**Characterization of Phase Properties and Deformation in Ferritic-Austenitic Duplex
Stainless Steels by Nanoindentation and Finite Element Method**

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Abstract

The phase properties and deformation behavior of the δ -ferrite and γ -austenite phases of CF-3 and CF-8 cast duplex stainless steels were characterized by nanoindentation and microstructure-based finite element method (FEM) models. The elastic modulus of each phase was evaluated and the results indicate that the mean elastic modulus of the δ -ferrite phase is greater than that of the γ -austenite phase, and the mean nanoindentation hardness values of each phase are approximately the same. The elastic FEM model results illustrate that greater von Mises stresses are located within the δ -ferrite phase, while greater von Mises strains are located in the γ -austenite phase in response to elastic deformation. The elastic moduli calculated by FEM agree closely with those measured by tensile testing. The plastically deformed specimens exhibit an increase in misorientation, deformed grains, and subgrain structure formation as measured by electron backscatter diffraction (EBSD).

Keywords: Steel, Micromechanics, EBSD, Nanoindentation, Finite Element Method

1. Introduction

Cast duplex stainless steels (CDSS), such as CF-3 and CF-8, are commonly used as structural materials in cooling water pipes, valve bodies, pump casings, and elbows of commercial light water nuclear reactors. These systems operate at service temperatures between 280 °C (553 K) and 320 °C (593 K), and it is well known that thermal embrittlement occurs due to phase decomposition of the body-centered cubic (b.c.c.) δ -ferrite phase under these conditions [1]–[3]. In an effort to evaluate the possibility of extending the service life of these reactors up to 80 years and predict the evolution of the mechanical response of CF-3 and CF-8 CDSS, it is necessary to analyze the deformation behavior and mechanical response in these materials. Studies have been conducted to determine the bulk mechanical properties of CDSS in order to verify their reliability as long-term components. The microhardness, impact toughness, and ultimate tensile strength (UTS) have been evaluated, and the results show that some effects of phase decomposition can be measured by changes in these properties [4]–[9]. However, decomposition occurs in the ferrite phase, which often constitutes less than 20 vol.% of the microstructure [7]–[9]. Despite the aforementioned studies and the interest in thermal aging embrittlement of CF-3 and CF-8 steels, the differences in mechanical properties of the constituent phases are not well known. Further, stress and strain distributions and partitioning in these steels related to the different properties of the constituent phases have not been investigated.

Finite element method (FEM) modeling based on microstructures observed by optical microscopy or scanning electron microscopy is an increasingly relevant approach to gain insight into deformation behavior, stress and strain distributions, and failure initiation in dual-phase steels and stainless steels [10]–[14]. Duplex steels can be modeled following a composite

material model of mechanical behavior, where the bulk mechanical behavior of the steel is dependent on the morphology and distribution of the phases and their corresponding elastic moduli, E , Poisson's ratios, ν , and volume fractions, f , [15]–[17]. In general, the literature contains limited results on the constituent phase properties of some duplex stainless steels [18], [19], but there are no reported results for the phase properties of the CF-3 and CF-8 steels. With these properties evaluated, it is possible to analyze the micromechanical behavior of the duplex steels by FEM, which can help elucidate structure-property relationships in these steels, as demonstrated recently on TRIP and DP 590 steels [12], [20].

Instrumented nanoindentation provides an established technique to measure the elastic properties of metals and alloys by permitting selection of a single-phase grain. The relationship between indentation and elastic properties was initially discussed by Tabor [21]. Oliver and Pharr [22] presented a method (henceforth referred to as the Oliver and Pharr method) to determine the elastic modulus and hardness of a single-phase grain from the load-displacement curve of a sharp nanoindentation test that has been implemented in ASTM Standard E2546-07 [23]. More recently, nanoindentation has been employed to measure plastic properties, such as the strain-hardening exponent, n , and yield strength, σ_y , of metals and alloys that obey power-law strain-hardening behavior. Dao, et al. formulated a reverse analysis method (henceforth referred to as the Dao method) combining a dimensionless analytical approach with experimental nanoindentation data and tested its validity on 6061-T6511 and 7075-T651 aluminum (Al) alloys [24]. Ogasawara, et al. applied a modified approach to the Dao method (henceforth referred to as the Ogasawara method) in order to obtain the strain-hardening exponent and yield strength of a wider range of alloys that obey power-law strain-hardening behavior, including brass, gold (Au), copper (Cu), and Al specimens [25]. The extant literature indicates that both the Oliver and

Pharr and Dao methods provide reasonably accurate results for the elastic properties of many metals and alloys, including some dual-phase steels and stainless steels [17], [19], [24], [26]. The applicability of using instrumented nanoindentation to determine plastic properties of duplex stainless steels has not, however, been resolved.

