Estimation of the effective conductivities of complex cast iron microstructures using FIB-tomographic analysis

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Received 14 February 2009; revised 1 July 2009; accepted 2 July 2009
Available online 28 July 2009

Abstract

We present the influence of microstructural characteristics of different graphite morphologies on the effective properties of cast iron. First, the properties are compared with existing analytical bounds and predictive schemes. The estimation of the properties of nodular, flake and all intermediate graphite morphologies becomes possible after the analysis of the graphite crystal structure and growth mechanisms. These properties are implemented in a two-dimensional finite element model. It is shown that the consideration of microstructural parameters—volume fraction, surface density and particle shape—provides good estimates of the properties of cast iron with simply connected nodular graphite particles, but is insufficient for interconnected structures with vermicular and flake graphite. The agreement of the experimental results with three-dimensional simulations on real tomographic data proves that the connectivity of phases is one of the determining factors for material properties.

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Keywords: Effective conductivity; Simulations; Tomography; Graphite morphology; Cast iron

1. Introduction

1.1. Influence of the graphite morphology on the properties of cast iron

Cast iron can be presented as a complex composite consisting of an iron matrix and different graphite inclusions. Properties of the matrix can vary depending on the phase composition [1,2]. However, it is the graphite morphology that has the decisive effect on the properties of cast iron [3-6]. The challenge of prediction of the cast iron properties based on the graphite morphology is connected with the variety of size, shape and spatial arrangement of the graphite inclusions (see Ref. [7]).

Graphite has the highest thermal and the lowest electrical conductivity among all microconstituents of cast irons. Thus, in the extreme case of cast iron with interconnected flake graphite (FG), the heat flux occurs mainly through graphite particles, and in the electrical conductivity problem the same particles serve as barriers for the electron transport. On the other side, the thermal and electrical conductivity of the ductile iron with disconnected nodular graphite (SG) inclusions approaches the values for steels. All intermediate graphite morphologies, including vermicular graphite (CG), lead to characteristic conductivity values between those of the above-mentioned extreme cases.

Hence, these or other physical characteristics can be used for the non-destructive testing as an indicator of the microstructure and can even be integrated into the quality control by the processing. Electromagnetic properties were found to be promising for this application [8]. In order to correctly interpret the measured electromagnetic signal and internal microstructure, the complete understanding of the influence of the microstructure on the electric and magnetic properties is required.

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As pointed out by Hashin and Shtrikman [9], the problem of the prediction of the effective magnetic permeability, dielectric constant, electrical conductivity, thermal conductivity and diffusivity of heterogeneous media are mathematically analogous. Considering the complexity of the task, in this work the problem was narrowed to the analysis of the microstructure effect on the electrical properties. The analogy was drawn with thermal properties and compared with literature values. The goal is to correlate the property with the quantitative microstructure characteristics and when possible suggest a model which is acceptable for other material properties, e.g. magnetic permeability. Knowing that the shape of graphite particles in the same, e.g. pearlitic, matrix causes various cast iron properties (Table 1), partial influence of the graphite morphology will be primarily considered in this work.

1.2. Calculation of the effective properties of the composites

An absolute requirement for the prediction of the effective properties of different materials is the knowledge about properties and arrangement of the microstructural constituents. Empirical as well as analytical correlations between microstructural parameters and certain properties work as long as a sufficient amount of microstructural information is available. Either the property depends only on one microstructural characteristic, e.g. density is proportional only to the volume fraction, or the microstructures are completely characterized. The microstructure can be considered fully characterized when its four basic characteristics – volume fraction ($V_f$), specific surface area ($S_f$), density of the integral of mean curvature ($M_f$) and total curvature ($K_f$) – are known [10]. They can be either calculated for the simple microstructure with the help of stereological equations [10,11] from two-dimensional (2-D) micrographs or directly acquired from 3-D measurements [12]. Excluding the simplest arrangements, the determination of such information is often laborious and time-consuming. Additionally, tomographic methods working on different scales (computed tomography, synchrotron or focused ion beam tomography) may be required.

