TEST REPORT

Project Name:
TTSL - Brake Drums

Testing:
1) Molecular Structure
2) Chemical Analysis

Items Tested:
1) Molecular structure / chemical analysis prepared from sample brake drums

Goal of Testing:
1) Characterize alterations to material molecular structure resulting from Thermal Cycling treatment process

For questions regarding this test report, please contact:

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1. Executive Summary

The aim of this report is to perform the following:

1. Determine microstructural and chemical change to materials resulting from application of the Thermal Cycling treatment process to brake drums.
2. Identify key transformation mechanisms associate with Thermal Cycling.

In summary, Scanning Electron Microscopy (SEM), EDAX X-ray intensity mapping, chemical analysis and metallographic examination all indicate a microstructural transformation to the brake drum ferrous material resulting from the Thermal Cycling treatment.

The brake drums were determined to consist of gray cast iron. The graphitic flakes present in the iron matrix were observed to have a finer structure and more even distribution for treated material vs the untreated material. As well, silicon and manganese inclusions within the matrix experience an increase in size and frequency. The treatment can be seen to have encouraged diffusion of carbon from the graphite flakes and into the inclusions. It also induces diffusion of iron into the carbide flakes.

The Thermal Cycling treatment therefore has enhanced precipitation and diffusion reactions within the material and at the grain boundaries. These transformation mechanisms will act to relieve residual stresses within the microstructure and enhance strength and wear properties.

This analysis identifies a key mechanism of microstructural change that occurs due to application of the Thermal Cycling treatment. Understanding this mechanism is critical to evaluating an optimized approach to improving treated material properties and ultimately, product performance, through the application of Thermal Cycling.

It is recommended that further testing be conducted to fully understand the transformation mechanism associated with Thermal Cycling, and the benefits the treatment bestows upon the mechanical properties of the treated materials. Recommended testing includes metallographic and chemical composition investigation of a broader range of materials, and tensile testing, hardness testing, impact toughness testing and wear resistance / abrasion testing of these materials.

2. Introduction

Thermal Cycling is an innovative and cost effective process of enhancing the mechanical properties of many materials commonly used in commercial and industrial technologies.

Testing was performed on brake drums that were treated with the Thermal Cycling process (treated) and standard brake drums that did not reveive the treatment (untreated). The goal of the testing was to determine the microstructural and chemical change to the brake drum material resulting from application of the Thermal Cycling treatment process to brake drums. In particular, the testing sought to identify key transformation mechanisms associate with Thermal Cycling.

The testing conducted for this project consisted of metallographic characterization and SEM / EDAX chemical compositional mapping. These testing methods evaluate the molecular transformation to the metallic atomic structure resulting from application of the Thermal Cycling treatment process.

Test results of the material from the two brake drums were compared to evaluate the relative change resulting from the treatment process. The ferrous phases present in the steels, their respective ratio's, and the chemical composition and distribution of the various phases present in the steels were determined and compared.

The two brake drums provided for testing are shown below.

drum, treated *drum, untreated*

3. Methods

Chemical Mapping / Analysis

Samples of the material were removed from the drums by cutting and the cross-sectioned surfaces were ground with grinding medium from 60 grit down to 1000 grit, sequentially. The ground surface was then polished with 0.3 µm and then 0.05 µm alumina to generate a scratch free mirror finish. Specimen surfaces were examined by Scanning Electron Microscopy. SEM specimens were also examined with EDAX to generate a chemical map and analysis of the surface.

EDAX involves analyzing the x-rays produced by the interaction of the electron beam with the specimen and provides a detailed analysis of the chemical composition of the material. As well, EDAX is one of the few methods available that also provides a map of the elemental distribution in the material.

Metallography

The molecular structure of the drums was determined by standard metallographic techniques. Samples of the material were removed from the drums by cutting and the cross-sectioned surfaces were ground with grinding medium from 60 grit down to 1000 grit, sequentially. The ground surface was then polished with 0.3 µm and then 0.05 µm alumina to generate a scratch free mirror finish. Specimens were etched with 5% Nital (5% nitric acid by volume, dissolved in methanol) for 15 seconds. Specimen surfaces were then observed with a optical microscope.