This investigation characterizes the properties of the ferrite and austenite phases in the CF-3 and CF-8 CDSS by instrumented nanoindentation and then uses these properties to develop a microstructure-based finite element method (FEM) model. We compare the differences between the Oliver and Pharr and Dao analysis methods to determine the elastic properties by nanoindentation. The limitations of the Dao and Ogasawara methods in determining the plastic properties of these dual-phase steels are discussed. The FEM model is used to analyze the elastic deformation of the constituent phases. We discuss the distribution of stresses and strains in the CDSS microstructures determined by elastic FEM, including evaluating partitioning of the stresses and strains between the two phases and comparing the effects of microstructure morphology and constituent phase properties of the steels. The bulk elastic modulus calculated by FEM is compared with that measured by tensile testing. The misorientation distribution resulting from tensile plastic deformation is measured by electron backscatter diffraction (EBSD) using kernel average misorientation (KAM) and grain orientation spread (GOS) analysis.

2. Experimental Procedures

2.1. Materials and Metallographic Preparation

The steels investigated in this study were obtained from Monett Company, Inc. as 17.8 cm × 3.3 cm × 2.9 cm rectangular keel blocks that were cast to the CF-3 and CF-8 standard specification outlined in ASTM A351-14 [30]. All blocks of each steel type were statically cast

from the same melt followed by a solution treatment at 1065 °C (1338 K) for two hours and quenched in water at room temperature in accordance with the ASTM standard. The nominal chemical composition of the two steels as measured by optical emission spectroscopy (OES) is provided in Table 1. Specimens were mechanically cut into blanks for machining and the blanks were further reduced to approximately 10 mm × 10 mm × 3 mm specimens and cold mounted in epoxy for metallography. The specimens were ground and polished to 0.3 μ m surface finish and etched with Kalling's reagent No. 1 at room temperature following standard procedures to reveal the dual-phase microstructure.

Table 1. Nominal composition (wt.%) of the CF-3 and CF-8 cast duplex stainless steels (CDSS).

Steel		Composition (wt.%)						
		Fe	Cr	Ni	Si _{max}	Mn _{max}	Mo _{max}	C _{max}
CF-3	ASTM A351	Balance	17.0–21.0	8.0–12.0	2.00	1.50	0.50	0.03
	Unaged	69.52	19.69	8.40	0.98	1.07	0.28	0.02
CF-8	ASTM A351	Balance	18.0–21.0	8.0–11.0	2.00	1.50	0.50	0.08
	Unaged	69.44	19.85	8.30	0.97	0.99	0.35	0.06

2.2. Optical Microscopy and Volume Percentage (Fraction) Calculations

Optical microscopy was performed employing a Buehler[®] ViewMet Inverted Metallograph. The volume percentage (fraction) of the b.c.c. δ -ferrite and face-centered cubic (f.c.c.) γ -austenite phases in the microstructure were determined employing three different methodologies: (1) manual point counting in accordance with the ASTM E562-11 standard, (2) digital counting of pixels using the Image-J version 1.50b software in accordance with the ASTM E1245-03 standard, and (3) the Schoefer diagram based on composition in accordance with the ASTM

A800-14 standard [27]–[29]. The percentages determined by methods (1) and (2) were measured by utilizing 20 optical micrographs from each steel as specified in the standards.

2.3. Tensile Testing

Cylindrical test specimens with a diameter of 6.35 mm (0.25 in.) and a gauge length of 25.4 mm (1.0 in.) were fabricated in accordance with the ASTM E8-13a standard [30]. Tensile tests were performed employing an Instron[®] 8502 test frame equipped with an 8800 controller in accordance with the ASTM standard procedures. A preload of ~100 N was applied before the start of each test and a calibrated extensometer with 50% elongation capability was used for continuous measurement of strain using strain-rate control rate of 0.00025 s^{-1} . The extensometer was removed at its elongation limit and the tensile specimens were tested to failure using crosshead speed control rate of $0.006125 \text{ mm s}^{-1}$. Determination of the bulk plastic tensile properties was carried out following the procedures outlined in the ASTM E8-13a Standard [30].

2.4. Electron Backscatter Diffraction (EBSD)

The misorientation distribution resulting from tensile plastic deformation of the γ -austenite and δ -ferrite phases were investigated by the EBSD technique using a FEI Inspect Field Emission Gun (FEG) scanning electron microscope (SEM) instrument equipped with TSL EBSD detector. Specimens taken from the transverse section of the tensile specimen gauge lengths were prepared by standard metallographic techniques followed by jet electropolishing using a solution of 5 vol.% perchloric acid in methanol at approximately $-30 \text{ }^{\circ}\text{C}$ with an applied voltage of 23 V. Experiments were performed on a 70° pre-tilted holder at an accelerating voltage of 20 kV and aperture size of $50 \text{ }\mu\text{m}$. The EBSD scans were performed at two different magnifications. The scanned area was either $\sim 0.25 \text{ }\mu\text{m} \times \sim 0.5 \text{ }\mu\text{m}$ or $\sim 0.75 \text{ }\mu\text{m} \times \sim 1.5 \text{ }\mu\text{m}$ and the step size for data collection was $0.4 \text{ }\mu\text{m}$ and $1.2 \text{ }\mu\text{m}$, respectively. Data analyses were

performed using TSL OIM software. Kernel average misorientation (KAM) studies were conducted using an exclusion angle of 5° and considering the second nearest neighbors. Grain orientation spread (GOS) analyses were performed using a 5° tolerance angle.

