Application of Chords for Quantitative Characterization of Multi-Constituent Microstructures

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Abstract A modified quantitative microstructure characterization technique has been developed based on constituent chord-length distribution (CLD). The modified technique, called the categorical CLD (CCLD), is capable of quantifying both size and volume fraction of the constituent of interest in a multi-constituent microstructure as a function of adjacent microstructure. The capability of the CCLD is demonstrated by its application to the investigation of the effect of microstructure on the stability of retained austenite (RA) during mechanical deformation in transformation-induced plasticity steels. By quantifying microstructures deformed to different levels of strains, it is found that RA is stabilized by fine grain size and adjacent bainitic ferrite.

Keywords Chord-length distribution · TRIP · Multi-constituent microstructures

Introduction

Quantification of constituent size in a microstructure is crucial to understanding the properties of some materials. For example, yield strength and toughness increase with decreasing grain size in metallic materials; fluid permeability in a porous media increases with pore size [1]; the potency of pinning particles in particle strengthening decreases with increasing size of particles [2].

While the *average size* of a constituent can be routinely determined using well-established techniques in stereology

[3], the measurement of *size distributions* is a more challenging task. The most rigorous way to determine a size distribution is to reconstruct the three-dimensional microstructure containing the constituent of interest and extract and measure the volume of each particle/grain. Manual and automatic serial sectioning systems [4, 5] and dual-beam focus ion beam (FIB) systems [6] have been developed for such tasks and have been successful in characterizing various types of materials including metals and ceramics. However, the time, cost, and effort involved in three-dimensional reconstruction are significantly more than conventional metallography. In addition, three-dimensional reconstruction requires highly reliable automatic constituent identification, which can be particularly challenging for multi-constituent microstructures.

Chord-length distribution (CLD), on the other hand, is an analog to size distribution and can be efficiently measured on two-dimensional micrographs [7]. Chords are linear segments that lie entirely inside the constituent of interest, with both ends on the boundary. Figure 1 is a schematic that shows the procedures involved to measure a CLD. An array of test probes (grid) is superimposed on the micrograph, the length of each chord is measured and then the CLD can be obtained. For isotropic microstructures, it suffices to sample with test lines along any arbitrary direction. For highly anisotropic microstructures, an orientation randomization technique such as cycloidal probes and vertical sections must be used [8].

The measured CLD is influenced by the size and morphology of the constituent of interest [9]. For example, monosized spheres will produce a distribution of chord-lengths because chords intercept the spheres at different distances from the sphere center. In general, the true size distribution or volume distribution cannot be calculated exactly from the CLD. Nonetheless, if an assumption of

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Fig. 1 Procedure of measuring CLD from two-dimensional micrographs

simple geometries such as spheres is acceptable, the constituent size distribution can be calculated from the CLD [9]. Whether the CLD or volume distribution is more relevant depends on the nature of the size–property relationship. For example, the mean intercept grain size, which is used to calculate grain boundary strengthening, is the mean of CLD. CLD is also more relevant than the constituent volume distribution in the transportation properties of porous media [7].

The effect of constituent size is more complicated in a complex multi-constituent microstructure. In transformation-induced plasticity (TRIP) steels, it is widely accepted that the mechanical stability of retained austenite (RA) increases when the size of it decreases [10–12]. In addition to grain size, the adjacent micro-constituents also affect its stability [12]. Furthermore, the RA grain size can be dependent on the microstructure that forms around it during heat treatment [11]. Therefore, a simple grain size analysis using CLD in TRIP steels does not provide a complete quantitative understanding of the combined effects of surrounding microstructure on its stability. In general, if the micro-constituent size influences the material properties of interest, one may expect the extent of the effect to be dependent on its adjacent micro-constituents. To enable proper analysis of the size effect in a multi-constituent microstructure, the categorical CLD (CCLD)

has been developed and demonstrated by its application to the investigation of the effects of microstructure on RA stability to stress/strain-induced transformation in TRIP steel.

Categorical Cord Length Distribution

The CLD analysis has been modified to characterize micro-constituent size as a function of adjacent microconstituents. To achieve that, chords are categorized according to the types of interface where each end lies. In an N -constituent microstructure, without loss of generality, assume the first constituent is of interest. The CCLD can be designated as $n^{ij}(l)$, indicating the frequency of chords of length l with one end on the interface between the first and i th constituent and the other on the interface between first and j th constituent. Assuming all possible types of interfaces are present $(N - 1)^2$ categories of chords exist in an N -constituent microstructure. One may also define “super-categories” by combining categories as needed. For example, adjacency to constituent n can be defined as any chord that has *at least one* end on the interface between first and n th constituent.

