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Automated Optical Serial Sectioning Analysis of Phases in a Medium Carbon Steel

Bryan Turner, Satya Ganti, Bill Davis and Veeraraghavan Sundar

Abstract This work presents a three dimensional microstructural analysis of medium carbon steels, based on serial sectioning using optical microscopy. The microstructural phases considered here are ferrite and pearlite. Typically conventional 2D microscopy is used to analyze these phases (ASTM E112-13 in Standard test methods for determining average grain size. ASTM International, West Conshohocken, PA, 2013), with eddy current (Khan et al. in J Mater Process Technol 200(1):316–318, 2008) or ultrasonic methods (Araújo Freitas et al. in Mater Sci Eng A 527(16):4431–4437, 2010) as alternatives. Medium carbon steel samples were heat treated in a furnace and either slowly cooled in the furnace, or cooled at higher rates. This resulted in different pearlitic microstructures in the samples. The analysis was conducted with Robo-Met.3D®, a fully automated metallography system for serial sectioning. Based on optical microscopy, volume cubes of hundreds of microns on edge were imaged and reconstructed to conduct grain size and phase volume analysis. The system proved capable of collecting image data with high fidelity, and controlled slice thickness in an automated manner. The resultant dataset was useful in visualization and analysis of pearlite and ferrite phases in steel samples.

Keywords Serial sectioning · Grain size analysis · Microstructure
Phase analysis · Medium carbon steels · Metallography · Robo-Met

Materials and Serial Sectioning Methods

Robo-Met.3D is a fully automated metallography system that generates two dimensional optical microstructural data for three dimensional reconstructions and analysis. Common applications of Robo-Met.3D include studying additively

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manufactured components [4], analysis thermal barrier coatings [5], and fiber orientation effects in ceramic matrix composites [6].

- Material Composition and State

An off-the-shelf, un-heat treated, cold drawn 1045 medium carbon steel (1045 MCS) sample was chosen (as received). Samples were excised from rod, and were conventionally mounted in metallographic mounts ($\sim$38 mm diameter $\times$ 25 mm height) for automated serial sectioning. In this study, two different heat treatments were performed on the samples. The first heat-treated sample was heated to approximately 830 °C and furnace cooled (annealed) for about 12 h until room temperature was attained. The second heat treated sample was heat treated to approximately 830 °C, removed from the furnace and quickly dropped into water (quenched). The three samples were then serial sectioned for microstructural analyses.

- Grinding and Polishing for Serial Sectioning

A typical automated grinding and polishing program for materials of this type involves diamond suspensions of varying sizes—6, 3, 0.25 μ, and finishing with 0.05 μ colloidal silica. Polishing times may be varied automatically to achieve varying rates of material removal. The samples were etched with 2% Nital reagent to enhance contrast. Intermediate and final cleaning steps with water and ethanol are programmed in to keep cross contamination to a minimum.

- Image Acquisition and Processing

Optical images were automatically acquired with the Zeiss Axiovert microscope built into the Robo-Met.3D system. The 2D image tiles from each layer were stitched into montages, registered with the images from the next layer using Fiji software. A mosaic or montage of 2 $\times$ 2 tiles of 500$\times$ images was collated, with a resolution of 0.21 μ along the X and Y axes. A total of 50 slices, or sections were collected. Acquisition parameters are shown in Table 1.

For 2D analysis, single 2D image was segmented using WEKA segmentation filter in Fiji to identify the phases (pearlite—dark, ferrite-bright and martensite—light grey). For 3D analysis, the raw 2D images were stitched and aligned for accurate post-processing. The loaded z-stack of 50 slices was processed with subsampling along Y direction and full resolution along X and Z directions. A 3D isosurface was created without any filtering to extract the volume distribution of the phases. Thresholding was performed on pixel values as for the 2D analyses. A combination of Fiji and ImagePro software suites was used in this analysis.