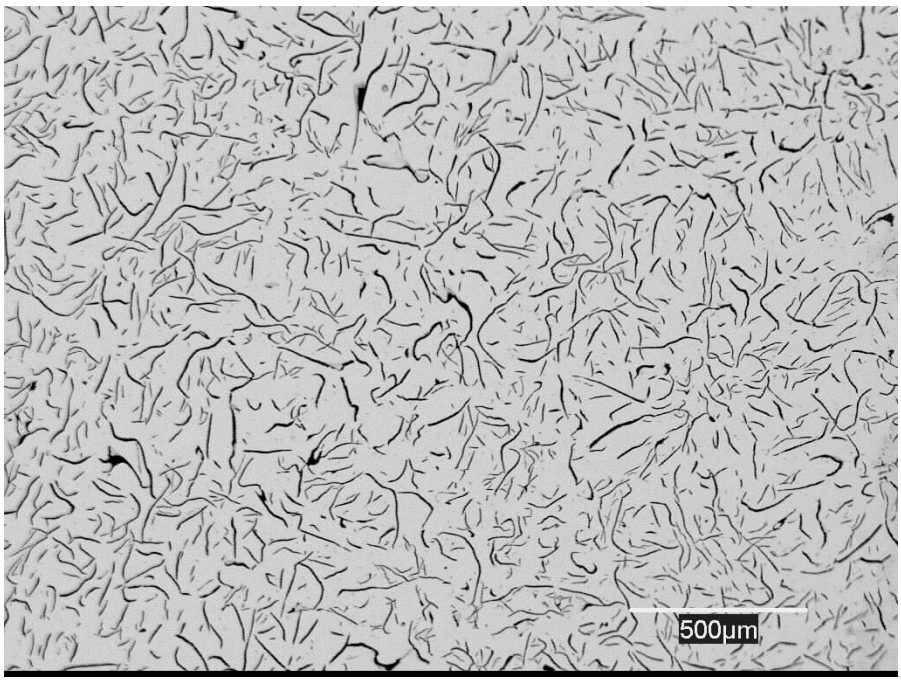
4. Results

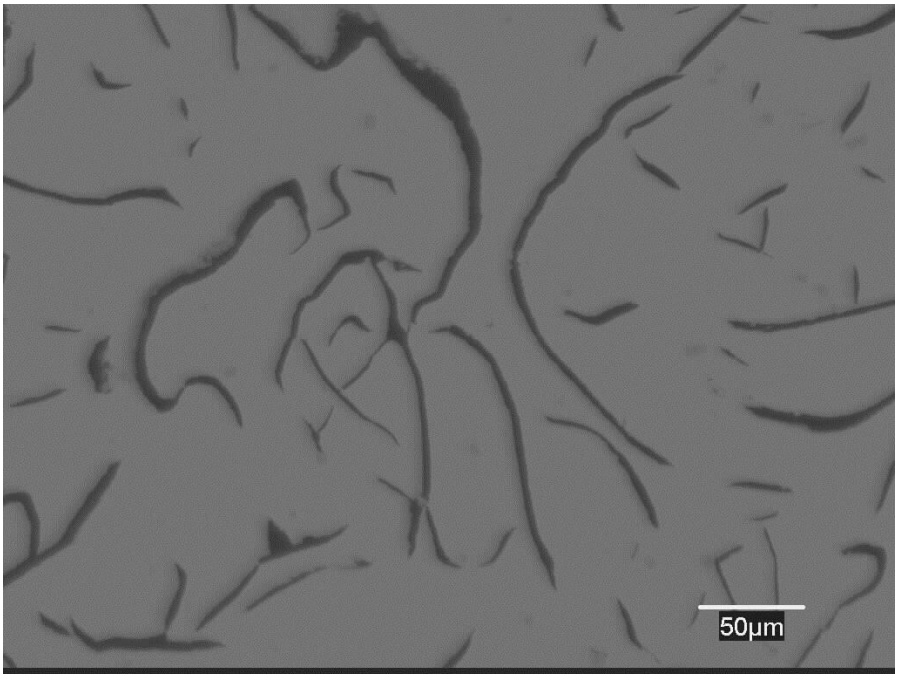
Chemical Analysis

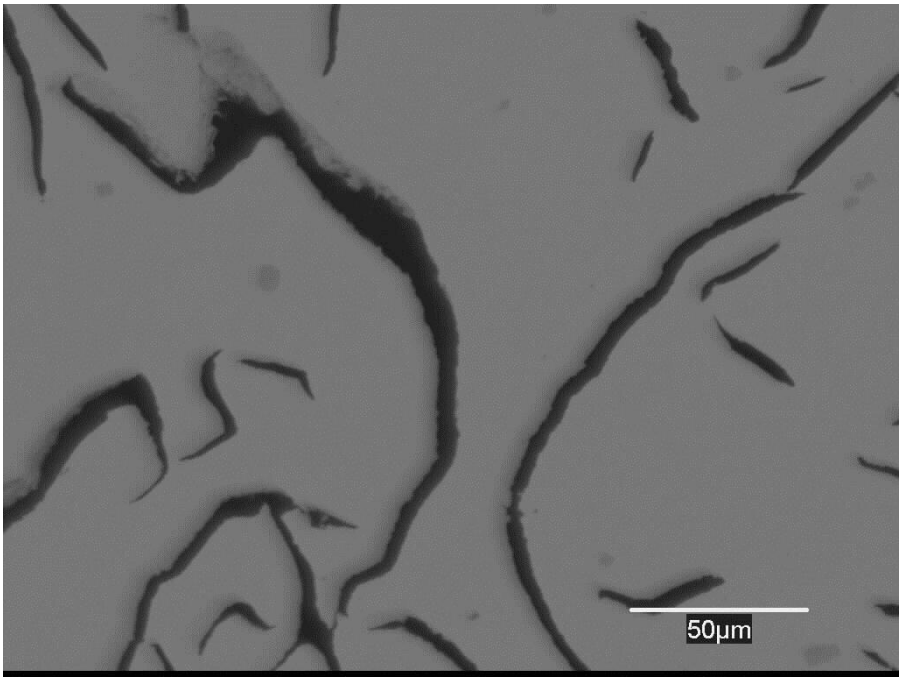
The samples were examined by SEM/EDAX using a LEO 440 SEM equipped with a Quartz XOne EDAX spectrometer. For this work a 10 kV electron beam voltage was used to obtain SEM images and EDAX spectra and X-ray intensity element maps from both samples.

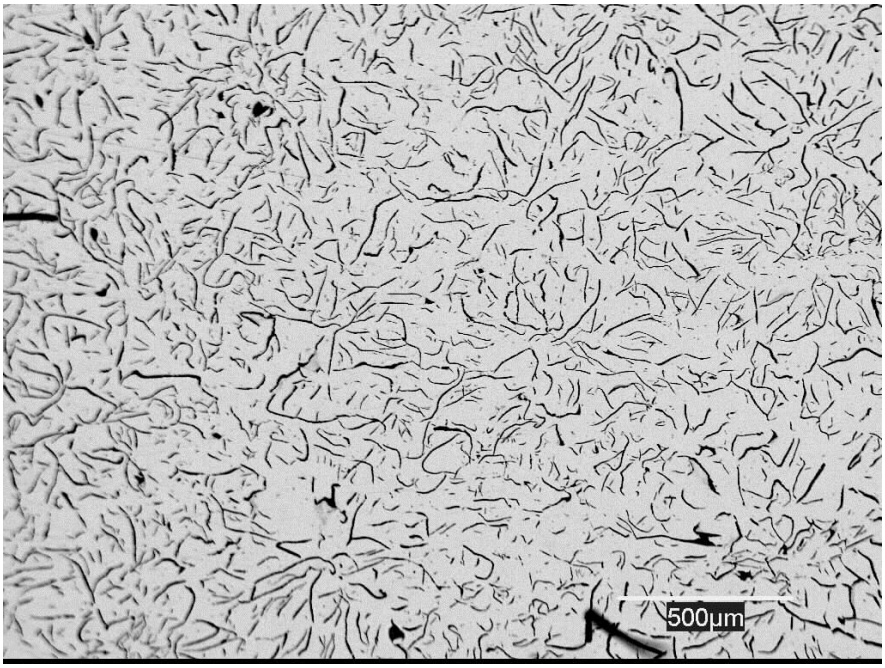
SEM micrographs of the treated and untreated materials are shown below. The material is a gray graphitic cast iron. The graphite flakes indicative of a gray cast iron are clearly visible in the SEM images.

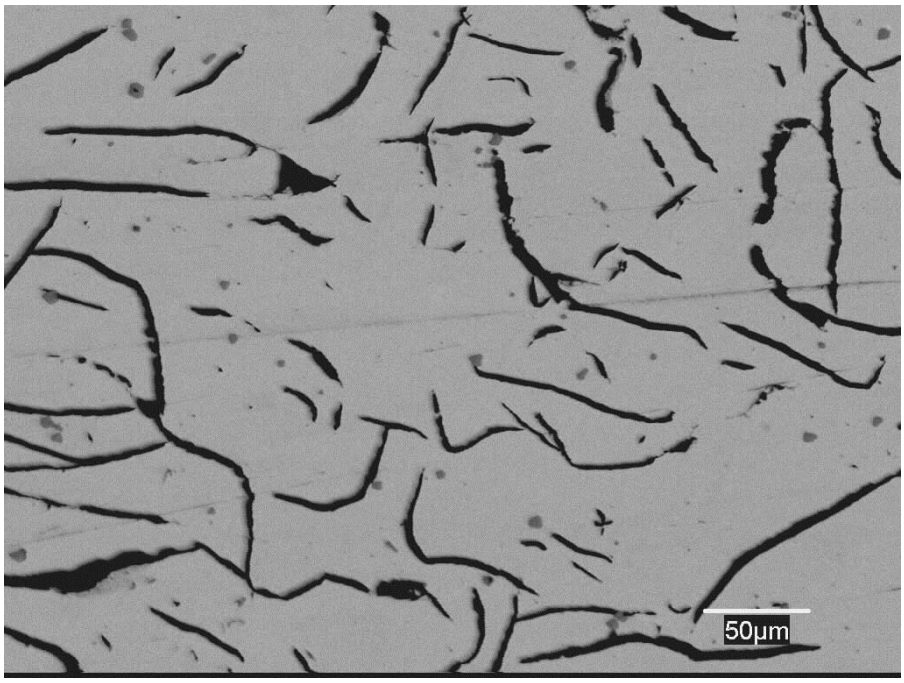
The following SEM micrographs show at increasing magnification first the untreated brake drum material (3 images) and then the treated brake drum material (following 3 images). Graphite flakes can be clearly seen as the black patterns, while the white material is the iron matrix. These images are typical for a standard gray cast iron.