2.5. Nanoindentation

Load-controlled nanoindentation testing was performed employing a calibrated Hysitron 900 Triboindenter with a diamond Berkovich tip to 4000 μN maximum load. The 40 s total indentation time was divided into three segments consisting of 15 s loading time, 10 s holding time, and 15 s unloading time. Testing was performed by creating three 2×2 grids of indents spaced 2 μm apart in each phase. Three CF-3 and three CF-8 specimens were indented for a total of 36 indents in both the δ -ferrite and the γ -austenite phases for both steels.

2.6. Finite Element Method Model

A two-dimensional (2-D) finite element method (FEM) model of the unaged CF-3 and CF-8 microstructures was developed in the ANSYS[®] Mechanical APDL version 16.2 software. The optical micrographs of the dual-phase microstructures were converted to vector format using RasterVect version 21.5 software. The vectorized microstructure images were meshed in the ANSYS[®] software using a triangular Solid, Plane 183 element. There are six nodes per element and the elemental edge dimension is 0.075 units (equivalent to $\sim 3.2 \mu\text{m}$). The elastic material model properties of the ferrite and austenite phases utilized in the FEM model were obtained by instrumented nanoindentation, as discussed below. A uniform tensile displacement was applied in the y-direction along the top row of nodes. The bottom row of nodes was constrained in the y-direction and the center bottom node was constrained in the x-direction in order to simulate a strain-rate controlled tensile test [15], [31]. The displacement was applied to the equivalent of 0.1% strain, which is within the elastic deformation region. The deformation conditions were

validated with models consisting of a single-phase δ -ferrite crystal, a single-phase γ -austenite crystal, and a rectangular bi-crystal model with one δ -ferrite/ γ -austenite heterophase interface and found to produce no extraneous stresses or artifacts in either the x- or y-directions. The mesh size dependency at the defined element size of 0.075 units was evaluated and found to be negligible.

3. Results and Discussion

3.1. Microstructural Characterization

An optical micrograph of the CF-3 steel, Fig. 1, illustrates an example microstructure consisting of a network of island-shaped δ -ferrite phase (dark color) in the continuous γ -austenite matrix phase (light color). The microstructure of the CF-8 steel has a qualitatively similar appearance to that of the CF-3 steel. This microstructure is consistent with those reported in the literature of a steel with a composition similar to CF-8, with a ferrite content of less than 20% by volume [9], [32].

Figure 1. An optical micrograph of an example CF-3 cast duplex stainless steel (CDSS) microstructure illustrating the face-centered cubic (f.c.c.) γ -austenite (light) matrix phase and a network of body-centered cubic (b.c.c.) δ -ferrite (dark) phase islands.

The b.c.c. δ -ferrite phase volume percentage of the CF-3 steel is greater than the CF-8 steel when measured by the three aforementioned methods, Table 2. By the manual point counting method, the CF-3 steel ferrite volume percentage is 11.5 ± 1.1 vol.% while that of the CF-8 steel is 9.1 ± 0.9 vol.%. The digital counting method results in a ferrite volume percentage of 12.1 ± 1.0 vol.% for the CF-3 steel and 10.0 ± 0.4 vol.% for the CF-8 steel. The Schoefer method based on the CF-3 and CF-8 nominal compositions predicts ferrite volume percentages of 18 ± 6.3 vol.% and 14 ± 4.8 vol.%, respectively. The Schoefer diagram is based on Cr equivalency and Ni equivalency values, where the magnitude of each element's contribution to the b.c.c.-forming chromium equivalency term, Cr_{eq} , and f.c.c.-forming nickel equivalency term, Ni_{eq} , affect the formation of either the δ -ferrite or the γ -austenite phase, respectively [33]. The CF-3 steel has a Cr_{eq}/Ni_{eq} ratio of 1.34, whereas the CF-8 steel has a ratio of 1.25, resulting in a larger expected ferrite volume percentage in the former. Since the Schoefer diagram is not specific to the CDSS used in this study and is based on generalized composition and element equivalency data of many duplex stainless steels, the values measured by the manual point counting method and the digital counting of pixels method are likely more accurate. The directly measured values are, however, within the range determined by the Schoefer diagram for both steels.

Table 2. Ferrite volume percentage in the unaged CF-3 and CF-8 cast duplex stainless steels (CDSS) as determined by three different methods.

Ferrite Volume Percentage (%)			
Steel	Manual Point Counting [ASTM E562-11]	Digital Counting of Pixels [ASTM E1245-03]	Schoefer Diagram [ASTM A800-14]
CF-3	11.5 ± 1.1	12.1 ± 1.0	18 ± 6.3
CF-8	9.1 ± 0.9	10.0 ± 0.4	14 ± 4.8

3.2. Tensile Properties and Misorientation Distribution Mapping

Engineering stress-strain curves for the CF-3 and CF-8 stainless steels are presented in Fig.