Table 1 Serial sectioning data acquisition parameters

1045 MCS	Overall magnification	Resolution in x − y (μ)	Sectioning rate (μ/section)	No. of sections analyzed	Dimensions analyzed (μ)
As-received	500×	0.21	1.8	50	451 × 28 × 92
Annealed	500×	0.21	4.6	50	451 × 282 × 230
Quenched	500×	0.21	3.2	50	460 × 290 × 160

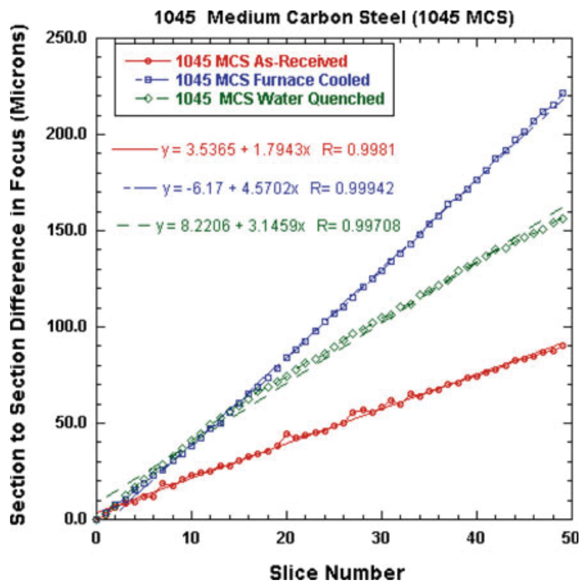

Fig. 1 Material removal plot for 1045 MCS as-received, annealed and quenched samples

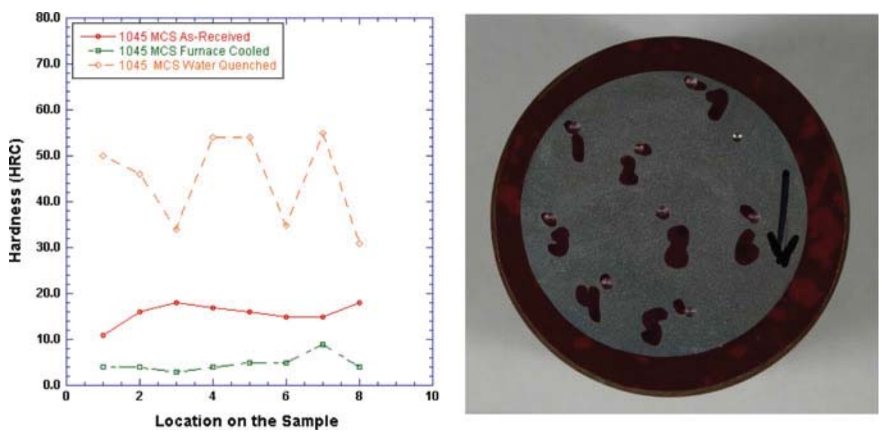

Fig. 2 Rockwell hardness C measurements for 1045 MCS as-received, furnace cooled and water quenched samples (left) and location of eight measurements on the sample (right)

- Linearity of Material Removal Rate

Robo-Met.3D uses z-focus or z-height of the optical microscope for accurate material removal measurements between sections, rather than fiducial marks. As shown in in Fig. 1, a highly linear material removal rate of between 1.8 and 4.6 μ was maintained for all samples.

- Material Hardness

As expected, HRC hardness values were lowest for annealed sample (HRC 4.1), followed by the as-received (HRC 16.4) and water quenched samples (HRC 44.9) as shown in Fig. 2.

Analysis Results

The 2D and 3D image sets (Figs. 3, 4 and 5) were analyzed for the relative fraction of pearlite and ferrite phases. The 3D data may be considered more representative as it minimizes section-dependent variation [7].

Based on the heat-treatment conditions (especially cooling rate), we have observed different percentages of the three phases as shown in Table 2.

The as-received sample showed 71.0% pearlite and 29.0% ferrite, and the annealed sample showed 70.1% pearlite and 29.9% ferrite which approximate with the ideal values of 75.0% pearlite and 25.0% ferrite. Incomplete conversion to about 28% martensite was observed in the quenched material as agitation was not used in the quenching bath. The quenched sample was predominantly pearlitic.