1.2.1. Bounds

The properties can be bracketed by bounds or limits: upper and lower values between which the effective properties are located [13]. The parallel and serial addition of the corresponding properties is the simplest of the mixing rules (ROM). Hashin–Shtrikman (HS) variation bounds establish more rigorous upper and lower bounds on the effective electrical conductivity $\sigma_{\text{eff}}$ of macroscopically isotropic composites with an arbitrary microstructure [14]:

$$\sigma_{\text{lo}} + \frac{V_h}{\frac{1}{\sigma_v} - \frac{1}{\sigma_h}} + \frac{V_h}{\frac{1}{\sigma_v} + \frac{1}{\sigma_h}} \leq \sigma_{\text{eff}} \leq \sigma_{\text{hi}} = \sigma_h + \frac{1 - V_h}{\frac{1}{\sigma_v} - \frac{1}{\sigma_h}}$$

(1)

where $\sigma_h$ and $V_h$ are the electrical conductivity and volume fraction of the high-conductive phase, respectively, and $\sigma_v$ and $V_v$ are the electrical conductivity and volume fraction of the low-conductive phase. For low to moderate phase contrast in the considered property, such a procedure can bracket the property with bounds that are relatively close. The Hashin–Shtrikman bounds are the best possible bounds on the effective conductivity of isotropic two-phase composites given only the volume fraction information.

1.2.2. Predictive schemes

For large contrast, however, as in the electrical conductivity of graphite/iron matrix in cast iron, the bounds are far apart and the admitted values can vary by one to several orders of magnitude. In this case it is preferable to use an analysis that takes the actual basic arrangement and shape of the two phases into account. Most of the predictive schemes are based on the solution of the intensity field within an ellipsoidal inclusion embedded in a matrix subjected to a remotely applied field [15]. At low inclusion concentrations, the problem is often simply solved by assuming no interaction between inclusions.

Comparing different approaches [15], the differential effective medium approach (DEM) [14,16] was found to be one of the best predictive schemes that adequately estimates the effective conductivity of two-phase materials of a non-conducting material embedded in a conducting matrix, for both equiaxed and angular particles [15] and for Al–Si alloys [17], as well as for closed-cell aluminum foams [18].

The formula for predicting the effective electrical conductivity $\sigma_{\text{eff}}$ is a function of the volume fraction of inclusion phase $V_i = 1 - V_m$ for the case where conductivity of the inclusions $\sigma_i = 0$ and the inclusions are spheres (Eq. (2)) or randomly oriented spheroids (Eq. (3)). Here

### Table 1
Comparison of the properties of FG-250, CG-500 and SG-700 [38,40].

<table>
<thead>
<tr>
<th>Property</th>
<th>Measuring unit</th>
<th>FG-250</th>
<th>CG-500</th>
<th>SG-700</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tensile strength, $R_m$</td>
<td>MPa</td>
<td>250-350</td>
<td>500</td>
<td>700</td>
</tr>
<tr>
<td>Fracture strain, $e_f$</td>
<td>%</td>
<td>0.3-0.8</td>
<td>1</td>
<td>2</td>
</tr>
<tr>
<td>Elastic modulus, $E$</td>
<td>GPa</td>
<td>103</td>
<td>170</td>
<td>177</td>
</tr>
<tr>
<td>Thermal conductivity, $\lambda$</td>
<td>W (m$^{-1}$ K$^{-1}$)</td>
<td>45</td>
<td>40</td>
<td>30</td>
</tr>
<tr>
<td>Electrical conductivity, $\sigma$</td>
<td>10$^7$ S m$^{-1}$</td>
<td>1.37</td>
<td>1.67$^*$</td>
<td>1.85</td>
</tr>
<tr>
<td>Maximum permeability, $\mu$</td>
<td>$\mu$ H m$^{-1}$</td>
<td>220-330</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

$^*$ Measured value for CG-400.
\( V_m \) is the volume fraction and \( \sigma_m \) is the electrical conductivity of the matrix.

\[
\sigma_{\text{eff}} = \sigma_m (1 - V) \sqrt[3]{2/9} \tag{2}
\]

\[
\sigma_{\text{eff}} = \sigma_m (1 - V) \sqrt[3]{1 - 3V^2} \tag{3}
\]

In the latter case, the effective polarization factor \( S^y \) is calculated from the aspect ratio \( q \) of the spheroid according to equations summarized in Ref. [17].

