

# Influence of Microstructure and Strain Hardening Rate on Acoustic Nonlinearity Parameter in Stainless Steel 316L during Tensile Loading

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### **ABSTRACT**

The accumulation of dislocations, which are atomic defects in materials subjected to plastic deformation, can cause structural failures. Early detection of such dislocation-related damage is essential to prevent these failures. The acoustic nonlinearity parameter  $\beta$  has been shown to be sensitive to the nonlinearity of dislocation motions, and prior research has shown a relationship between  $\beta$  and dislocation parameters in various damage mechanisms. While most work thus far reports that  $\beta$  generally increases with increased plastic deformation, recent research showed that  $\beta$  can decrease during monotonic tensile loading in stainless steel 316L characterized by in situ nonlinear ultrasonic measurements. The objective of this research is to examine the correlation between the decrease of  $\beta$  with plastic strain as reported in this recent study, and the initial microstructure and strain hardening rate. The initial microstructure, characterized with electron backscatter diffraction (EBSD), shows an increase in dislocation density and a reduction of grain area, which can possibly result in a decrease in  $\beta$ . Further, it is shown that the decrease rate of  $\beta$  monotonically decreases with hardening rate, providing a evidence that the decrease in  $\beta$  may relate to the shift from planar slip to wavy slip. These results help interpret the underlying mechanisms for the decrease in  $\beta$  during tensile loading.

**Keywords:** Nonlinear ultrasound, Rayleigh wave, Acoustic nonlinearity parameter, EBSD, Grain Area, Kernel Average Misorientation, Strain hardening rate, Plastic deformation, In situ measurements

#### INTRODUCTION

Nonlinear ultrasound (NLU) is a nondestructive measurement to evaluate the degree of material nonlinearity. This technique involves the propagation of an ultrasonic wave of fundamental frequency through a material containing dislocation-based damage. Due to the nonlinear stress-strain response, a proportion of the wave's energy is transferred to the second harmonic. The acoustic nonlinearity parameter  $\beta$  is a measure that characterizes the degree of nonlinear ultrasonic response exhibited by such defects. The measured  $\beta$  is composed of two components, the intrinsic material nonlinearity ( $\beta^{lat}$ ) and the nonlinearity of defects ( $\beta^{def}$ ). The first component,  $\beta^{lat}$ , remains almost constant during plastic deformation, while the second component,  $\beta^{def}$ , continuously changes [2]. According to previous studies,  $\beta$  exhibits a linear relationship with dislocation density [3, 4]. However, it is also proportional to the fourth power of dislocation characteristic length, encompassing parameters such as monopole loop length and dipole height.

Recent work showed that in contrast to most prior work, beta decreased with increasing plastic strain during in situ NLU measurements of monotonic tensile loading [1]. Here, we further study the microstructure of this material to better understand the decrease of beta. In the prior study, a stainless steel (SS) 316L specimen was prepared by hotrolled and subsequently water quenched, then solution annealed at 1100C for 1 hour and water quenched, resulting in a yield stress of approximately 220 MPa and a Young's modulus of 200 GPa. Then specimen underwent 4

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incremental loading processes, with a total strain of 6.0% in stress control and a strain rate of 0.001  $s^{-1}$ . During the tensile test, the specimen was held at fixed stress levels for NLU measurements. The loading paths and the resulting correlation between the normalized  $\beta$  value and the true plastic strain are illustrated in Figure 1 [1]. The results indicate that normalized  $\beta$  reduces with increasing plastic deformation and saturates at a specific strain value. Kim et al. proposed that the decrease of  $\beta$  might be attributed to the scaling of  $\beta$  with monopole loop length during plastic deformation, which is influenced by the initial microstructure. [1]