We have also performed preliminary grain size analysis on un-heat-treated sample as shown in Fig. 6. Image-pro premier-3D software is equipped to perform semi-automated (interactive) grain analysis per ASTM E112 standard.

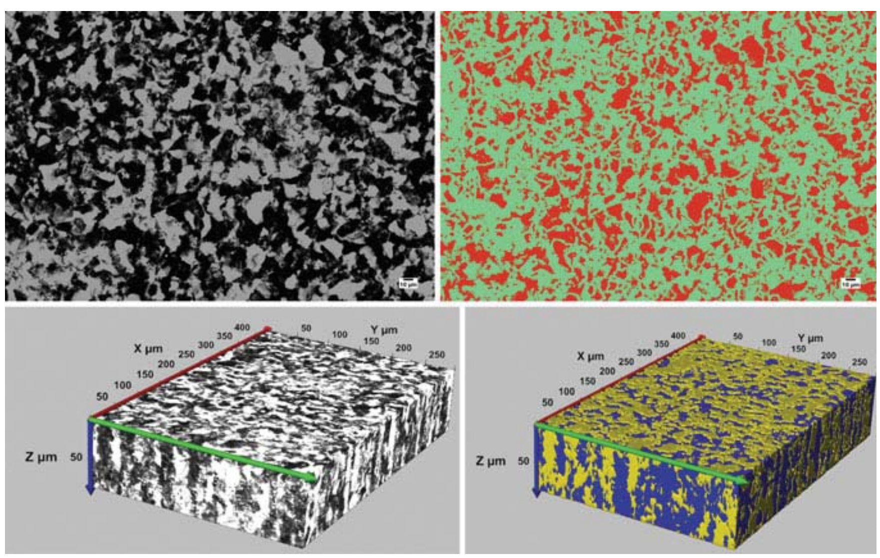

Fig. 3 1045 as-received steel—Top: 2D raw image (left) and WEKA segmented image: pearlite-green, and ferrite-red (right); bottom: 3D image (2D raw images aligned stack of 50 slices) (left) and segmented 3D image (pearlite-yellow; ferrite-blue) (right)

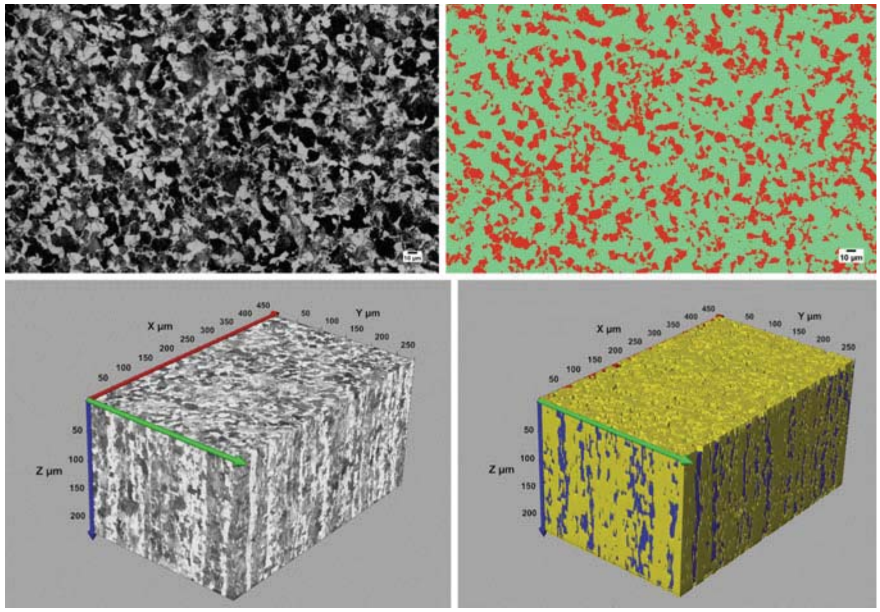

Fig. 4 1045 annealed steel—top: 2D raw image (left) and WEKA segmented image: pearlite-green, and ferrite-red (right); bottom: 3D image (2D raw images aligned stack of 50 slices) (left) and segmented 3D image (pearlite-yellow; ferrite-blue) (right)