1.2.3. Finite element methods

As opposed to the procedures described above, there are intentions to calculate macroscopic material properties from knowing the special microstructure arrangement and local physical property. Such works are motivated by the development of the numerical methods for the solution of partial differential equations, such as finite element methods (FEM).

The estimation of the effective properties of particle-reinforced metal–matrix composites is based on the analysis of a real structure and can be done mainly in two different ways: one can directly import the experimentally obtained real structure into a FE software [19,20] or generate model structures that have similar statistical functions as the experimentally obtained ones [21]. The statistically equivalent modeling approach becomes very difficult in 3-D, since, besides the position of the particles, their sizes, orientations, and shapes must be taken into account according to the corresponding marked correlation functions [22]. Due to these difficulties, direct simulation of real structures seems to be more straightforward. Before simulating the real structure, one has to solve also the problem of sampling, i.e., how the different simulation windows should be selected from the large reconstructed volume. This simulation window should not be smaller than a representative volume element [23], i.e., the smallest material volume that contains all microstructural information and can be considered for estimation of the effective properties.

It is clear that the effective properties of cast iron with different graphite morphologies cannot be predicted by only considering the graphite volume fraction. The goals of this work are thus first, to compare the experimentally determined properties with existing bounds and predictive schemes; secondly, to find correlations between the effective properties and 3-D graphite morphology; and last, to try to estimate them using FE methods considering the different graphite crystal structure.

2. Experimental part

2.1. 2-D and 3-D microstructure characterization and analysis

The microstructural analysis of cast iron was performed in 2-D with the help of optical (Olympus BX60) and scanning electron microscopy (Dual Beam Strata 235 of FEI Company, Eindhoven, NL). The 3-D analysis of the graphite shape and spatial distribution was done using focused ion beam (FIB) microstructure tomography. The general principle of FIB-tomography is described in Ref. [24]. Details for the analysis of the graphite inclusions in cast iron are given in Ref. [25]. A TSL EBSD system was used for the analysis of the crystallographic orientation of the pearlitic assemblies. STEM or TEM observations were required to analyze the structure of the graphite inclusions of different morphology.

2-D and 3-D quantitative image analysis was performed using a4i Analysis (Olympus) and the software system Modular Algorithms for Volume Images, MAVI, of Fraunhofer ITWM, respectively.

2.2. Measurement of the electrical conductivity

The electrical conductivity at room temperature was measured using a four-point direct current technique. The equipment used was from Keithly Instruments, Inc., Cleveland, USA (Model 2400 Series Source Meter and Model 2000 Multimeter). The measured values are linearly fitted in Ohm’s line, the slope of which gives the electric resistance \( R \) of the sample. The specific, i.e., geometry independent, electrical resistivity \( \rho \) can be calculated according to the following formula: \( \rho = R \frac{h}{w \cdot d} \), where \( h \) is the height and \( w \) the width of the sample; \( d \) is the distance between contacts. The electrical conductivity is reciprocal to the electrical resistivity: \( \sigma = \frac{1}{\rho} \).

2.3. 2-D and 3-D simulations

The electrical and thermal properties of the cast iron were estimated using FEM simulations. The simulations in 2-D were performed with the FlexPDE (PDE Solutions Inc.) program, in 3-D using the GeoDict Software, developed at ITWM for the simulation of structures and structure–property relationships [26,27]. Both finite element simulations are based on numerically solving the Laplace equation for the thermal (4) and electrical (5) problem.

\[
\nabla (-\lambda \cdot \nabla T) = 0 \tag{4}
\]

\[
\nabla (-\sigma \cdot \nabla V) = 0 \tag{5}
\]

Here \( \lambda \) and \( \sigma \) are given thermal and electrical conductivity and \( \nabla T \) and \( \nabla V \) the temperature and voltage gradient.