2. The bulk elastic modulus of the CF-3 steel is approximately 189.1 GPa, whereas it is approximately 169.6 GPa in the CF-8 steel. The CF-3 stainless steel exhibits a yield strength of 280 MPa and an ultimate tensile strength (UTS) of 530 MPa. The corresponding values in the CF-8 steel are greater, at 286 MPa and 570 MPa, respectively. The CF-3 and CF-8 steels exhibit approximately the same ductility of 60% and 61% total elongation, respectively. While the yield strength and ductility of the two steels are similar, the greater bulk tensile strength of the CF-8 steel can be attributed to the greater C concentration in CF-8 and the presence of $M_{23}C_6$ carbides at the heterophase interfaces [33].

Figure 2. Engineering stress-strain curves for the CF-3 and CF-8 steels. Both steels reach their ultimate tensile strength (UTS) at approximately 45% elongation. Dashed lines approximate remaining curves to the fracture stress after reaching the elongation limit of the extensometer.

Figure 3. Kernel average misorientation (KAM) maps superimposed on interface boundary maps having a misorientation angle $\theta > 5^\circ$ for CF-3 (a) in undeformed and (b) 45% strained (deformed) specimens; and for CF-8 (c) in undeformed and (d) 45% strained (deformed) specimens. The degree of misorientation is indicated by the color scale.

The KAM maps of undeformed and 45% strained CF-3 specimens, Figs. 3(a) – (b), and CF-8 specimens, Figs. 3(c) – (d), respectively, illustrate the misorientation distribution within the microstructures. The undeformed CF-8 exhibits greater misorientation than the undeformed CF-3 steel. One likely contributor to the measured difference between the undeformed steels is the

greater nominal concentration of C in the CF-8 and the presence of the aforementioned $M_{23}C_6$ -type carbides at the heterophase interfaces. Additionally, both CF-3 and CF-8 exhibit greater misorientation at the δ -ferrite/ γ -austenite interfaces than in the interior of the grains in the undeformed condition, which indicates a higher dislocation density at these locations [34].

Figure 4. Grain orientation spread (GOS) maps for 45% strained (deformed) (a) CF-3 and (b) CF-8 specimens. The degree of misorientation with respect to each grain is indicated by the color scale.

The KAM maps after plastic deformation, Figs. 3(b) and 3(d), illustrate that the amount of misorientation has increased in both steels. Furthermore, plastic deformation leads to deformation of the grains, an increase in the quantity of high-angle boundaries, and formation of subgrain structure throughout the microstructure of both steels. As seen from the grain orientation spread (GOS) maps of the 45% deformed tensile specimens, Figs. 4(a) – (b), there is no clear trend regarding the location of high or low misorientation based on the grain or phase structure. Thus, GOS analysis illustrates that the greatest local misorientation can be located in

either the ferrite phase or the austenite phase. This suggests that the extent of plastic deformation is similar in both phases.

3.3. Ferrite and Austenite Elastic Properties Evaluated by Nanoindentation

An example load-displacement curve for the f.c.c. γ -austenite phase in the CF-3 steel, Fig. 5, illustrates the response of the phase during loading, holding, and unloading. The response of the b.c.c. δ -ferrite phase in the CF-3 steel and the same phases in the CF-8 steel have a qualitatively similar appearance but exhibit quantitative differences. The initial linear part of the unloading curve is assumed to be demonstrative of entirely elastic behavior as it measures linear recovery during removal of the Berkovich tip [24]. The stiffness, S , which is used to determine the elastic modulus, E , of each phase in both steels was calculated as the slope of the unloading segment of the curve starting from the maximum indentation load, P_{max} , and fitting the first 67% of the curve (i.e. $1.00 \times P_{max}$ to $0.33 \times P_{max}$) by a linear least squares method [35]. The quantity E^* is a function of the elastic modulus and Poisson's ratio of the indenter tip, E_i and ν_i , and the modulus and Poisson's ratio of each phase, E and ν , by the relationship: $E^* = \left[\frac{1-\nu^2}{E} + \frac{1-\nu_i^2}{E_i} \right]^{-1}$, where ν is assumed to be 0.29 for both phases in these CDSS [36]. The quantity E^* is related to the indentation load-displacement curve by a projected area of elastic contact, A , and the quantity S . Although the mean stiffness value of each phase is the same in both the Oliver and Pharr and Dao methods, the quantity A is obtained by different calculations leading to slightly different values of A , shown in Table 3. An estimate of the quantity A is determined directly from the geometry of the Berkovich indenter and the maximum depth, h_{max} , in the Oliver and Pharr method [22], whereas it is determined by simultaneously solving a system of two equations for the quantities A and E^* in the Dao method [24].