The CCLD is a versatile instrument for characterizing multi-constituents microstructures. The CCLD captures the grain size distribution of a specific constituent with

different neighboring constituents. The average constituent size can be calculated by the average of the CCLD:

$$\langle D^{ij} \rangle = \int_0^\infty n^{ij}(l) l dl \quad (1)$$

where $\langle D^{ij} \rangle$ is the average size of the constituent of category ij . The constituent volume fraction of each category can also be calculated from the CCLD:

$$V_V^{ij} = \frac{\langle D^{ij} \rangle N^{ij}}{L} \quad (2)$$

where V_V^{ij} is the volume fraction of the constituent of category ij and N^{ij} is the total number of chords measured with probes of total length of L . In Eq 2, $\langle D^{ij} \rangle N^{ij}$ is the total length of chords of category ij . Equation 2 is derived from the stereological relationship [3]:

$$V_V = \frac{l}{L} \quad (3)$$

where L is the total length of lineal probes superimposed on a micrograph, l is the total length of the probe that falls inside the constituent of interest.

In addition, interface areas can also be calculated from the CCLD:

$$\langle A_V^i \rangle = \frac{2 \left(\sum_{j \neq i} \int_0^\infty n^{ij}(l) dl + 2 \int_0^\infty n^{ii}(l) dl \right)}{L} \quad (4)$$

where $\langle A_V^i \rangle$ is the area of interface between the first and i th constituents per unit volume. Each chord of category ij contains one intercept between first and i th constituent and each chord of category ii contains two intercepts between first and i th constituent. Therefore, the term $\left(\sum_{j \neq i} \int_0^\infty n^{ij}(l) dl + 2 \int_0^\infty n^{ii}(l) dl \right)$ calculates the total number of intercepts between first and i th constituents. Equation 4 is derived from the stereological relationship [3]:

$$\langle A_V \rangle = \frac{2I}{L} \quad (5)$$

where I is the number of intercepts between the test probes and interface of interest, and L is the total length of test probes. Although average constituent size, volume fractions, and interface areas can be measured using other stereological techniques, it is more efficient to derive these properties from a single measurement of the CCLD.

Experiments

CCLD has been applied to characterize the microstructure of TRIP steels both pre- and post-deformation. A typical TRIP microstructure contains four micro-constituents: polygonal ferrite (PF), bainitic ferrite (BF), RA, and martensite. Under deformation, RA is transformed to martensite by stress-assisted or strain-induced mechanisms. The transformation rate, or the mechanical stability of RA, is crucial to the mechanical properties of TRIP steels. The mechanical stability of RA is affected by carbon content and alloying [13]. The microstructure properties, such as RA grain size and adjacent microconstituents, also affect the stability [10–12], but quantitative knowledge of their effect is lacking due to the complexity of the microstructure. However, the ability of the CCLD method to categorize constituent properties based on surrounding microstructure makes it an optimal tool for characterizing the complex TRIP microstructures. The CCLD tool is used in this study to characterize the effects of RA size and surrounding microstructure on its mechanical stability.

Materials, Tests, and Characterization

The composition of the TRIP steel is shown in Table 1 [14]. The steel was cold-rolled to a thickness of 1.2 mm. To produce a TRIP microstructure, the steels are intercritically annealed, tempered at an intermediate temperature, and quenched to room temperature. Figure 2 is a schematic showing the temperature profile during the heat treatment. Tempering temperatures and times are critical to the volume fraction and stability of RA as well as the morphology and volume fraction of BF. Since the BF in TRIP microstructures can be difficult to identify, various combinations of tempering temperatures and times were evaluated with the specific purpose of producing easily discernible bainite. In addition, the objective was to maintain the mechanical properties (tensile strength and total elongation) similar to those expected of TRIP 780 grade steel [15]. The optimal heat treatment parameters that meet both requirements were found to be intercritical annealing at 790 °C and tempering at 375 °C for 500 s.

