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Digital image analysis to quantify carbide networks in ultrahigh carbon steels

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ABSTRACT

A method has been developed and demonstrated to quantify the degree of carbide network connectivity in ultrahigh carbon steels through digital image processing and analysis of experimental micrographs. It was shown that the network connectivity and carbon content can be correlated to toughness for various ultrahigh carbon steel specimens. The image analysis approach first involved segmenting the carbide network and pearlite matrix into binary contrast representations via a grayscale intensity thresholding operation. Next, the carbide network pixels were skeletonized and parceled into braches and nodes, allowing the determination of a connectivity index for the carbide network. Intermediate image processing steps to remove noise and fill voids in the network are also detailed. The connectivity indexes of scanning electron micrographs were consistent in both secondary and backscattered electron imaging modes, as well as across two different (50× and 100×) magnifications. Results from ultrahigh carbon steels reported here along with other results from the literature generally showed lower connectivity indexes correlated with higher Charpy impact energy (toughness). A deviation from this trend was observed at higher connectivity indexes, consistent with a percolation threshold for crack propagation across the carbide network.

1. Introduction

Ultrahigh carbon steels (UHCS) have been used for many years in various applications demanding high strength and wear resistance. UHC steels have carbon contents from 1 to 2.1 wt%, in excess of the 0.76 wt% Fe—C eutectoid composition (Fig. 1), and as a result there is precipitation of proeutectoid cementite (Fe_3C) during cooling from casting and/or heat treatment processes. The hard and brittle cementite contributes to the high hardness and wear resistance of UHCS, which is desirable for applications involving the cutting or shaping of other metals. UHCS have been used in rolling mills as far back as 1913 [1] and are also commonly used in some tool steels. There has been interest over the last few decades in using UHCS for other applications where traditionally lower carbon content steels have been utilized, such as sheet and automotive steels [2,3]. Recent UHCS research has focused on improving ductility/toughness by modifying existing manufacturing processes to improve the resulting microstructure.

A major goal in UHCS processing is modification of the network of brittle carbides (primarily cementite) distributed throughout the microstructure. Networks of proeutectoid cementite form in UHCS alloy during cooling, primarily due to precipitation in the $\gamma + Fe_3C$ phase field (depending on the steel composition and cooling rate, a small amount of eutectic solidification may also occur). The carbide network provides crack initiation sites and pathways for crack propagation that reduce UHCS toughness/ductility [4,5] by allowing cracks to circumvent the ductile matrix. Fig. 2 shows an example of a crack propagating preferentially within the carbide network. Modification of the network, namely breaking it up, has the potential to reduce pathways for crack propagation and force cracks to travel through the matrix, thereby increasing toughness.

It is possible to process UHCS so as to completely eliminate the network or significantly reduce its connectivity. In this case, toughness may depend on microstructural parameters such as the type of eutectoid transformation products and the prior austenite spacing [6]. Successful approaches have included thermomechanical processing [3,7] and chemistry modification by small additions of rare earths or niobium [8–10]; both approaches have resulted in greatly improved mechanical properties measured at room temperature. These relatively new manufacturing methods have not yet achieved widespread adoption, possibly because of additional processing difficulty and cost. Another possible processing alternative is using heat treatments alone [11–13]. In all cases, these studies utilize optical microscopy (OM) or scanning electron microscopy (SEM) to qualitatively evaluate the carbide network connectivity in relation to processing conditions.

While there is interest in breaking up the network in UHCS, there currently exists no metric for quantifying the degree of network break-up beyond calculating the network volume fraction. Establishing a standard method for quantifying connectivity of carbide networks

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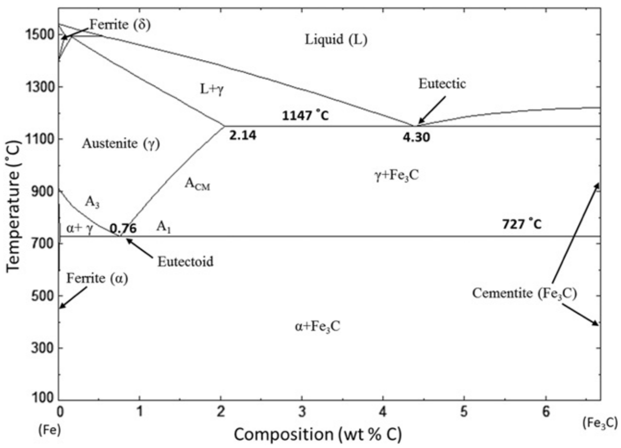

Fig. 1. Fe—C phase diagram indicating temperature/composition regions of interest in UHCS. [This phase diagram was generated using the Factsage® thermochemical software [32]].

would improve evaluation and comparison of proposed network modification processes.