For 2-D simulations, the cast iron microstructure was represented by a pearlitic matrix with ellipsoidal graphite inclusions positioned parallel and perpendicular to the flow. The effective conductivity of the composite with randomly oriented ellipsoids can be then approximated by the mean value of these two cases.

Computations of the thermal and electrical conductivity problems on the 3-D data sets were done in \( x \), \( y \), and \( z \)-directions on real tomographic data. Here for the first time the voxel with different edge length which are characteristic for FIB-tomography were implemented in the simulations. The mathematics involved in the simulation and
the solver of the GeoDict software has been comprehensively described by Wiegmann and Zemitis [28].

The simulated thermal and electrical conductivity problem is based on the literature values and calculations of the conductivities for individual microstructure constituents according to equations described in Helsing and Grimvall [29] summarized in Table 3 (see Section 3.1.2).

3. Results and discussion

3.1. Effective properties of cast iron

3.1.1. Electrical conductivity of cast iron with different graphite morphology

The electrical conductivity of a number of cast iron samples with different graphite morphologies was measured. Later, their microstructure was analyzed in 2-D and quantified. Table 2 summarizes measured electrical conductivity and microstructural characteristics of the graphite phase. For the calculation of the mean values for the particle features the particle area was considered.

No clear influence of the graphite volume fraction \( V_F \) on the electrical behavior of the cast iron could be confirmed. Cast iron samples with nodular, vermicular and flake graphite have characteristic values for the electrical conductivity and for the specific surface area \( S_\nu \), particle density per unit area \( N_A \), size \( \sigma \) (e.g. MaxFeret) and shape \( \sigma \) (aspect ratio \( q \)) of the graphite particles. Although, as these dependencies are not continuous functions over all graphite morphologies, none of them alone suffices to describe the correlation between graphite microstructure and electrical properties of cast iron.

In order to compare measured values with existing analytical bounds and predictive schemes the properties of the individual microstructural constituents have to be known.

3.1.2. Estimation of the properties of the microstructure constituents

Considerable input to the estimation of physical properties of cast iron was done by Helsing and Grimvall [29].

3.1.2.1. Ferrite. Thermal and electrical conductivity of alloyed ferrite (Table 3) was calculated according to Eqs. (15)-(17) of Ref. [29], knowing the chemical composition in at.% of the cast iron samples.

3.1.2.2. Cementite. The same authors [29] have summarized all known estimations, calculated the electrical conductivity of cementite \( \rho (\text{Fe}_3\text{C}) \approx 1.07 \, \mu \Omega \cdot \text{m} \) and chosen as thermal conductivity for subsequent modeling \( \lambda (\text{Fe}_3\text{C}) = 8 \, \text{W} \cdot \text{m}^{-1} \cdot \text{K}^{-1} \).

3.1.2.3. Graphite. Graphite has hexagonal lattice symmetry. The electrical and thermal conductivities are strongly anisotropic. Along the hexagonal lattice the conductivities are much higher than perpendicular to them. Experiments at room temperature on pyrolytic graphite [30] result in \( \lambda_\parallel \approx 2000 \, \text{W} \cdot \text{m}^{-1} \cdot \text{K}^{-1} \) and \( \lambda_\perp \approx 10 \, \text{W} \cdot \text{m}^{-1} \cdot \text{K}^{-1} \). The maximum attainable conductivity is very sensitive to lattice defects [30]. The graphite conductivities chosen for the modeling are summarized in Table 3.