Figure 5. An example load-displacement curve for the f.c.c. γ -austenite phase in a CF-3 stainless steel specimen exhibiting 15 s loading segment, 10 s holding segment, and 15 s unloading segment when tested by instrumented nanoindentation.

The value of the quantity E for each phase in both the CF-3 and CF-8 CDSS is solved by the above equation and presented in Figs. 6(a) – (b). The Dao method mean elastic modulus agrees to within 12% of the Oliver and Pharr method mean elastic modulus for the γ -austenite phase in both the CF-3 and CF-8 steels, and to within 5% for the δ -ferrite phase. Additionally, the Dao method mean elastic modulus is consistently lower in value than the Oliver and Pharr method by approximately between 8 and 20 GPa, Figs. 6(a) – (b), due to the different estimates of the projected area of contact. In general, the elastic moduli determined by the two methods are in good agreement and the mean elastic modulus of the ferrite phase is greater than that of the austenite phase by approximately 10 GPa in both the CF-3 and CF-8 stainless steels. The elastic modulus values obtained are also slightly less than those reported in a study by Gadelrab, et al.

of a Uranus 50 duplex stainless steel, when employing the Oliver and Pharr method [19]. Guo, et al. reported greater values for the elastic moduli of the constituent phases in a Z3CN20-09M duplex stainless steel, although their methodology was not specified [18]. In each study, the ferrite phase is reported to possess a greater elastic modulus than the austenite phase.

Table 3. Mean values of the elastic parameters in the ferrite and austenite phases of the CF-3 and CF-8 CDSS derived from the individual load-displacement curves and the estimated area of contact determined by the Oliver and Pharr (O&P) and Dao methods.

<i>Material</i>		<i>Measured</i>			<i>Calculated</i>	
Stainless Steel	Phase	$h_{\max}$ (nm)	$P_{\max}$ (μN)	S ($\mu\text{N}/\text{nm}$)	$A^{\text{O\&P}}$ ($\text{nm}^2 \cdot 10^{-5}$)	A^{Dao} ($\text{nm}^2 \cdot 10^{-5}$)
CF-3	Ferrite	173.9	3995.5	169.4	7.84	8.14
	Austenite	176.0	3995.5	166.0	8.02	8.50
CF-8	Ferrite	176.6	3994.3	171.1	8.11	8.50
	Austenite	176.0	3994.5	162.5	7.96	9.22

The Oliver and Pharr and Dao methods are also utilized to determine the nanoindentation hardness, H , of the two phases in both the CF-3 and CF-8 CDSS. In both methods, the nanoindentation hardness is calculated directly from the quantity $P_{\max}$ and the quantities $A^{\text{O\&P}}$ and A^{Dao} , Table 3. The calculated mean nanoindentation hardness values of each phase in both stainless steels are slightly greater than 5 GPa when using the Oliver and Pharr method. The Dao method calculations show that the mean nanoindentation hardness values of the b.c.c. δ -ferrite phase for both steels agree closely with those from the Oliver and Pharr method. The mean nanoindentation hardness values of the f.c.c. γ -austenite phase are, however, approximately

Figure 6. Comparison of calculated mean elastic modulus, E , and nanoindentation hardness, H , for the b.c.c. δ -ferrite and f.c.c. γ -austenite phases between the Oliver and Pharr and Dao methods for the (a) CF-3 and (b) CF-8 stainless steels. The mean values of both quantities are slightly greater in the ferrite phase of both steels.

between 5% and 10% lower than those from the Oliver and Pharr method, Figs. 6(a) – (b). The ferrite phase mean nanoindentation hardness is slightly harder than that of the austenite phase.

Furthermore, the Dao method shows a greater difference in the quantity H between the two phases of the steels when compared to the Oliver and Pharr method. This could be demonstrative of a real physical difference in nanoindentation hardness between phases, indicating that the Dao method is better suited toward elucidating this difference due to the use of experimentally

measured data in developing the dimensionless equations [24]. Alternatively, the difference could be caused by a better fit of the nanoindentation data to the dimensionless equations for the b.c.c. δ -ferrite than the f.c.c. γ -austenite phase. In contradistinction, the Oliver and Pharr method does not rely on calibration to experimentally measured data [22].

3.4. Ferrite and Austenite Plastic Properties Evaluated by Nanoindentation

The Dao and Ogasawara methods each provide an approach to determining the plastic behavior of each phase – in terms of the representative stress, σ_r , at a specified value of the plastic strain, r , and the quantity n – based on dimensionless equations derived from the parabolic loading portion of the nanoindentation curve, Fig. 5. The quantity σ_y can be determined from the calculation of n and σ_r by utilizing a power-law relationship. However, values of σ_r determined for the ferrite and austenite phases in the CF-3 and CF-8 steels in this investigation were found to be an order of magnitude higher than the experimentally determined bulk yield stress. In a majority of cases, the equations failed to return a value for the quantity n between 0 and 1, and the values that could be determined for the quantity n varied widely. Because the calculation of yield stress is a function of both the representative stress and strain-hardening exponent, the yield stress could not be calculated when the quantity n was indeterminate. The σ_y values that could be calculated were unreasonably high following the trend for the quantity σ_r .