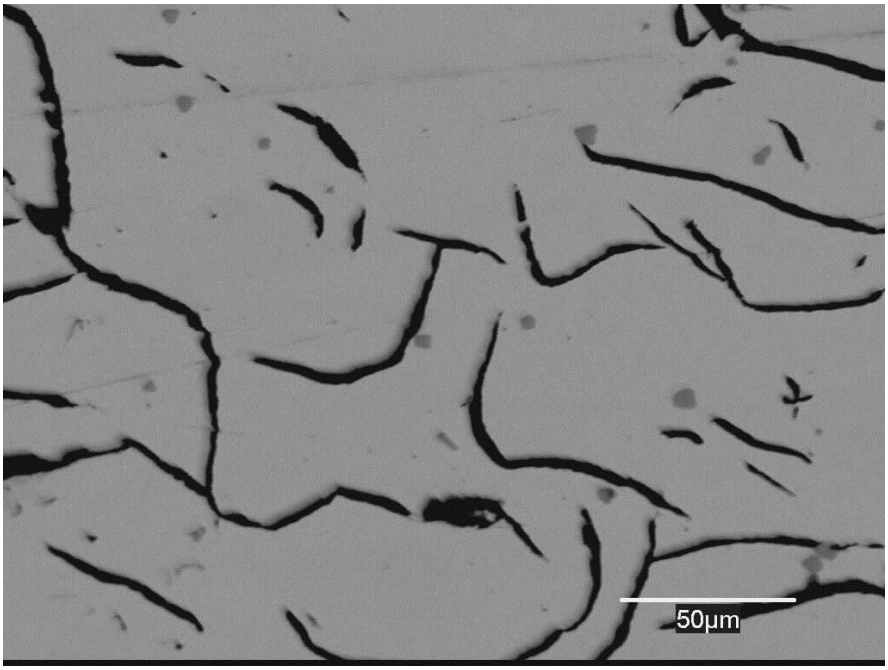






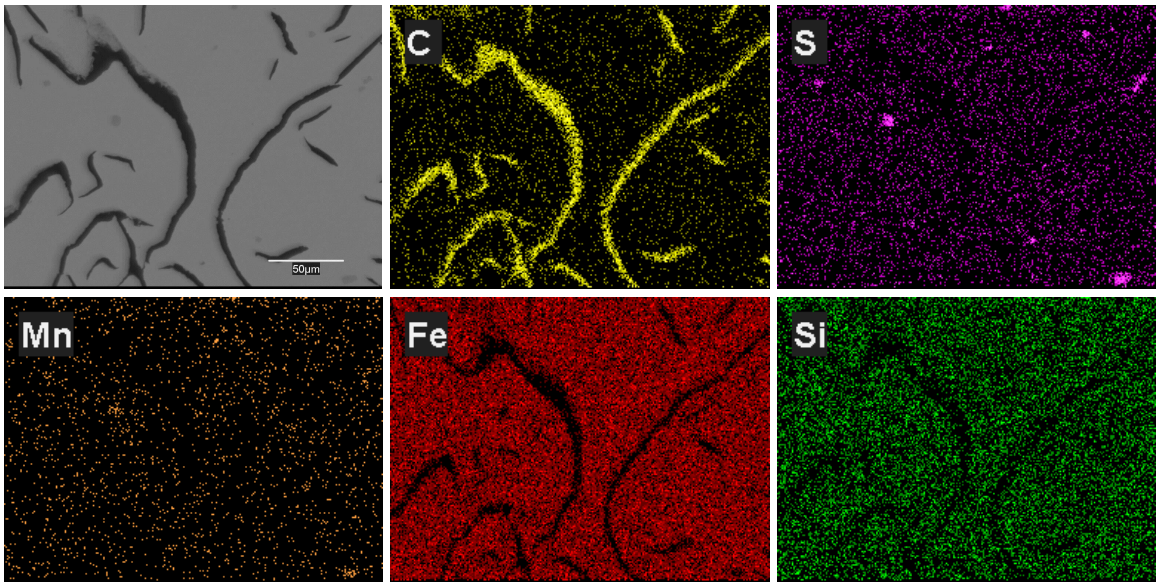




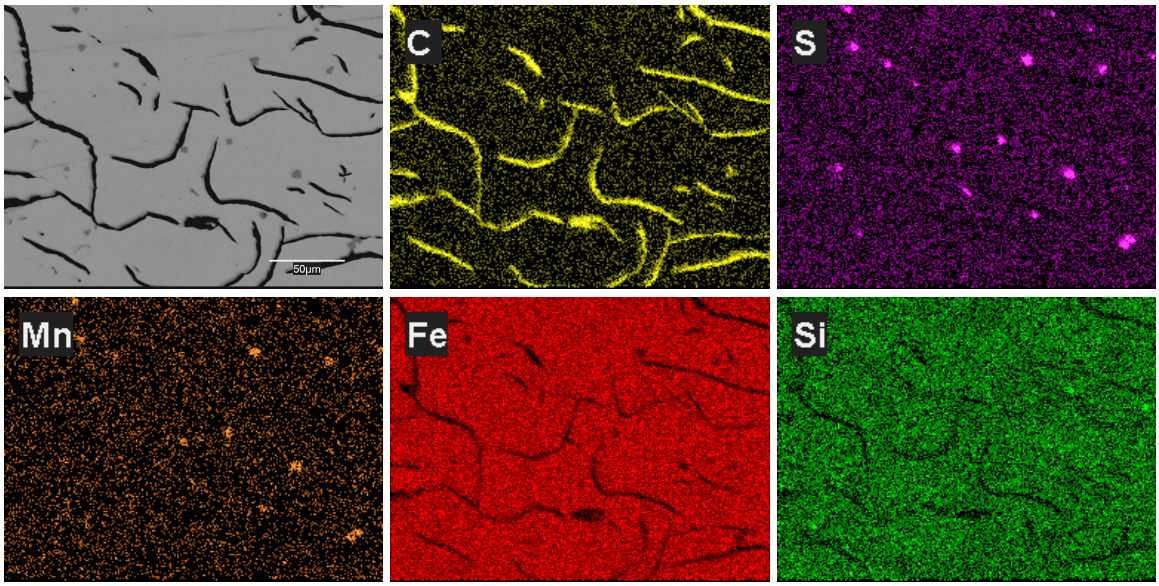


Note that the reference calibration scale at the lower right hand of the image indicates the relative size of the examination area and of the various structures present. The black lines are graphitic flakes, occurring along the metallic grain boundaries. The pale gray circular regions are suspected inclusions. Their composition will be determined by the EDAX analysis.