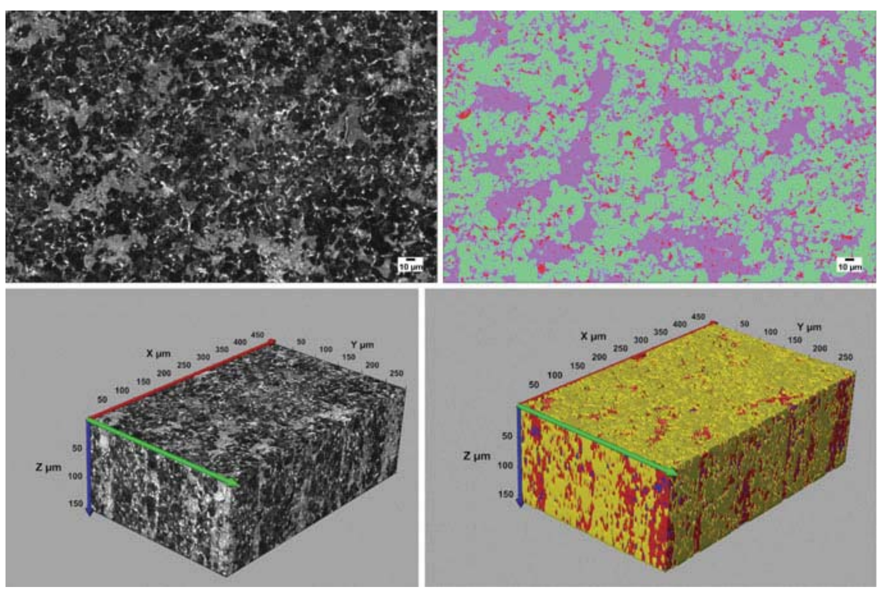

Fig. 5 1045 water quenched steel—top: 2D raw image (left) and WEKA segmented image: pearlite-green, martensite-purple and ferrite-red (right); bottom: 3D image (2D raw images aligned stack of 50 slices) (left) and segmented 3D image (pearlite-yellow; martensite-red; ferrite-blue) (right)

Table 2 Area (2D) and volume(3D) percentages of phases

Material	Phase volume fraction (%)		
1045 medium carbon steel	Pearlite	Ferrite	Martensite
As received	71	29	–
Annealed	70.1	29.9	–
Water quenched	66.2	6.02	27.8

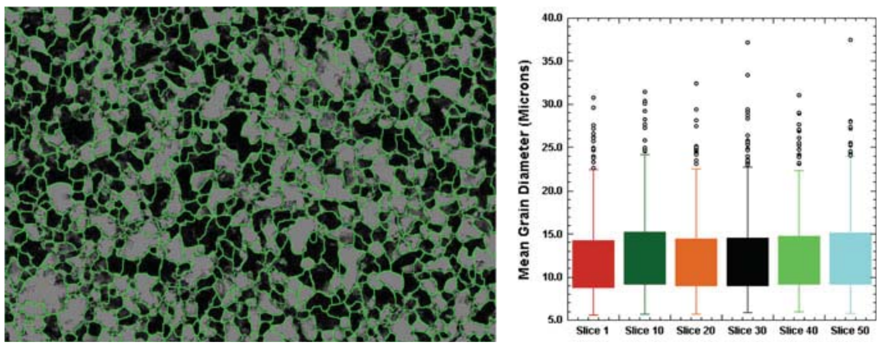

Fig. 6 Grain network outline overlaid on raw image (left); 2D statistical distribution of mean grain diameter (center)

Conclusion

The study successfully demonstrates the use of automated serial sectioning for phase analyses and interactive grain size analyses. The RoboMet.3D®system proved capable of collecting image data with high fidelity, and controlled slice thickness in an automated manner. Experimental results have shown the effect of different heat treatment conditions on the hardness and microstructure of steel. The resultant datasets were useful in visualization and analysis of pearlite, ferrite and martensitic phases in 1045 steel samples.

Further Information

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