These results suggest that there are limitations in applying these methods to determine the plastic properties of some stainless steels. Guelorget, et al. [37] were also unable to return a solution for the quantity σ_y when using the Ogasawara method in a 316L austenitic stainless steel. The limitations could be caused by deviation from the region of applicability of the Dao method ($n \leq 0.3$ or $0.3 < n \leq 0.5$ and $\frac{\sigma_y}{E^*} < 0.03$) and the Ogasawara method ($0 \leq n \leq 0.5$ and

$3 \leq \frac{\bar{E}}{\sigma_r} \leq 3300$; where $\bar{E} = \frac{E}{1-\nu^2}$). Solutions outside of these parameters are subject to greater variation and there exists the possibility of a non-unique solution. Furthermore, it is possible that the δ -ferrite and the γ -austenite phases in the CF-3 and CF-8 CDSS do not strictly follow power-law strain-hardening behavior, as is the case for some duplex steels [38], [39].

3.5. Elastic Deformation Analysis by Finite Element Method

We consider here the elastic properties determined by nanoindentation using the Oliver and Pharr method, Figs. 6(a) – (b), where the results indicate that the b.c.c. δ -ferrite phase is elastically stiffer than the f.c.c. γ -austenite phase for both steels. The elastic modulus values were utilized in the material models of the 2-D FEM microstructure-based models of the CF-3, Fig. 7, and CF-8 steels. The Poisson's ratio is assumed to be 0.29 for both phases and applicability of the von Mises yield criterion is assumed.

Figure 7. Microstructure-based finite element method (FEM) model mesh of the CF-3 microstructure in Fig. 1. The cyan regions are areas defined by γ -austenite elastic properties and the purple islands are areas defined by δ -ferrite elastic properties.

The FEM results for the example CF-3 and CF-8 microstructures in Figs. 8(a) – (d) illustrate the effects of applied elastic deformation on the δ -ferrite and γ -austenite phases. Contour plots of the von Mises stresses determined by FEM, Figs. 8(a) – (b), demonstrate higher δ -ferrite phase stresses (yellow, orange, and red contours) and the lower γ -austenite phase stresses (blue and green contours). The highest stresses (red contours) occur in the narrowest regions of the ferrite phase. Similarly, the von Mises strains, Figs. 8(c) – (d), exhibit significant heterogeneity in both steels'

Figure 8. Contour plots of the FEM results for (a) von Mises elastic stresses in CF-3, (b) von Mises elastic stresses in CF-8, (c) von Mises elastic strains in CF-3, and (d) von Mises elastic strains in CF-8. Higher stresses and strains are represented by warm coloration (red, orange,

yellow) and lower stresses and strains are represented by cool coloration (green, blue, indigo). Legends are in units of MPa for stress maps and $\mu\text{m}/\mu\text{m}$ for strain maps. microstructures. In general, the strains are greater in the γ -austenite phase (green, yellow, orange, and red contours) than in the δ -ferrite phase (blue contours). The maximum stress determined by FEM for this applied deformation condition is 188 MPa for CF-3, Fig. 8(a), whereas it is 187 MPa for CF-8, Fig. 8(b). In both cases, the stresses determined from the FEM model are about 100 MPa less than the bulk yield stress. The maximum von Mises strain is calculated as 0.1028 % in the CF-3 steel, Fig. 8(c), and 0.1043 % in the CF-8 steel, Fig. 8(d). The bulk elastic modulus calculated from the FEM model by using the stress in the y-direction and the applied strain are 179.3 GPa and 174.1 GPa for CF-3 and CF-8, respectively, which are within 6% of the experimentally measured bulk values.

Since the von Mises elastic stresses and strains exhibit significant heterogeneity in the microstructure of both steels, the average stresses and strains in each phase were calculated in order to quantify the visual results by determining partitioning ratios of the form $\sigma_\gamma/\sigma_\delta$ and $\varepsilon_\gamma/\varepsilon_\delta$, which permit direct comparison of the ferrite and austenite response. The partitioning ratios of the von Mises stresses ($\sigma_\gamma/\sigma_\delta$) for CF-3 and CF-8 are 0.984 and 0.978, respectively, which indicates that the stresses are higher in the ferrite phase. The partitioning ratios of the von Mises strains ($\varepsilon_\gamma/\varepsilon_\delta$) indicates that the strains are higher in the austenite phase, where the ratios are 1.022 and 1.038 for CF-3 and CF-8, respectively. Furthermore, the stress and strain distributions are influenced by differences in morphology of the microstructures in the steels. The CF-3 exhibits greater stress and strain homogeneity in the matrix phase away from the heterophase interfaces when compared to CF-8. Larger areas of uniform coloration in the relatively unconstrained regions of the austenite matrix phase away from the heterophase interfaces can be observed in Figs. 8(a) and 8(c) when compared to Figs. 8(b) and 8(d).