EDAX X-ray intensity maps are shown for the materials below.



untreated



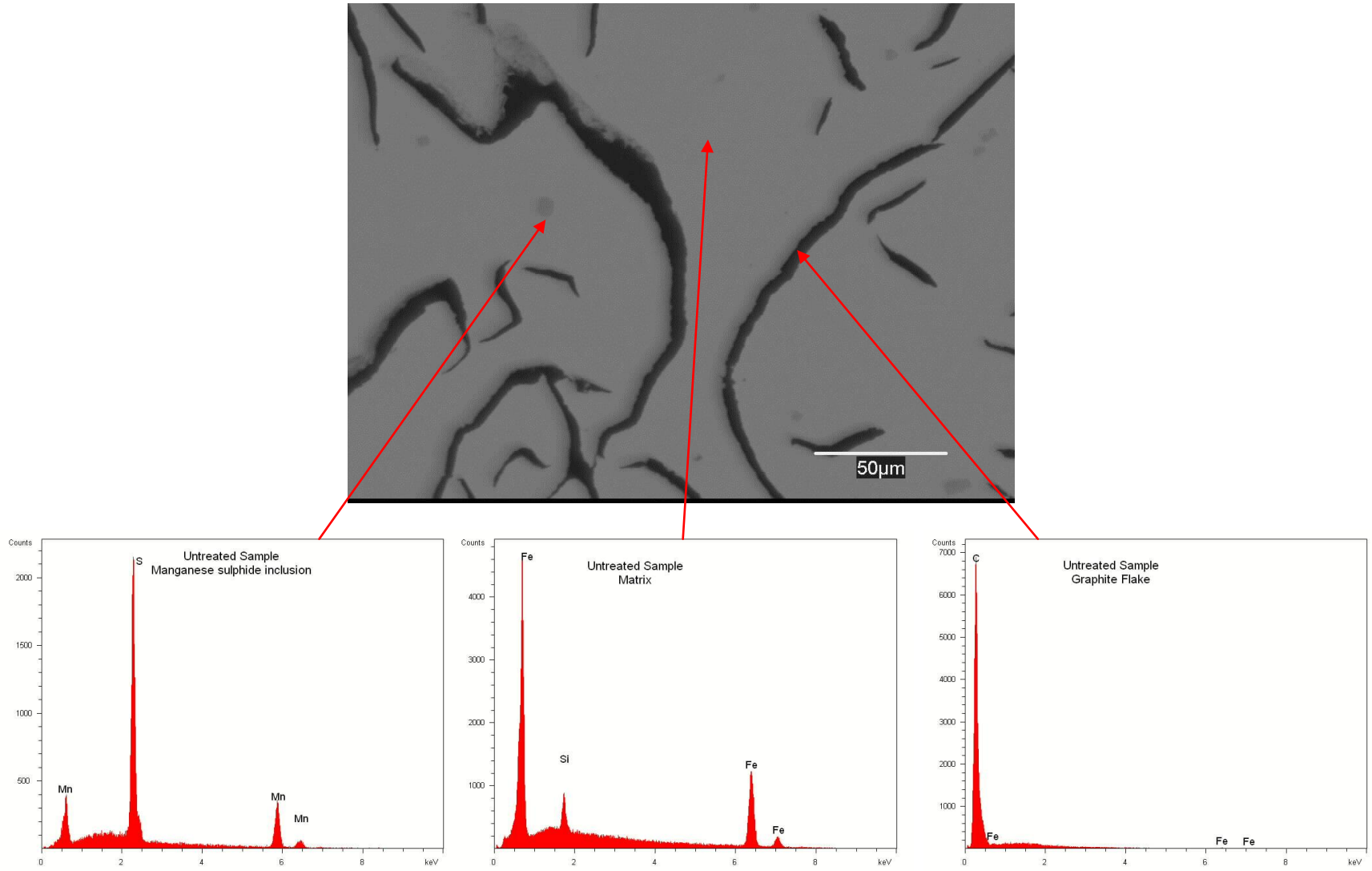
treated

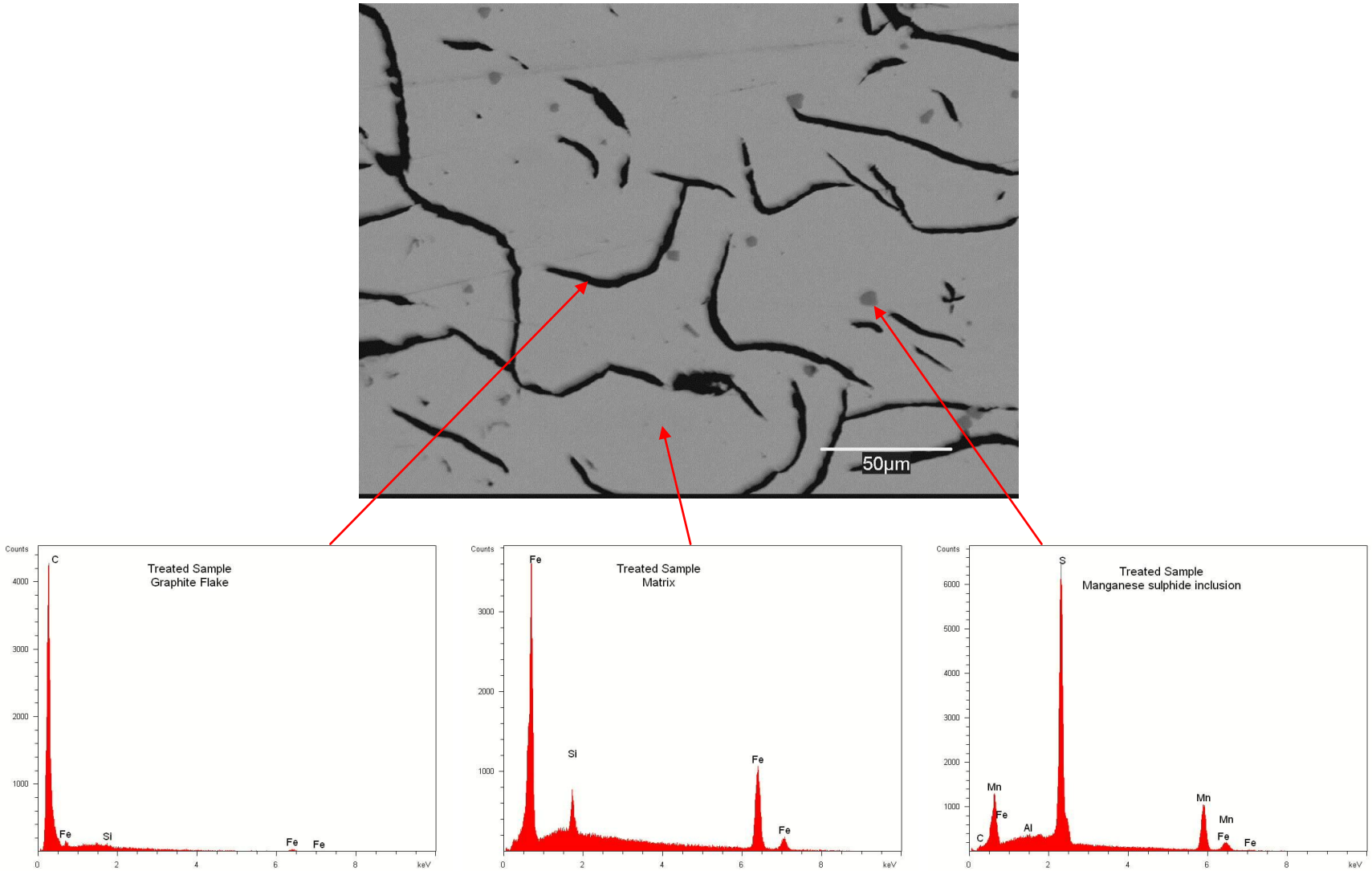
For each material The first image shows a general micrograph of the material surface. The second image, marked C, shows regions of concentrated carbon in yellow. The image marked Fe shows the iron distribution in the sample. The other maps show the presence of Sulfur (S), Silicon (Si) and manganese (Mn).