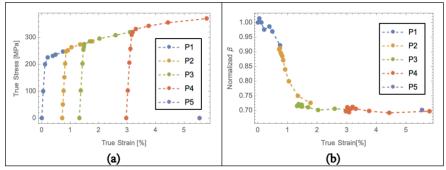


Figure 1: (a) The stress-strain diagram for SS 316L sample shows 4 different loading paths, beginning at true plastic strain levels of 0%, 0.72%, 1.3%, and 3%. NLU measurements were taken at the marked points on the diagram (more details in [1]). (b) Dependence of normalized  $\beta$  on total true strain for all loading paths. [1]

## MICROSTRUCTURE IMPACT ON B

The microstructural features, such as grain area and Kernel Average Misorientation (KAM), were evaluated using the electron backscatter diffraction (EBSD) before and after the complete deformation process. MTEX, a MATLAB toolbox, was used to calculate the features. To capture the microstructural characteristics, two EBSD images were acquired, covering an area of 800 by 500 square micrometers. The deformed surface's image was obtained from the central region of the sample, which underwent the maximum deformation, while the undeformed one was captured from the sample's grip, which had no deformation. EBSD showed a decrease in average grain area of roughly 13%, whereby the value decreased from 817 to 709  $\mu m^2$ . Figure 2 shows that the deformed surface exhibited a higher density of smaller grains compared to the undeformed surface.

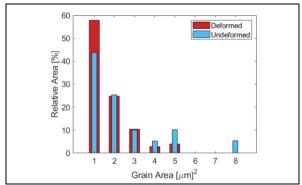


Figure 2: Histograms of the relative percentage of grain area before (light blue) and after (red) the entire deformation process.

KAM is a metric used in materials science to quantify the degree of crystallographic misorientation between neighboring grains in a polycrystalline material [5]. KAM is defined as the average misorientation angle between a given crystal orientation and its neighboring orientations, within a defined kernel or window of nearby orientations. The resulting KAM maps for both undeformed and deformed surfaces are presented in Figure 3. The high intensity red areas in the images represent the localized deviations in orientation resulting from plastic deformation on

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preferred slip planes of individual grains. The inhomogeneous distribution of red areas with high density is mainly concentrated near grain boundaries, which is attributed to the accumulation of Geometrically Necessary Dislocations (GND) in strain gradient fields caused by the geometrical constraints of the crystal lattice. Each pixel was assigned a single KAM value, and the average KAM value was determined by dividing the sum of all pixel KAM values by the total number of pixels. The average KAM value demonstrated an increase of more than twofold after deformation, rising from 0.2271 to 0.5080.

The presented EBSD results clearly demonstrate a decrease in grain area and a concurrent increase in dislocation density after plastic deformation. Based on Kubin and Mortensen's model [6], there is a linear correlation between the density of GND and the KAM value. Consequently, the KAM values indicate an increase in GND density that is more than two-fold. Given that  $\boldsymbol{\beta}^{def}$  is linearly proportional to the total dislocation density, this indicates that the decrease in  $\boldsymbol{\beta}$  is primarily influenced by the reduction in other parameters such as L or h, which have a greater impact on  $\boldsymbol{\beta}$  than the increase in dislocation density in the plastic regime. In particular, the decreasing  $\boldsymbol{\beta}$  could be due to the creation of more pinning points, which increases the probability of dislocation intersection due to the augmented dislocation density.

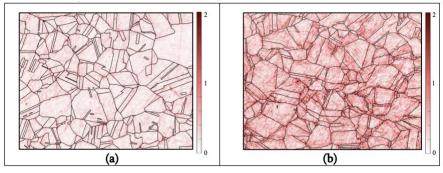


Figure 3: The KAM maps for two different surfaces, (a) undeformed and (b) deformed. Note the images were taken from different areas of the sample.

### CORRELATION BETWEEN **B** AND HARDENING RATE

The hardening rate is a measure of how much the material resists further plastic deformation as it is deformed, and is directly related to the evolution of dislocation density in a material. As a material undergoes plastic deformation, the dislocation density increases, which in turn leads to an increase in the material's hardening rate. This is because the increased dislocation density impedes dislocation motion, resulting in greater resistance to deformation [7]. The plastic zone's hardening rate and the rate of  $\beta$  decrease have been calculated and are presented in Figure 4.