The heterogeneity of the stress and strain distributions can be explained by the difference in elastic modulus of the phases. The δ -ferrite phase will resist elastic deformation more than the γ -austenite phase. Thus, stress is transferred from the γ -austenite matrix to the δ -ferrite islands in order to maintain compatibility at the interfaces. The stresses are highest in ferrite islands that are elongated in a direction parallel to the direction of applied deformation. Choi, et al., attributed this phenomenon in TRIP steel to a state of elevated stress triaxiality, where the constraints placed on the microstructure result in higher stresses perpendicular to the applied deformation direction [12]. A larger value of the hydrostatic component of stress is expected in these locations as a result, which suggests that these are regions of interest for void initiation during the onset of ductile failure [40].

In these steels, both phases are considered to be ductile [41] and thus both phases are expected to deform plastically. The regions exhibiting the highest von Mises stresses and strains are the most likely locations for the onset of yielding. Since the yield stress values for the constituent phases could not be determined in the present study by nanoindentation due to limitations of the extant models, the plastic stress and strain distributions were not investigated by FEM in this study. Analysis of the misorientation distributions related to the plastic strain after deformation of the CF-3 and CF-8 steels suggests that the strengths of the constituent phases are similar. Since the elastic deformation stresses are found to be higher in the δ -ferrite phase than in the γ -austenite phase, and assuming equal values for the yield stress, the ferrite phase would plastically deform prior to the austenite phase. However, the actual location for the onset of yielding will depend on the exact magnitude of the yield stresses relative to the stress and strain distributions in the microstructure.

4. Conclusions

The elastic modulus and nanoindentation hardness have been determined by instrumented nanoindentation for the b.c.c. δ -ferrite and f.c.c. γ -austenite phases in CF-3 and CF-8 CDSS. The elastic moduli were utilized in a microstructure-based FEM model of example CDSS microstructures, which reveal the stress and strain distributions and partitioning during elastic deformation. The misorientation distribution after plastic deformation of the tensile specimens was evaluated by EBSD. The following conclusions have been made from this investigation:

- (1) The ferrite volume percentages in the CF-3 and CF-8 CDSS were calculated by the manual point counting method, as 11.5 ± 1.1 vol.% and 9.1 ± 0.9 vol.%, respectively. These values are consistent with those determined via digital pixel counting method, which determine the ferrite volume percentages as 12.1 ± 1.0 vol.% and 10.0 ± 0.4 vol.% for CF-3 and CF-8, respectively.
- (2) The CF-3 steel is has a bulk yield strength of 280 MPa and a tensile strength of 530 MPa. The CF-8 has higher yield and tensile strengths, which were measured as 286 MPa and 570 MPa, respectively. Electron backscatter diffraction (EBSD) mapping of the deformed tensile specimens reveals that plastic deformation leads to deformation of the grains and formation of subgrain structure. The CF-8 is shown to possess greater misorientation than CF-3 in the undeformed condition and both steels have their greatest misorientation at the grain boundaries and δ -ferrite/ γ -austenite heterophase interfaces. Plastic deformation results in increased misorientation in both steels.

(3) Two methods, those formulated by Oliver and Pharr and by Dao, et al., were utilized to determine the elastic modulus and nanoindentation hardness of δ -ferrite and γ -austenite in CF-3 and CF-8. The measured values for the elastic modulus, E , by the Oliver and Pharr method are $E_{CF-3}^{\delta} = 185.3 \pm 39.6$ GPa, $E_{CF-3}^{\gamma} = 178.6 \pm 33.0$ GPa, $E_{CF-8}^{\delta} = 183.4 \pm 25.2$ GPa, and $E_{CF-8}^{\gamma} = 173.1 \pm 19.6$ GPa. The Dao method results for elastic modulus are $E_{CF-3}^{\delta} = 176.6 \pm 43.5$ GPa, $E_{CF-3}^{\gamma} = 166.7 \pm 32.9$ GPa, $E_{CF-8}^{\delta} = 175.3 \pm 33.0$ GPa, and $E_{CF-8}^{\gamma} = 153.6 \pm 22.9$ GPa.

(4) Calculation of the elastic modulus by FEM results in $E = 179.3$ GPa and $E = 174.1$ GPa for CF-3 and CF-8, respectively, which agree to within 6% of the values measured by bulk tensile testing.

(5) The FEM results illustrate that the b.c.c. δ -ferrite phase exhibits greater elastic stresses than the f.c.c. γ -austenite phase, while the austenite phase exhibits greater elastic strains. The corresponding partitioning ratios based on the average stresses and strains in each phase were calculated as $\frac{\sigma_{\gamma}}{\sigma_{\delta}} = 0.984$ and $\frac{\varepsilon_{\gamma}}{\varepsilon_{\delta}} = 1.022$ for CF-3 and $\frac{\sigma_{\gamma}}{\sigma_{\delta}} = 0.978$ and $\frac{\varepsilon_{\gamma}}{\varepsilon_{\delta}} = 1.038$ for CF-8.