3.1.2.4. Pearlite. Pearlite has to be considered as a separate single phase, with anisotropic conductivities along the cementite lamellae \( \sigma \parallel \) and perpendicular to them \( \sigma \perp \). The effective properties were calculated from the estimated values of parallel and serial coupling. The volume fraction of cementite was calculated to be \( V_{\nu} (\text{Fe}_3\text{C}) = 0.114 \). EBSD analysis of the crystallographic orientation of the pearlitic assemblies did not show any texture. Thus, for a specimen, which can be considered as a polycrystalline material with

<table>
<thead>
<tr>
<th>Sample</th>
<th>( \sigma ) (10^6 S m^{-1})</th>
<th>Field features</th>
<th>Particle features, area weighted</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>( V_F ) (%)</td>
<td>( S_\nu ) (\mu m)</td>
<td>( N_A ) (10^{-3} \mu m^2)</td>
</tr>
<tr>
<td>FG</td>
<td>1.36 ± 0.05</td>
<td>11.2 ± 0.7</td>
<td>623 ± 127</td>
</tr>
<tr>
<td>CG</td>
<td>1.68 ± 0.08</td>
<td>8.2 ± 0.6</td>
<td>374 ± 88</td>
</tr>
<tr>
<td>SG</td>
<td>1.88 ± 0.05</td>
<td>8.9 ± 0.6</td>
<td>206 ± 1</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Sample</th>
<th>( \lambda ) (W m^{-1} K^{-1})</th>
<th>( \sigma ) (10^6 S m^{-1})</th>
<th>( \rho ) (\mu\Omega m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pure ferrite [41]</td>
<td>78.5</td>
<td>10</td>
<td>0.1</td>
</tr>
<tr>
<td>Alloyed ferrite</td>
<td>29.91</td>
<td>2.70</td>
<td>0.37</td>
</tr>
<tr>
<td>Cementite [29]</td>
<td>8</td>
<td>0.93</td>
<td>1.07</td>
</tr>
<tr>
<td>Graphite ( \lambda_F, \sigma_F, \rho_F ) [29,42]</td>
<td>500</td>
<td>2</td>
<td>0.5</td>
</tr>
<tr>
<td>Graphite ( \lambda_N, \sigma_N, \rho_N ) [29,42]</td>
<td>10</td>
<td>0.0001</td>
<td>10,000</td>
</tr>
<tr>
<td>Lamellar alloyed pearlite (( \lambda_\parallel, \sigma_\parallel, \rho_\parallel ))</td>
<td>27.4</td>
<td>2.47</td>
<td>0.404</td>
</tr>
<tr>
<td>Lamellar alloyed pearlite (( \lambda_\perp, \sigma_\perp, \rho_\perp ))</td>
<td>22.8</td>
<td>2.20</td>
<td>0.453</td>
</tr>
<tr>
<td>Alloyed pearlite, Eq. (6)</td>
<td>24.3</td>
<td>2.29</td>
<td>0.436</td>
</tr>
</tbody>
</table>
random grain orientation, Eq. (6) [29] yields the estimated isotropic conductivity (see Table 3).

\[ \sigma_{\text{avg}} = \frac{1}{4} \left[ \sigma_1 + \left( \sigma_2^2 + 8\sigma_1\sigma_3 \right)^{1/2} \right] \]

(6)

Table 3 summarizes the conductivity values used for analytical models and FEM simulations.

3.1.3. Influence of the volume fraction; comparison with analytic bounds

It was already shown that the physical properties of cast iron do not depend on the volume fraction of graphite only. Thus, the comparison with simple bounds serves mainly to estimate the values of the effective conductivity of graphite morphologies. The bounds where calculated with both values for the graphite conductivity in \( \alpha \)- and \( c \)-crystallographic directions.

The contrast between the thermal conductivity of graphite in \( \alpha \)-direction and pearlite is relatively low. Thus the relatively narrow bounds comprise this effective property (see Fig. 1). This proves that the thermal conductivity in cast iron with these types of graphite is conducted mainly by the conductivity in the graphite in the \( c \)-direction: even more in flake graphite than in vermicular graphite. The conductivity of the cast iron with nodular graphite is in the range of the conductivity of the pearlitic matrix. This means first that the graphite conductivity in nodules is between the values for the conductivity in the \( \alpha \)- and \( c \)-crystallographic directions, approaching more the value of the conductivity of graphite in \( c \)-direction, and second that the effective cast iron conductivity is mainly controlled by the conductivity of pearlite.