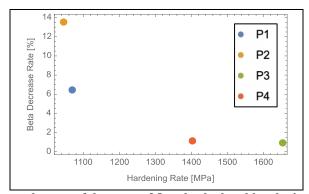


Figure 4: The correlation between the rate of decrease of  $\beta$  and calculated hardening rate during in situ NLU measurements of monotonic tensile loading in 316L stainless steel.

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Figure 4 shows the rate of decrease of  $\beta$  inversely correlates with the calculated strain hardening rate. Here, e.g. Path 3 exhibits the largest hardening rate and the lowest rate of decrease in  $\beta$ , while Path 2, which has the lowest hardening rate, shows the highest rate of decrease in  $\beta$ . This is likely due to the accelerated accumulation of dislocations and the resultant increase in dislocation density with a higher hardening rate, however systematic EBSD measurements could confirm this. Kim et al hypothesized a link between the saturation of  $\beta$  after 1.8% strain to the transition from planar slip to wavy slip [1], and this is supported by results in Fig 4. Here, a sharp rise in the hardening rate from Path 2 to Path 3, and after Path 3, the measured  $\beta$  saturated. The transition from planar slip to wavy slip is typically accompanied by an escalation in the hardening rate [8].

### **Conclusions**

This study used EBSD to analyze the correlation between the decrease in  $\beta$  during monotonic tensile loading in 316L and the initial microstructure and strain hardening rate. Results indicated that the grain area decreased and KAM analysis indicated that GND density increased over plastic strain, which suggests a corresponding increase in the total dislocation density and subsequently, a decrease in dislocation length. The correlation between  $\beta$  decrease rate and hardening rate provides a possible rationale that the saturation of  $\beta$  corresponds to the shift from planar slip to wavy slip. However, further analysis, using e.g. high resolution measurements of slip and localized plastic strain, is needed to confirm these hypotheses.

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#### REFERENCES

- (1) C. Kim and K. H. Matlack, "In situ nonlinear Rayleigh wave technique to characterize the tensile plastic deformation of stainless steel 316L," Ultrasonics, vol. 131, p. 106945, May 2023, doi: 10.1016/j.ultras.2023.106945.
- (2) Cantrell, John H., and William T. Yost. "Acoustic nonlinearity and cumulative plastic shear strain in cyclically loaded metals." Journal of Applied Physics 113.15 (2013): 153506.
- (3) J. Herrmann, J. Y. Kim, L. J. Jacobs, J. Qu, J. W. Littles, and M. F. Savage, "Assessment of material damage in a nickel-base superalloy using nonlinear Rayleigh surface waves," J Appl Phys, vol. 99, no. 12, 2006, doi: 10.1063/1.2204807.
- (4) J. H. Cantrell, "Substructural organization, dislocation plasticity and harmonic generation in cyclically stressed wavy slip metals," Proceedings of the Royal Society A: Mathematical, Physical and Engineering Sciences, vol. 460, no. 2043, pp.
- (5) L. Saraf, "Kernel Average Misorientation Confidence Index Correlation from FIB Sliced Ni-Fe-Cr alloy Surface," Microscopy and Microanalysis, vol. 17, no. S2, pp. 424–425, Jul. 2011, doi: 10.1017/s1431927611002996.
- (6) Kubin, L. P., and A. Mortensen. "Geometrically necessary dislocations and strain-gradient plasticity: a few critical issues." Scripta materialia 48.2 (2003): 119-125.
- (7) R. Shi, Z. Nie, Q. Fan, F. Wang, Y. Zhou, and X. Liu, "Correlation between dislocation-density-based strain hardening and microstructural evolution in dual phase TC6 titanium alloy," Materials Science and Engineering A, vol. 715, pp. 101–107, Feb. 2018, doi: 10.1016/j.msea.2017.12.098.
- (8) Feaugas, X., and C. Gaudin. "Ratchetting process in the stainless steel AISI 316L at 300 K: an experimental investigation." International Journal of Plasticity 20.4-5 (2004): 643-662.