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List of Tables

Table 1. Nominal composition (wt.%) of the CF-3 and CF-8 cast duplex stainless steels (CDSS).

Steel		Composition (wt.%)						
		Fe	Cr	Ni	Si _{max}	Mn _{max}	Mo _{max}	C _{max}
CF-3	ASTM A351	Balance	17.0–21.0	8.0–12.0	2.00	1.50	0.50	0.03
	Unaged	69.52	19.69	8.40	0.98	1.07	0.28	0.02
CF-8	ASTM A351	Balance	18.0–21.0	8.0–11.0	2.00	1.50	0.50	0.08
	Unaged	69.44	19.85	8.30	0.97	0.99	0.35	0.06

Table 2. Ferrite volume percentage in the unaged CF-3 and CF-8 cast duplex stainless steels (CDSS) as determined by three different methods.

Ferrite Volume Percentage (%)			
Steel	Manual Point Counting [ASTM E562-11]	Digital Counting of Pixels [ASTM E1245-03]	Schoefer Diagram [ASTM A800-14]
CF-3	11.5 ± 1.1	12.1 ± 1.0	18 ± 6.3
CF-8	9.1 ± 0.9	10.0 ± 0.4	14 ± 4.8

Table 3. Mean values of the elastic parameters in the ferrite and austenite phases of the CF-3 and CF-8 CDSS derived from the individual load-displacement curves and the estimated area of contact determined by the Oliver and Pharr (O&P) and Dao methods.

Material		Measured			Calculated	
Stainless Steel	Phase	h _{max} (nm)	P _{max} (μN)	S (μN/nm)	A ^{O&P} (nm ² ·10 ⁻⁵)	A ^{Dao} (nm ² ·10 ⁻⁵)
CF-3	Ferrite	173.9	3995.5	169.4	7.84	8.14
	Austenite	176.0	3995.5	166.0	8.02	8.50
CF-8	Ferrite	176.6	3994.3	171.1	8.11	8.50
	Austenite	176.0	3994.5	162.5	7.96	9.22

List of Figure Captions and Figure Details

Figure 1. An optical micrograph of an example CF-3 cast duplex stainless steel (CDSS) microstructure illustrating the face-centered cubic (f.c.c.) γ -austenite (light) matrix phase and a network of body-centered cubic (b.c.c.) δ -ferrite (dark) phase islands.

Color figure Recommended. 1 column width.

Figure 2. Engineering stress-strain curves for the CF-3 and CF-8 steels. Both steels reach their ultimate tensile strength (UTS) at approximately 45% elongation. Dashed lines approximate remaining curves to the fracture stress after reaching the elongation limit of the extensometer.

Color figure Recommended. 1 column width.

Figure 3. Kernal average misorientation (KAM) maps superimposed on interface boundary maps having a misorientation angle $\theta > 5^\circ$ for CF-3 (a) in undeformed and (b) 45% strained (deformed) specimens; and for CF-8 (c) in undeformed and (d) 45% strained (deformed) specimens. The degree of misorientation is indicated by the color scale.

Color figure Required. 1.5 column width.

Figure 4. Grain orientation spread (GOS) maps for 45% strained (deformed) (a) CF-3 and (b) CF-8 specimens. The degree of misorientation with respect to each grain is indicated by the color scale.

Color figure Required. 1 column width.

Figure 5. An example load-displacement curve for the f.c.c. γ -austenite phase in a CF-3 stainless steel specimen exhibiting 15 s loading segment, 10 s holding segment, and 15 s unloading segment when tested by instrumented nanoindentation.

Color figure Recommended. 1 column width.

Figure 6. Comparison of calculated mean elastic modulus, E , and nanoindentation hardness, H , for the b.c.c. δ -ferrite and f.c.c. γ -austenite phases between the Oliver and Pharr and Dao methods for the **(a)** CF-3 and **(b)** CF-8 stainless steels. The mean values of both quantities are slightly greater in the ferrite phase of both steels.

Color figure Recommended. 1 column width.

Figure 7. Microstructure-based finite element method (FEM) model meshes of the CF-3 microstructure in Fig. 1. The cyan regions are areas defined by γ -austenite elastic properties and the purple islands are areas defined by δ -ferrite elastic properties.

Color figure Required. 1 column width.

Figure 8. Contour plots of the FEM results for **(a)** von Mises elastic stresses in CF-3, **(b)** von Mises elastic stresses in CF-8, **(c)** von Mises elastic strains in CF-3, and **(d)** von Mises elastic strains in CF-8. Higher stresses and strains are represented by warm coloration (red, orange, yellow) and lower stresses and strains are represented by cool coloration (green, blue, indigo). Legends are in units of MPa for stress maps and $\mu\text{m}/\mu\text{m}$ for strain maps.

Color figure Required. 2 column (full page) width.