For large phase contrast, however, as in the electrical conductivity of cast iron, the bounds are far apart (see Fig. 2). It is remarkable that the experimental values of the electrical conductivity are situated far away from the bounds calculated with graphite conductivity equal to the graphite conductivity in the \( \alpha \)-direction and concentrate entirely within the bounds calculated using the graphite conductivity in the \( c \)-direction. Considering the theories about graphite growth and crystallographic structure [31–34], it can be assumed that the value of effective graphite conductivity is between the conductivities in the \( \alpha \)- and \( c \)-direction and is much lower than the conductivity of the pearlitic matrix. Thus, it was proven that graphite serves as an obstacle along the path of the electrons.

3.1.4. Influence of graphite shape; comparison with predictive schemes

Graphite shape is one of the most important factors. Nevertheless the known approaches for the estimation of

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Fig. 1. Bounds for the thermal conductivity for the cast iron calculated by the rule of mixture (ROM) and Hashin–Shtrikman (HS) upper and lower bounds (see Eq. (1)) with graphite conductivity in \( \alpha \)- and \( c \)-crystallographic directions, compared with the values from the literature [38].

Fig. 2. Bounds for the electrical conductivity for the cast iron calculated by the rule of mixture and Hashin–Shtrikman upper and lower bounds (see Eq. (1)) with graphite conductivity in \( \alpha \)- and \( c \)-crystallographic directions, compared with the experimental results.
the effective properties of composite materials with high contrast in the properties of the individual constituents \((c_i = 0)\) do not correlate well with the experimental results (Fig. 3). Here the in 2-D experimentally determined values of the aspect ratio (Table 2) were used to estimate the effective cast iron properties according to the differential effective medium (DEM) scheme for randomly oriented spheroidal inclusions (Eq. (3)). With the given shape parameter values this scheme considerably overestimates the experimental results. According to the scheme, the values of the aspect ratio of the vermicular and flake graphite particles should be below 0.1.

The models do not take into account the level of connectivity of the phases involved and thus they cannot accurately predict the effective conductivity of interpenetrating phase composites.

Comparing the experimental results with existing models it can be concluded that the following microstructural parameters need to be considered for the correct estimation of the effective properties of cast iron with different graphite morphology:

- volume fraction of the graphite phase \((V_g)\);
- graphite shape, size and density, which are generally derived from the basic parameters surface area density \((S_g)\) and the density of the integral of the mean curvature \((M_g)\), which are accessible from the 2-D image analysis;
- graphite 3-D arrangement, which can be described by the fourth basic characteristic – the density of the integral of the total curvature \((K_g)\), which for complex structures is accessible only from the 3-D image analysis.

Considering the fact that cast iron with specific graphite morphology (shape) possesses specific values of the electrical and thermal conductivity and assuming the possible existence of cast iron of mixed microstructure, the fractions of the respective graphite types have to be described quantitatively. Thus, the correct classification of each individual graphite particle has to be performed.

Besides, in order to make sufficiently good estimates of the effective conductivity of the graphite of different morphology, special attention has to be paid to the characterization of the graphite crystallographic structure.

3.2. Effect of the graphite morphology on the effective properties

3.2.1. Application of graphite classification

The graphite morphology of the analyzed samples was classified using the algorithm developed in Refs. [7,35]. Graphite particles in each cast iron sample were classified and their area fraction was calculated for the fraction of all graphite inclusions equal to 100%. Although the fraction of the graphite is different in different cast iron samples (see Table 2), the electrical conductivity was normalized. Thus, the results for all cast iron samples with flake (FG), vermicular (CG) and nodular (SG) graphite can be compared in Fig. 4.

If all graphite particles were classified as vermicular graphite the data would concentrate on the origin of the coordinates system. In the analyzed cast iron samples with vermicular graphite some particles were assigned to flake type (I) and some to nodular type (IV–VI). The amount of the respective type can be seen on the projection on the \(x-y\) plane. In samples with flake graphite over 70% of area of the graphite particles were classified as type I, the rest is mostly type III. Almost all particles were classified as types IV–V or VI in the cast iron sample with nodular graphite.