Both samples show that the graphitic flakes consist predominately of pure carbon - i.e., therefore not Fe_3C (cemetite). The matrix consists of iron with silicon diffused throughout the iron. The inclusions can seen to consist of manganese and sulphur. This would be present in the form of manganese sulphide. The graphite flakes are finer and more evenly distributed in the treated material. As well, the manganese sulphide inclusions are larger and more frequent.

Below is shown for the untreated (first) and treated (following page) materials the carbon peak associated with the graphite flakes, the iron peak associated with the matrix material, and the silicon peak associated with the silicon inclusions. The strong carbon peak associated with this graphitic flakes can be seen. The presence of silicon in the strongly ferrous matrix is evident. And it can be observed that the inclusions are composed of both manganese and sulphur.

Note multiple peaks evident for each element (ie., iron and manganese). Each peak corresponds to characteristic x-ray ejection for a different electron orbit. Electron disturbance in a lower orbital shell will result in a higher frequency characteristic x-ray.





The chemical composition of the untreated and treated specimens, in each of the area of interest (graphite flakes, matrix and inclusions) is shown. The treated material can be seen to have experienced atomic diffusion as a result of the Thermal Cycling treatment. Specifically, carbon has difussed away from the graphitic flakes, and iron and a small amount of silicon has diffused into the flakes. As well, carbon and iron have diffused into the manganese sulphide inclusions.

Description	C	Si	S	Mn	Fe
graphite flake	99.2				0.8
matrix		2.4			97.6
MnS inclusion			36.6	63.4	

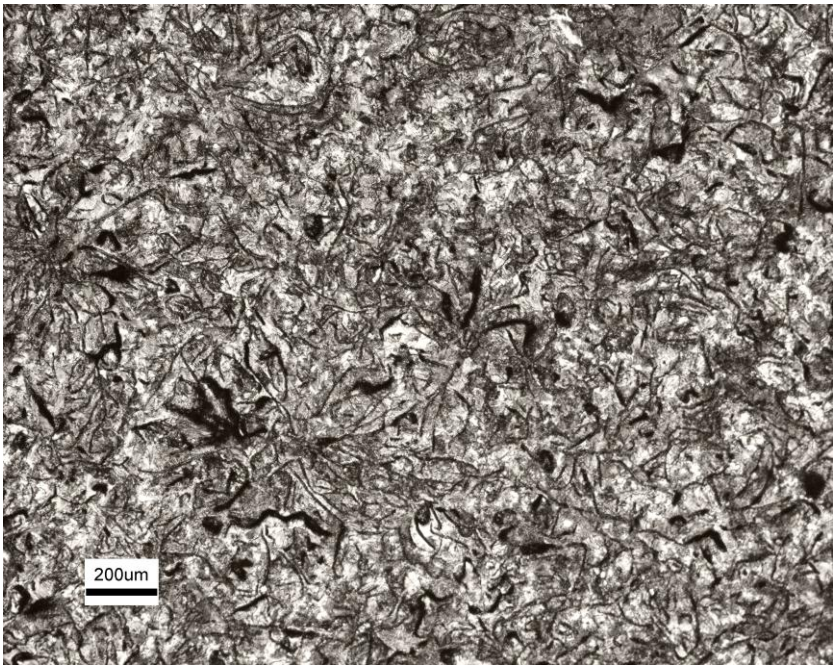
Untreated

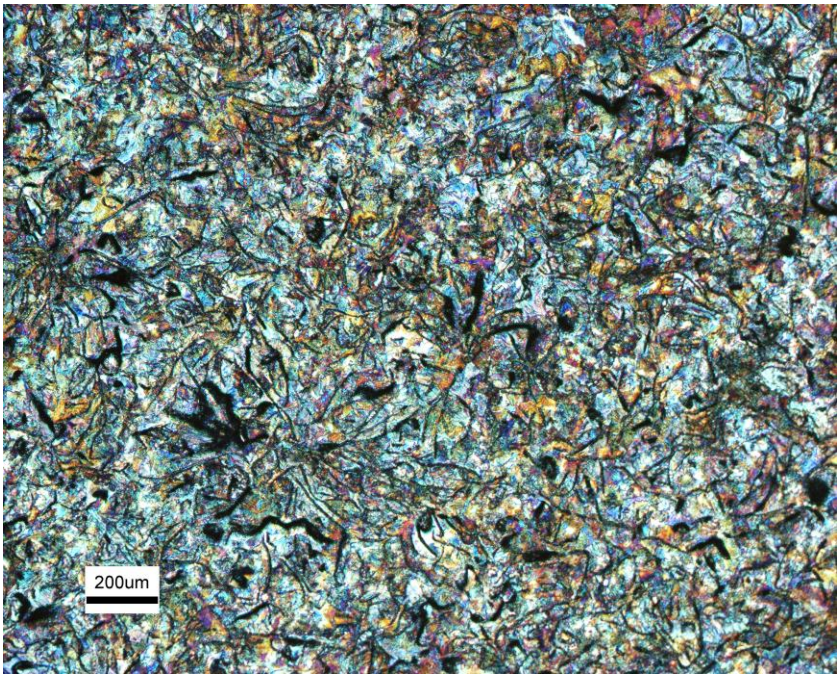
Description	C	Al	Si	S	Mn	Fe
graphite flake	97.1		0.1			2.8
matrix			2.3			97.7
MnS inclusion	4.4	0.2		29.3	56.5	9.6