The steel was machined to produce ASTM E-8 standard tensile specimens, and the specimens were uniaxially deformed to various strain levels at a displacement rate of 0.0423 mm/s in a screw-driven tensile frame. The ultimate tensile strength of the heat-treated TRIP steel is 832 MPa

Table 1 Chemical composition of the steel in wt.% [14]

C	Mn	Si	Ni	Cr	Mo	Ti	Nb	V	Al	N	S	P	Cu
0.19	1.59	1.63	0.02	0.03	<0.01	0.003	<0.003	<0.003	0.036	0.019	0.002	0.013	0.01

Fig. 2 Schematic of the temperature profile for the heat treatment of TRIP steels

Fig. 3 Engineering stress–strain curve of the heat-treated TRIP steel under uniaxial tension

and total elongation is 30%, as shown in Fig. 3. Metallography samples were taken from the uniformly deformed gauge sections. Standard metallography procedures were used to produce a mirror polish on the longitudinal–transverse plane with respect to the rolling direction, and the samples were then etched in 4% picric acid in ethanol. A JEOL JSM-7000F field emission scanning electron microscopy (FE-SEM), in secondary electron mode, was used to capture images the microstructure. For each strain level, five to seven images of $6000\times$ magnification ($320\text{ }\mu\text{m}^2$ for each image) were recorded for analysis.

Measurement of the CCLD

Figure 4 shows SEM micrographs of the longitudinal sections taken before deformation (Fig. 4a) and after deformation to an engineering strain of 25% (Fig. 4b, c). All micro-constituents can be characterized based on morphology and/or etching response. Martensite is identified in the

martensite/RA (MA) islands by its fine substructure (Fig. 4c). BF is distinguished from PF by its sheaf-like morphology and RA films between the sheaves (Fig. 4c).

The CCLD is measured from the SEM micrographs by a human operator. In this study, no significant anisotropy is observed in any sample and therefore lineal probes are used to sample the microstructure. To achieve a larger sampling density, measurements were performed along two perpendicular directions: longitudinal and thickness directions. A computer program was developed to facilitate the measurement. The operator marks the location and type of interface of both ends of each chord on an SEM image. The program outputs are then analyzed using Matlab. The number of chords measured in this study ranges from 480 to 855 for each strain level.

Categories and super-categories of physical relevance were defined and analyzed. The TRIP microstructure is composed of four micro-constituents, and therefore nine categories of chords exist $((N - 1)^2)$. Six (super-)categories, which are listed in Table 2, were defined for characterizing the effects of PF, BF, and martensite on RA stability. Volume fraction and average grain size of each (super-)category were calculated for each strain level using Eq 1 and 2. Interface areas of each type were calculated using Eq 4.

Results and Discussion

Evolution of Volume Fractions and Interface Areas with Deformation

Figure 5 shows the evolution of volume fraction of each (super-)category with plastic strain. The RA volume fraction decreases at different rates as a function of strain for the different (super-)categories. Comparison of the rate of decrease in volume fraction shows that RA “Adjacent to BF” or “Inside BF” transforms to martensite at a lower rate compared to RA “Adjacent to PF” or “Inside PF.” This observation is consistent with other studies that reported that film RA inside BF is more stable than the granular RA inside PF [11, 12].

The exception to the general trend of decreasing RA volume fraction with plastic strain is the austenite adjacent to martensite. The volume fraction of this super-category increases with strain and saturates at large strains ($>10\%$). The increase in volume fraction can be attributed to the way martensitic transformation occurs. Both thermal and mechanically induced martensite forms in plates or laths, which propagate through austenite grains along the habit plane, but the thickness of plates or laths are limited. As a result, the RA grains only partially transform, and the volume fraction of the partially transformed RA increases

Fig. 4 SEM micrographs of the longitudinal sections of heat-treated TRIP steels: **a** before deformation, **b** after deformation to 25% engineering strain, and **c** higher magnification image of microstructure after deformation to 25% engineering strain

Table 2 (Super-)categories of physical relevance for assessment of the effects of microstructure on RA stability

(Super-)category	Definition
All	All RA
Adjacent to BF	At least one chord-end on the BF/RA interface
Inside BF	Both chord-ends on the BF/RA interface
Adjacent to PF	At least one chord-end on the PF/RA interface
Inside PF	Both chord-ends on the BF/RA interface
Adjacent to martensite	At least one chord-end on the RA/martensite interface

with plastic strain. As plastic strain increases further, the RA available for transformation decreases and therefore, partially transformed RA increases less rapidly.

The evolution of RA interface area with strain is also dependent on the surrounding microstructure. Figure 6 shows the evolution of different types of interface areas

Fig. 5 Evolution of volume fractions of RA with different adjacent constituents under increasing engineering strain

with engineering strain. The slower decrease of RA/BF interface area relative to the RA/PF interface area is consistent with the slower decrease of volume fraction of RA in “Adjacent to BF.” The increase in RA/martensite interface area is consistent with the increase in the volume fraction of RA in “Adjacent to Martensite,” which is caused by partial transformation of RA.