The diagram shows well that the electrical conductivity of cast iron correlates with the fraction of the respective graphite morphology. When a cast iron sample with ver-

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![Fig. 3. Comparison of the experimental values of the electrical conductivity of cast iron with nodular (SG), vermicular (CG) and flake graphite (FG) with effective conductivity values predicted according to the differential effective medium (DEM) scheme for spheres (Eq. (2)) and random spheroids (Eq. (3)) with different aspect ratios \(q\).](image_url)

![Fig. 4. Electrical conductivity of cast iron with flake (FG), vermicular (CG) and nodular (SG) graphite presented vs. area fraction of graphite classified as flake (I), vermicular (III) or the graphite types IV–V + VI according to DIN ISO 945. Fraction of graphite types refers to 100% total graphite content.](image_url)
micellar graphite contains particles which were classified as flake graphite (type I), the conductivity reduces. If it contains nodular graphite particles (type VI), the conductivity does not change significantly.

Proving that the graphite shape plays the most important role for the properties of the cast iron, an attempt can be made to find a simple model and to conduct finite element simulations. In order to perform a reasonable approximation of the effective conductivity, the properties of the individual phases have to be known. Whereas for the pearlite matrix it was possible to calculate the thermal and electrical conductivity (see Section 3.1.2 and Table 3), the analysis of the graphite crystal structure had to be performed in order to estimate the conductivity of the anisotropic complex graphite particles of different shape.

3.2.2. Graphite crystallographic structure

The crystallographic structure of the graphite inclusions was successfully analyzed on the TEM foils acquired using a target preparation with a FIB workstation [36]. For the nodular graphite the radial growth of the conical crystals as suggested by Refs. [31,32] was observed (see Fig. 5a).

For the flake graphite, the [0 0 1] crystallographic direction of the graphite lattice is mainly perpendicular to the graphite–matrix interface (Fig. 5b). Two different regions equivalent to two different growth mechanisms are seen on the TEM specimen through the vermicular graphite particle (Fig. 5c): one of the nodular graphite on the bottom and one of the flake graphite at the top. The observed crystallographic structure is consistent with the assumption made by Llorca-Isern et al. [33] and Tartera et al. [34], and developed by Velichko et al. [36].

3.2.3. Estimation of the graphite effective conductivity using 2-D simulations

The effective conductivity of nodular graphite has been estimated using the Double and Hellawell model [31,32] from the radial and tangential conductivities of graphite (Table 3). To do so, a model of a single graphite nodule embedded in a pearlitic matrix was used for the simulation. The distribution of the anisotropic properties within the single nodule is calculated from the properties of graphite crystal in a- and c-direction. Assigning the origin of the coordinate system to the center of the spherulitic graphite particle the conductivity in each point (x, y) can be defined with trigonometric functions as follows:

\[
\theta = \text{abs} \left[ \arctan \left( \frac{y}{x} \right) \right]
\]  
\[
z = \text{abs} \left[ \arctan \left( \frac{x}{y} \right) \right]
\]  
\[\lambda_a = \sqrt{\lambda_a^2 + \lambda_c^2 \cdot \sin(\theta - z)}\]  
\[\lambda_c = \sqrt{\lambda_a^2 + \lambda_c^2 \cdot \cos(\theta - z)}\]

where x and y are the coordinates of the point for which the properties are calculated, and \(\lambda_a\) and \(\lambda_c\) are the conduc-

---

Fig. 5. (a) Crystallographic structure of the nodular graphite shown in STEM image. TEM image of cast iron with (b) flake and (c) vermicular graphite [36]. The vermicular particle grows initially nodular like, branches grow in a-direction.
tivities of the graphite in a- and c-directions, respectively (see Fig. 6).

The simulated propagation of heat in a single graphite nodule corresponds well with the model proposed by Hasse [37]. However, it is very time-consuming to run the simulations when the conductivity in each graphite inclusion is defined with two different conductivities in each point. Comparing the heat and tension flux for the system with so defined conductivity with the flux for the system, where graphite conductivity is homogeneous, it was possible to approximate the anisotropic conductivity of the graphite nodule by a single isotropic value. For the following simulations an isotropic thermal and electrical conductivity of \( \lambda_{\text{sphere}} = 269 \, \text{W m}^{-1} \text{K}^{-1} \) and \( \sigma_{\text{sphere}} = 0.6 \times 10^8 \, \text{S m}^{-1} \), respectively, have been used for the ideal graphite nodule.