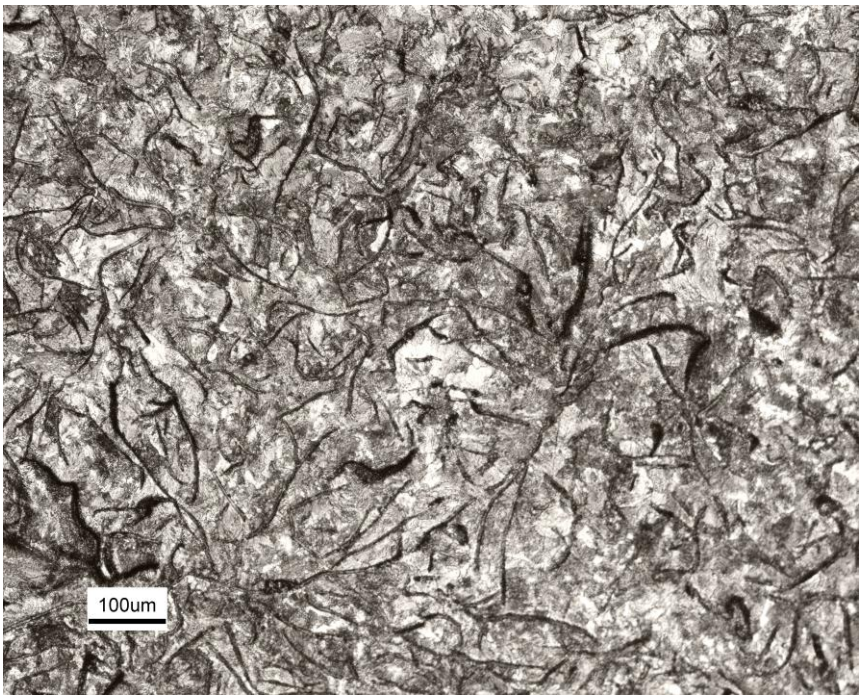
Treated

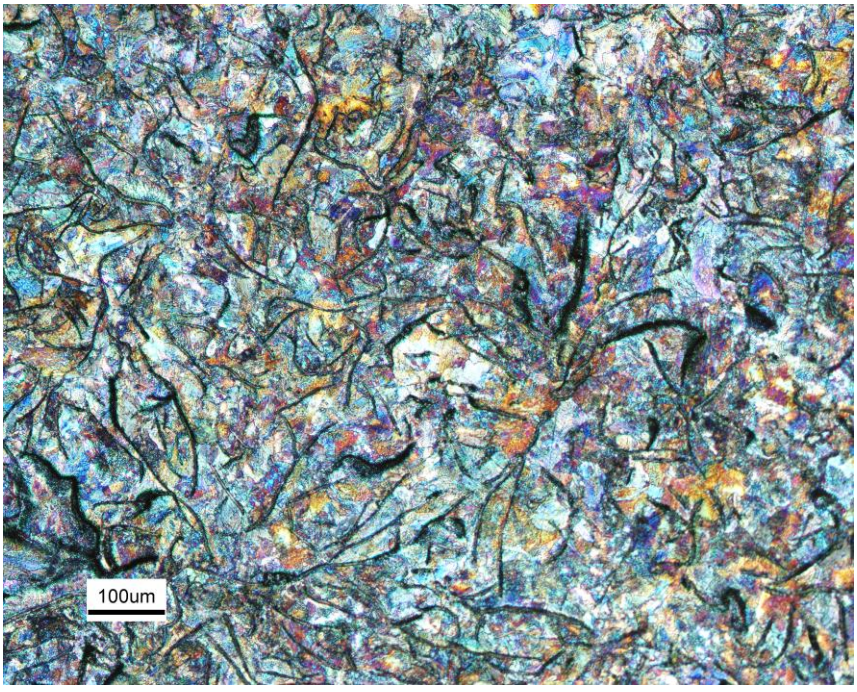
Microstructural Evaluation

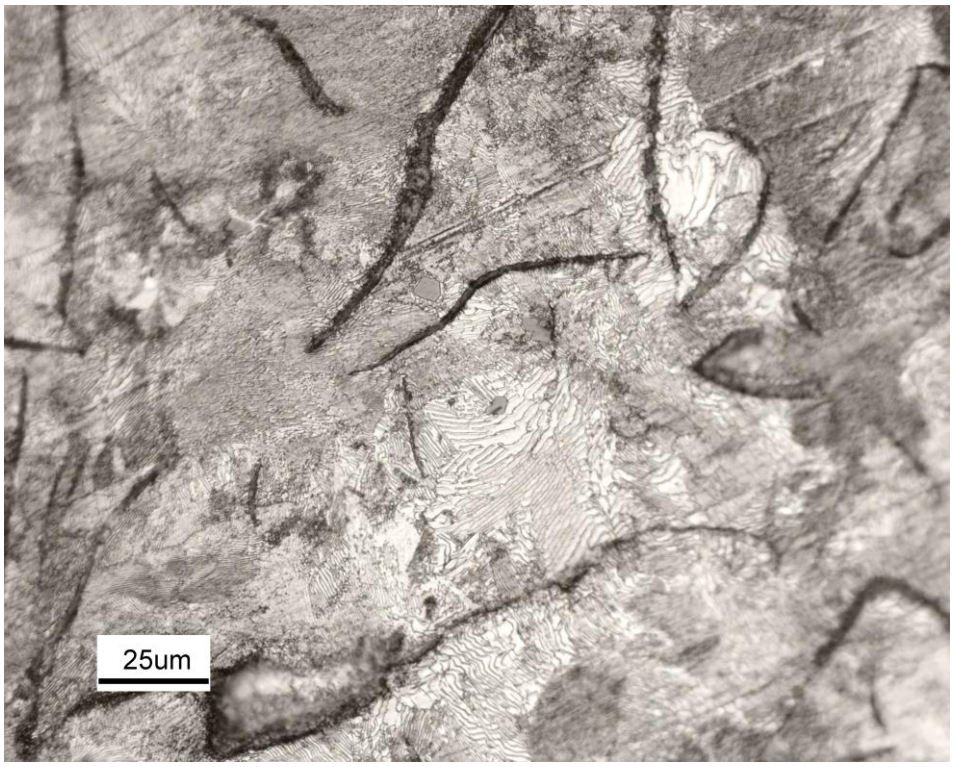
Optical micrographs of the treated and untreated materials are shown below. Material surfaces were polished to a mirror finish and etched in 5% Nital (5% nitric acid and 95% methanol, by volume) to reveal details of the microstructure. Black and white images are provided to show the graphite flakes and the metallic phases present more clearly. Color images show the grain boundaries more clearly. Variations in color correspond to distinct grains, resulting in differing angles of light reflection.