Fig. 6 Evolution of interface areas of different types under increasing engineering strain

Fig. 7 Evolution of average RA grain size with different adjacent constituents under increasing engineering strain

Evolution of Grain Size with Deformation

The evolution of average RA grain size of different (super-)categories reveals the confounding effects of RA grain sizes and adjacent constituents. Figure 7 is a plot of the evolution of average RA grain size of each (super-)category with engineering strain. The initial average grain size is dependent on the (super-)category. Most notably, the average grain size inside PF is more than twice that inside BF. Second, the evolution of average grain size is also dependent on adjacent micro-constituent or (super-)category. In general, the average grain size of RA decreases with increasing engineering strain, which is consistent with the previous observations that mechanical stability of RA increases with decreasing grain size [10, 12, 16]. The “inside BF” category appears to be an exception to the trend; the average RA grain size does not change significantly with engineering strain for this category. On the other hand, RA inside BF does transform to martensite as shown in Fig. 5, albeit at a relatively slow rate. The initial

Fig. 8 **a** Size distributions of RA inside BF and PF and **b** evolution of size distribution of RA inside BF

size distribution of RA “inside BF,” as shown in Fig. 8(a), is relatively narrow compared to the initial size distribution of RA “inside PF,” which can be attributed to the difference in RA morphology: film morphology inside BF and granular morphology inside PF. Longer chords compose a smaller fraction of the total chords in the narrower size distribution of RA “inside BF” than “inside PF.” The evolution of size distribution of the “Inside BF” category with strain shows that number of longer chords still decreases faster than shorter chords (Fig. 8b), but since the fraction of long chords is low for RA inside BF, the decrease in average grain size is not substantial. Based on this analysis, the higher stability of RA inside BF than that inside PF is at least partly accounted for by the finer RA grain size inside BF.

Assessment of the Effects of RA Grain Size and Surrounding Microstructure on RA Stability

The CCLD analysis shows that RA stability depends on both RA grain size and surrounding microstructure. The decrease of average RA grain size in all (super-)categories, though less pronounced for RA inside BF, shows that larger RA grains are more likely to undergo martensitic transformation. The effects of surrounding microstructure are shown by the significant differences in rate of

transformation for different (super-)categories. RA inside or in adjacent to BF is significantly more stable than RA inside or in adjacent to PF (Figure 5). The stabilizing effect of BF may be partially attributed to the finer RA grain size inside BF and adjacent to BF (Fig. 7). Yet it is also possible that RA might be stabilized by the surrounding BF itself through carbon enrichment [12] or strain-partitioning [11, 17]. Further investigation, including modeling of the CCLD data and other characterization techniques, is needed to clarify the mechanism of BF stabilization.

The CCLD analysis also reveals extensive partial martensitic transformation. The area of interface between martensite and RA increases with engineering strain and so does the volume fraction of RA adjacent to martensite. Experimental studies have found evidence that martensite stabilizes the surrounding RA [11, 12], and the stabilizing effect was attributed to the stress-shielding effect of the martensite, which has a relatively high strength compared to other constituents in the microstructure [17]. The increasing volume fraction of RA near martensite and interface area between RA and martensite with strain found in this study possibly supports the stabilizing effect of martensite. However, it could merely be due to the tendency of RA to partially transform rather than fully transform, which would also increase RA volume fraction near martensite by definition.

Summary and Conclusions

A new microstructure quantification instrument, the CCLD, has been developed to characterize the constituent of interest in multi-constituent microstructures. CCLD captures both size and adjacent microstructure for the constituent of interest. Apart from size distributions, various other microstructure attributes can be calculated from the CCLD including volume fraction and average size of constituents with different adjacent microstructures. In addition, interface areas between the constituent of interest and other constituents can also be calculated from CCLD.

CCLD has been applied to assess the effects of microstructure on the mechanical stability of RA in TRIP steels under uniaxial stress. (Super-)categories of physical relevance have been defined and analyzed. RA inside BF transforms much slower than that inside PF as shown by the volume fraction and interface area measurements. The slower transformation is at least partly attributed to the finer RA grain size inside BF. The possible stabilizing effect of martensite is supported by the increase in RA volume fraction near martensite with strain, though this could also be an artifact of the partial transformation of RA to martensite.

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