractions before and after shock loading.

	As-received	As-shocked
CSL ($\Sigma 3$) (%)	40.1 $\pm$ 2.0	32.8 $\pm$ 1.6
CSL ($\Sigma 11$) (%)	4.4 $\pm$ 0.2	3.6 $\pm$ 0.2
CSL ($\Sigma 25b$) (%)	1.1 $\pm$ 0.1	1.2 $\pm$ 0.1
Total # of CSL boundaries (%)	45.6 $\pm$ 2.3	37.6 $\pm$ 1.9