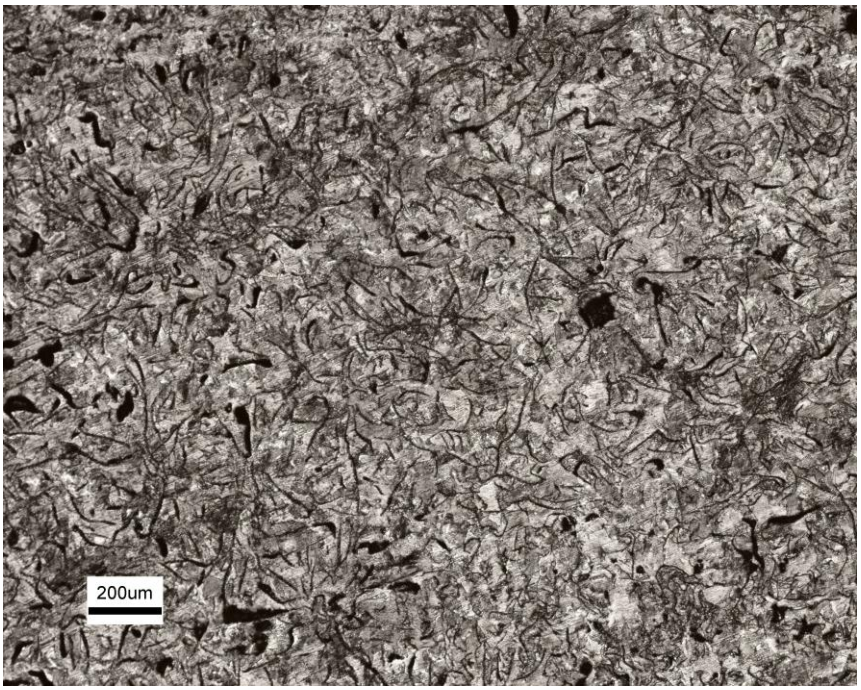


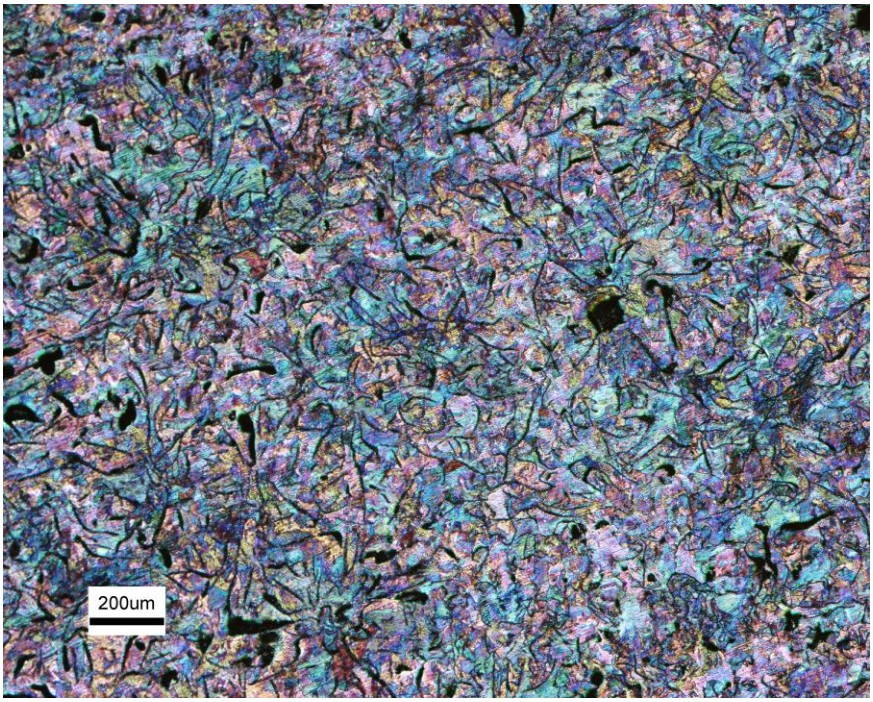


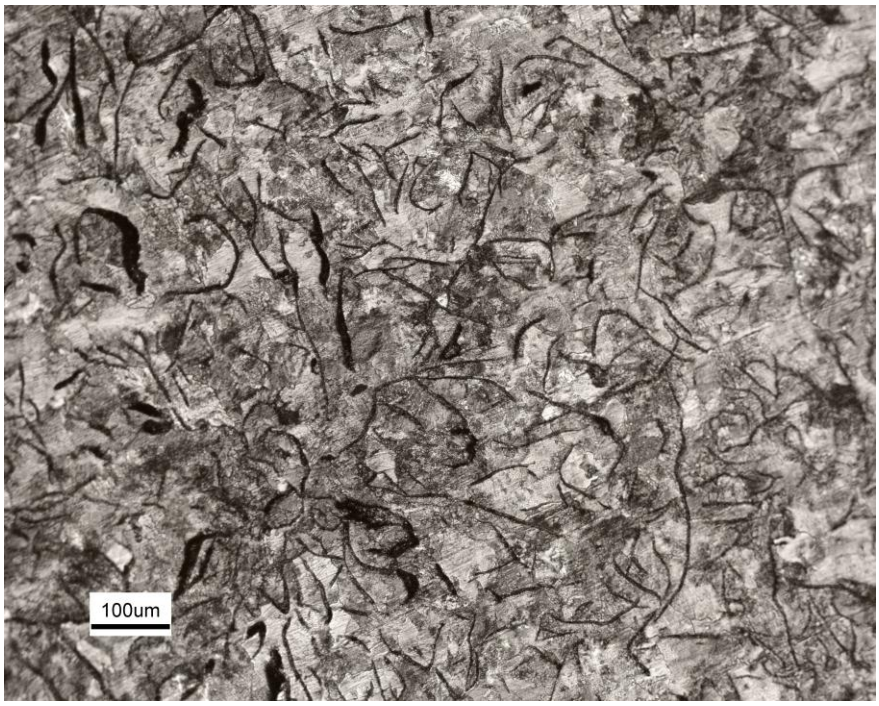


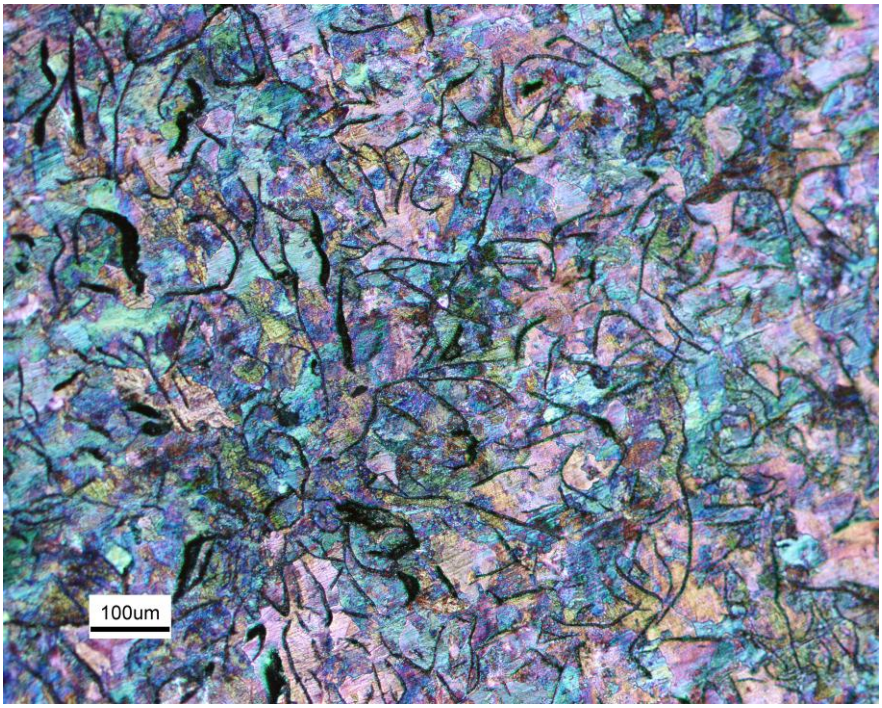
These first 5 micrographs are of the untreated material, shown in black and white and color, at increasing magnifications to better illustrate the various microstructural features.

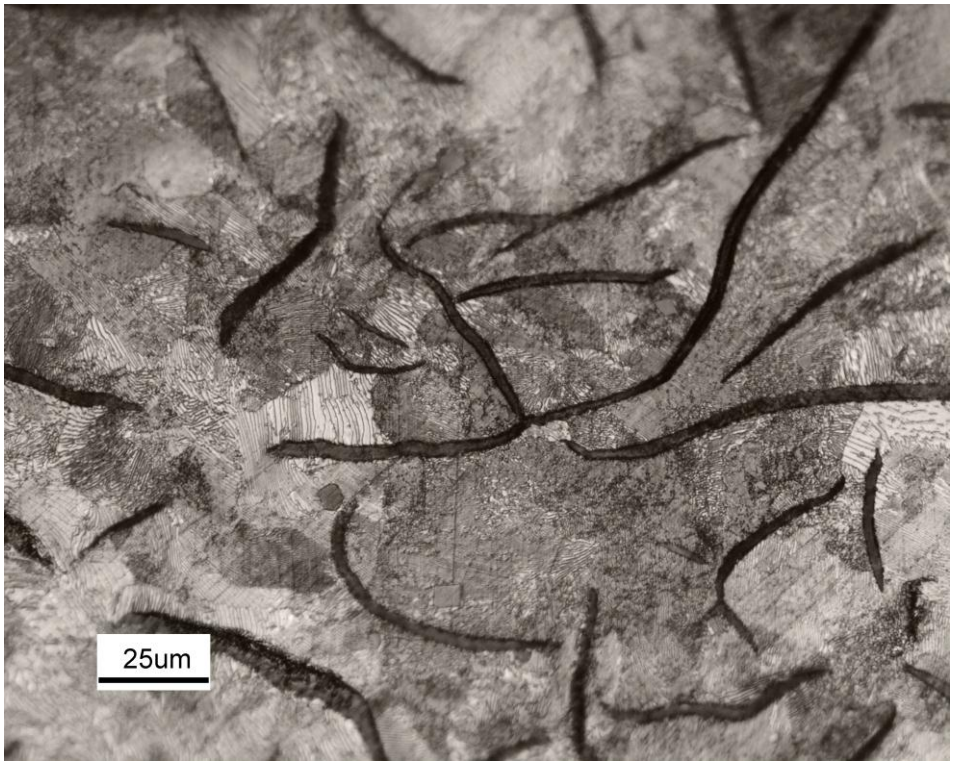
The metallic phase can be seen to consist of course pearlite, typical for a gray cast iron. The pearlite is indicated by the fine alternating black and white laminated patterns within the ferrous grains. The darker regions are composed of cemetite (iron carbide), while the lighter regions consist of ferrite. The graphite flakes can be seen to occur regularly along grain boundaries.











