

# A statistical sampling approach for measurement of fracture toughness parameters in a 4330 steel by 3-D femtosecond laser-based tomography

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## Abstract

A new approach using statistically random material volume sampling has been developed to model the variability of fracture toughnesses in steels. A recently developed femtosecond laser-based serial sectioning (FSLSS) technique was utilized to collect 3-D datasets showing the distribution of titanium nitride (TiN) phases in a 4330 high strength steel. Random volumes were sampled from widely spaced regions within the bulk steel specimen. Plastic zone sized volumes were sampled from the 3-D reconstructions to produce statistically representative volume elements containing TiN particles. Fracture toughness was calculated for 48 different plastic zone sized volumes using two different toughness models. Weibull analysis was performed to relate the modeled fracture toughness to the probability of occurrence. Variability in the fracture toughness gave a Weibull modulus of  $m = 1.4$ – $1.5$ , similar to the variability measured in 22 bulk sample specimens analyzed by Ruggieri et al. for a similar steel.

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## 1. Introduction

Most engineering materials contain precipitates or inclusions that nucleate voids that ultimately induce failure during mechanical loading [1–4]. Void nucleation by debonding or cracking of second-phase particles has been observed in numerous experimental studies on aluminum alloys and steel, where oxides, sulfides, nitrides and carbides may serve as damage initiation sites [1,5–8].

The present research focuses on 3-D characterization of second-phase particles in ultrahigh strength (UHS) steels that have been developed for applications requiring high strength, toughness and wear resistance. Applications for these materials include naval and other military armor, structural aerospace components, and impact resistant applications where brittle type failures are not acceptable.

The toughness of UHS steels is limited by the localization of plastic deformation at, and between, primary inclusion particles such as titanium nitrides (TiN) [9], and subsequent fracture or debonding that results in nucleation of microvoids [10]. Stress concentrations at microvoids developed by fractured TiN particles drive the linking up with other voided regions through ligament necking or shearing [11]. The heterogeneous stress distributions that occur due to shear localization near TiN particles reduce the fracture toughness of UHS steels [12]. Heterogeneity in microstructure, such as particle clustering, can produce local stress concentrations, void nucleation and coordinated void link-ups at lower stresses than would be expected for a volume with homogeneous microstructure. In effect, a microstructural heterogeneity such as a particle cluster acts as a larger cooperative defect. The degree of inclusion clustering and other material heterogeneities are of importance for void propagation and growth [13], while the 3-D geometry (and specifically size) of an inclusion

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affects particle fracture and its ability to nucleate voids [1]. These heterogeneities also result in variability in such properties as fracture toughness. Therefore, being able to predict where the largest flaws are, and how they can interact with each other, is fundamentally important to predicting how to process the material and modify the distribution of detrimental inclusion phases. This problem of small fractions of heterogeneously distributed microscale particles motivates the development of new tomographic techniques that can sample large material volumes. Using the recently developed femtosecond laser-based serial sectioning technique (FSLSS) [14], multiple 3-D steel microstructure volume elements have been sampled for characterization of the size and spatial distribution of TiN particles.

This paper is organized as follows. First, the new femtosecond laser-based tomography technique is introduced and is compared to existing tomography approaches. Next, the toughness models selected for demonstration of the statistical sampling approach are introduced. Details of experimental datasets are presented and their utility in predicting variability in fracture toughness is demonstrated.

### 1.1. Tomography approaches

The current state-of-the-art serial sectioning techniques, which can address large datasets at microstructural levels of detail, are painstakingly time-intensive experiments. The existing techniques vary in the amount of material that can be removed per slice, the minimum slice thicknesses, the minimum imaging resolution, the maximum volume which can be sectioned, the slice rate, and whether or not the technique is destructive or non-destructive. Here, only techniques that can address near mm<sup>3</sup> dataset volumes while capturing low volume fraction microstructure details will be discussed. The three most widely used tomographic methods are: mechanical serial sectioning, X-ray tomography and focused ion beam (FIB) serial sectioning. Mechanical sectioning is the most commonly used technique because in some instances it does not require any expensive or inaccessible hardware [15–23]. X-ray tomography systems are becoming widely available and fully automated, with various sample and source configurations implemented [24]. They have been applied to structural materials [25–27] and biomedical applications [28–30], and have also been designed for use with high energy synchrotron radiation [22,31–35]. FIB serial sectioning has the highest imaging resolution, but is limited to small sample volumes due to slow material removal rates. Despite this limitation, it has been used to address a large number of materials applications [36–45].

The newly developed FSLSS technique [14] presents a unique opportunity to access large volumes of data with fast acquisition rates (4–5 orders of magnitude faster than FIB serial sectioning). The technique generates 3-D datasets via a rapid layer-by-layer ablation process that results in limited collateral damage and permits direct light optical

imaging of ablated surfaces. Direct imaging is possible because of the low collateral damage [46,47] and minimal heat affected zone [48–50] surrounding the ablated regions. The primary parameters captured by the FSLSS technique in this study of TiN particles include: volume fraction ( $V_f$ ), mean particle size ( $\mu_{particle}$ ), average nearest neighbor distance ( $NN_{dist}$ ) and spatial distribution of the particles in three dimensions.

### 1.2. Fracture toughness models

There has been significant progress in the modeling of toughness [51,52], void growth and coalescence [53–55] in recent years. The emphasis of the present work has been to demonstrate a straightforward method for modeling variability in toughness using experimentally gathered 3-D datasets. As such, two well understood models have been used to calculate  $K_{IC}$  and  $J_{IC}$  toughness parameters, one model by Wang and Olson [56] and the other by Garrison and Moody [57]. The Garrison–Moody model relates  $K_{IC}$  to the void fraction parameter  $R_v/R_I$  (where  $R_v$  is the void radius at fracture and  $R_I$  is the initial void radius), the 3-D nearest neighbor spacing  $X_0$ , the yield stress  $\sigma_{ys}$ , and Young's modulus  $E$ :

$$K_{IC} = \sqrt{\frac{\pi}{4} E \sigma_{ys} X_0 \left( \frac{R_v}{R_I} \right)} \quad (1)$$

The Wang–Olson model relates  $J_{IC}$  normalized by the flow stress  $\sigma_0$  and the 3-D nearest neighbor distance  $X_0$  to a constant  $C_0$ , the void growth ratio  $\frac{R}{R_0}$  and a hardening exponent  $p$ , shown here:

$$\frac{J_{IC}}{\sigma_0 X_0} = C_0 \left( \frac{R}{R_0} \right)^p \quad (2)$$

The Garrison–Moody and Wang–Olson models both relate nearest neighbor spacing and the void growth ratio to toughness, but each model has been calibrated with different steel inclusion data, resulting in different hardening exponents and constants (such as  $C_0$ ), summarized in Table 1. Using these models, we must define the size of the volume element for analysis of TiN particles and incorporation into the toughness models.

### 1.3. Volume elements and the crack tip process zone

Representative volume elements (RVEs) can be collectively defined as the smallest volume that will have the

Table 1

Garrison–Moody and Wang–Olson modeling parameters. This table shows (from left to right) the type of model, the yield stress or flow stress of the steel, the void growth ratio, Young's modulus or the hardening exponent, and a fitting constant.

| Toughness model | $\sigma_{ys}$ or $\sigma_0$ | $R_v/R_I$ | $E$ or $p$ | $C_0$ |
|-----------------|-----------------------------|-----------|------------|-------|
| Garrison–Moody  | 1275 MPa                    | 4         | 200 GPa    | –     |
| Wang–Olson      | 1368 MPa                    | 4         | 2          | 0.23  |

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