The concept of network connectivity analysis is already well established in various fields. Percolation theory [14–16] provides an essential framework for describing networks in terms of connections between points/nodes on a grid. Transportation science [17,18] defines a "connectivity index" as a ratio of streets to intersections and cul-de-sacs. Tortuosity in porous substances [19], is found by quantifying the ratio of distance traveled along a convoluted path to the direct point to point distance. Medical science describes a connectivity index in cancellous human bone [20,21] in terms of the maximal number of connections needed to be broken to separate one network into two. For the case of UHCS steels, carbide networks provide a "pathway" for crack propagation. Thus in this work, the transportation science metric for network quantification is leveraged to quantify carbide networks in UHCS.

The purpose of this paper is to propose, demonstrate and standardize a potential method of analyzing OM/SEM images of UHCS microstructures and quantifying the network connectivity. In this study, commercial UHCS roll mill specimens were imaged and analyzed to produce quantified metrics for carbide network connectivity. These measurements were compared to experimental toughness measurements performed in this study as well as literature values. Details of the digital image analysis approach are described.

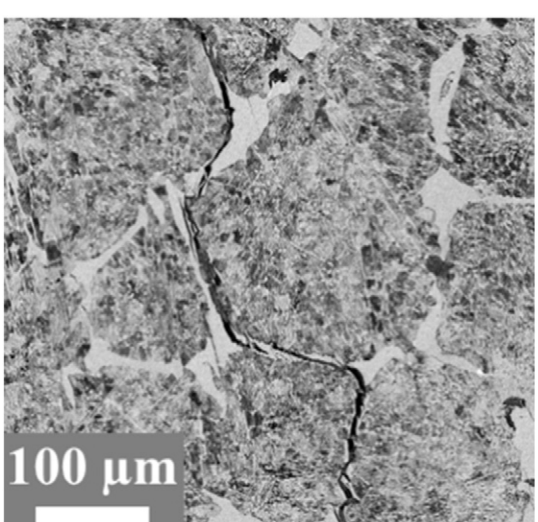

Fig. 2. Scanning electron micrograph of a crack propagating through the cementite network in an UHCS (BSE imaging mode).

2. Digital image analysis methods

This section describes our approach for quantifying network connectivity as applied for as-cast and heat treated UHCS specimens. ImageJ [22], an image processing software program in the public domain (http://rsb.info.nih.gov/ij/index.html), was utilized for network connectivity quantification. Network analysis requires deconstructing contrast features in micrographs into a network composed of links and nodes in space, illustrated in Fig. 3. Nodes may be endpoints, linked to exactly one other node, or internal junctions connected through links to two or more other nodes. The ratio of links to nodes, known as the connectivity index [18], is a useful parameter in evaluating such a simplified network. Information such as the probability of each node being an endpoint or junction, and the average link length, is also of interest.

The critical image processing step allowing these parameters to be extracted is skeletonization, an operation that thins the network to a single-pixel width so that nodes and links (Fig. 3) can be identified. The following sections describe the image processing steps developed to extract connectivity index from digital micrographs of UHCS materials: thresholding for phase segmentation, particle size filtering for artifact removal, outlier removal for network void removal, and skeletonization for network quantification.

2.1. Thresholding for phase segmentation

The initial objective was to digitally select and isolate the carbide network from other phases present in a typical UHCS micrograph. The first step was to ensure clear image contrast between constituent phases in the steel microstructure under OM/SEM imaging. For UHCS containing cementite and ferrite, the common etchant Nital (dilute nitric acid in ethanol or methanol) was useful towards this aim. Nital preferentially etched the ferrite, revealing proeutectoid cementite as well as cementite lamellae in the pearlite. The resulting rougher eutectoid pearlite morphology produced a higher local secondary electron (SE) yield than the smoother morphology network proeutectoid cementite. Thus the proeutectoid cementite network exhibited lower grayscale intensity in the SE image. Fig. 4(a) shows the typical appearance of a Nital-etched pearlitic UHCS microstructure as imaged by SEM. User-controlled gain settings ensured the proeutectoid cementite was dark and distinct from the lighter pearlitic matrix. There was some grayscale variation in the pearlite matrix due to spatial lath orientation variations. Ultimately, any suitable etchant process for the analyzed steel should yield such

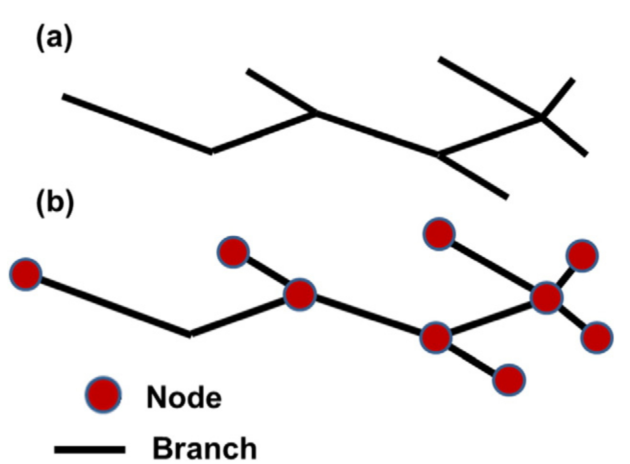

Fig. 3. Schematic illustration of (a) a simple network and (b) the same network indicating branches and nodes.