These next 5 micrographs are of the treated material, shown in black and white and color, at increasing magnifications to better illustrate the various microstructural features.

As low magnification, the treated and untreated materials appear very similar. However, at higher magnification, changes can be seen. For the treated material, the metallic phase can be seen to consist of pearlite, typical for a gray cast iron and the same as with the untreated material. However, the pearlite has become finer due to the treatment While the graphite flakes appear as finer and more evenly distributed throughout the material along the grain boundaries.

5. Discussion / Interpretation

The brake drums were determined to consist of gray graphitic cast iron. This is a typical material used in the fabrication of brake drums, engine blocks, flywheels and other automotive components. The presence of significant graphite flakes embedded within the iron matrix are a key identifier of gray cast iron. The iron microstructure was determined to be pearlite, consisted of alternating lamina of ferrite and cemetite. Pearlite is commonly observed with gray cast iron and indicates that the iron was slow cooled to room temperature following casting (i.e., not quenched, which would result in bainite or martensite). Slow cooling is typical for gray cast iron as this enhances the formation of the graphitic flakes. The high carbon content of gray cast iron ensures that the material is very hard and exhibits excellent wear characteristics. These materials however are typically brittle and exhibit low impact and fracture toughness, and do not typically exhibit a yield strength unique from the ultimate tensile strength. Gray cast irons typically contain 2.5 - 4.0% carbon. They also contain 1 - 3% silicon, manganese and sulfur. These additions result in an alloy that can be easily cast, reducing component cost. The silicon also stabilizes the carbon so that it forms graphite flakes rather than iron carbide (cemetite). Though a hard material, gray cast iron can be easily machined due to the presence of the carbide flakes, which are brittle and fracture easily during machining. Typical mechanical properties of various ASTM grades of gray cast iron are given below.

Properties of Gray Cast Iron

ASTM Number	Tensile Strength (Kpsi)	Compressive Strength (Kpsi)	Shear Modulus (Kpsi)	Modulus of Elasticity (Mpsi)		Endurance Limit (Kpsi)	Brinell Hardness H_b
				Tension	Torsion		
20	22	83	26	9.6-14	3.9-5.6	10	156
25	26	97	32	11.5-14.8	4.6-6.0	11.5	174
30	31	109	40	13.0-16.4	5.6-6.6	14	201
35	36.5	124	48.5	14.5-17.2	5.8-6.9	16	212
40	42.5	140	57	16.0-20	6.4-7.8	18.5	235
50	52.5	164	73	18.8-22.8	7.2-8.0	21.5	262
60	62.5	187.5	88.5	20.4-23.5	7.8-8.5	24.5	302

SEM, EDAX X-ray intensity maps, and metallographic examination all indicate that the graphitic flakes appear to have a finer structure and are more evenly distributed for the treated material. As well, the silicon and manganese inclusions have increased in size and frequency. This suggests that the Thermal Cycling treatment has enhanced precipitation reactions within the material and at the grain boundaries. This will have a tendency to enhance material wear properties and increase strength by reducing residual stresses within the microstructure.

From the analysis of the chemical composition of the untreated and treated specimens, it can clearly be seen that the Thermal Cycling treatment had a significant effect on the chemical properties of the various features of the material (graphite flakes, matrix and inclusions).

The data is repeated here for convenience of reference.

Description	C	Si	S	Mn	Fe
graphite flake	99.2				0.8
matrix		2.4			97.6
MnS inclusion			36.6	63.4	

Untreated

Description	C	Al	Si	S	Mn	Fe
graphite flake	97.1		0.1			2.8
matrix			2.3			97.7
MnS inclusion	4.4	0.2		29.3	56.5	9.6

Treated

The treatment can be seen to have encouraged diffusion of the carbon from out of the graphite flakes and into the inclusions. It also induces diffusion of iron and silicon into the carbide flakes and the inclusions. As well, aluminum, present in very low quantities within the matrix, experienced preferential diffusion into the inclusions.

The precise effect that this transformation would have on the mechanical properties of the material is difficult to predict based on this analysis alone. Mechanical testing of the materials would be required to quantify the effects.

However, this analysis is very important in that it identifies the actual mechanism (or at least, one of the mechanisms) of microstructural change that occurs due to the Thermal Cycling treatment. It is of key importance in understanding how to improve material properties that the mechanism of transformation is identified and characterized. This is a significant initial step in this direction. This knowledge can be used to identify materials (and products) that would benefit from being treated by Thermal Cycling.

6. Conclusion

SEM, EDAX X-ray mapping, and metallography all indicate a microstructural transformation within the brake drums resulting from the Thermal Cycling treatment. Application of Thermal Cycling has enhanced precipitation reactions and diffusion within the material and at the grain boundaries. This will have a tendency to enhance material wear properties and increase strength by reducing residual stresses within the microstructure.

The precise effect that this transformation has on the mechanical properties of the material is difficult to predict based on this analysis alone. Mechanical testing of the materials would be required to quantify the effects.

Significantly, this analysis identifies the mechanism, or a mechanism, of microstructural transformation that occurs due to the Thermal Cycling treatment. Understanding this mechanism is critical to evaluating the optimal approach to improving material properties and ultimately, product performance, through the application of Thermal Cycling.

7. Recommendations

It is recommended that the transformation mechanism of enhanced precipitation and diffusion reactions be studied in greater detail to optimize Thermal Cycling process parameters (ie., temperature, duration, ramp up and ramp down times, post treatment, etc).

Materials could be treated at various process parameters and the resulting microstructure and chemical composition can be evaluated to determine which set of parameters produced an optimal structure.

Mechanical properties should be measured to correlate microstructural change to property enhancement. Thermal Cycling process parameters could then be tailored to optimized preferred mechanical properties. Mechanical properties to be considered would be wear properties, strength, hardness and impact toughness.

A range of materials could be treated with varying Thermal Cycling process parameters to evaluate effect on microstructure and mechanical properties for various material, and the components manufactured from these components. This would determine the materials that experience the most significant benefits through the application of Thermal Cycling, narrowing and defining the product markets that the Thermal Cycling treatment would most significantly benefit through enhancement of critical mechanical properties for that product (i.e., wear properties of brake components, strength properties of beams and automotive frames, hardness of saw blades, impact toughness of hammer heads, etc)