3.3. 2-D FE simulation of the electrical conductivity using the simple ellipse model

For the estimation of thermal and electrical properties cast iron was presented as a pearlite matrix and 100 graphite inclusion, representing 10% of the total surface fraction, \( V_T = A_P = 10\% \). Different graphite morphologies were represented by ellipsoids with different shape described by shape parameters: compactness \( C \) (for the definition see Ref. [7]), between 0 (for flake graphite) and 1 (for nodular graphite).

As the properties of the graphite particles are defined by crystal structure and thus by growth mechanisms (see Ref. [36]), they can be calculated from the fraction grown according to the mechanism for spherical particles and for flake graphite:

\[
\lambda_{\text{gr}} = \lambda_{\text{sphere}} \cdot V_{\text{sphere}} + \lambda_a \cdot V_a \quad (11)
\]

\[
\lambda_{\text{gr}} = \lambda_{\text{sphere}} \cdot V_{\text{sphere}} + \lambda_c \cdot V_c \quad (12)
\]

where \( \lambda_{\text{gr}} \) is the resulting thermal conductivity of the graphite particle. The electrical conductivity \( \sigma_{\text{gr}} \) was calculated analogously. Approximating the shape of the graphite inclusions with 2-D ellipsoids, it should be considered that their conductivity will be different for the case parallel (Eq. (11)) and orthogonal (Eq. (12)) to the flux direction. For the ideal nodular graphite: \( V_{\text{sphere}} = C \approx 1 \), for ideal flake graphite: \( V_a = V_c = 1 - V_{\text{sphere}} = 1 - C, \, C \approx 0 \).

The simulation considers graphite shape, which correlates with the microstructure basic characteristic \( S_T \), and variable properties of the graphite inclusions in accordance with their crystallographic structure. Thus, the tendency of the mean curve, representing the isotropic distribution of the graphite particles, for both properties is somewhat consistent with the predictions in the literature [38,39] (Fig. 7a) and the experimental results (Fig. 7b).

The literature values of thermal conductivity for cast iron with vermicular and flake graphite approach the simulation results for the parallel case. In the case of electrical conductivity, flake graphite inclusions oriented perpendicular to the flux serve as a considerable obstacle in the path of the electrons and thus decrease drastically the electrical conductivity of the cast iron.
Thus, it was shown that the 2-D model provides an efficient approximation for the microstructure composed of simple not interconnected particles (e.g. nodular graphite), underestimates the effective values of thermal conductivity and overestimates the electrical conductivity for the microstructures with complex interconnected phases (vermicular and flake graphite).

The reason for this discrepancy with the experimental values for cast iron is the consideration of only two-dimensional information in the simulated model. As it was already shown in Ref. [12], each of the graphite morphologies is characterized not only by particle shape and size but also by connectivity, which is specific for each type. Thus, this fourth basic characteristic ($K_f$) which provides the information about the spatial arrangement and interconnection of complex graphite particles has to be included in the estimation of the effective properties of cast iron.

### 3.4. 3-D FE simulation on FIB data

The performed FIB-tomographic analysis on the cast iron [25] made it possible to perform 3-D simulations on
the real data sets with the help of the GeoDict software [27]. The 3-D image of cast iron with flake graphite IB (see Ref. [12]) was used to estimate the effective properties.

To simplify the 3-D simulation problem, an anisotropic approximation of the anisotropic properties of graphite phase was done. As it was shown before, flake graphite grows in the $a$-crystallographic direction and approaches the ideal hexagonal graphite structure more than any other graphite type. Knowing that for the cast iron with flake graphite morphology heat transport occurs mainly through the graphite flakes, the highest graphite conductivity (Eq. (11)) was chosen for simulation of thermal properties. On the other hand, flake graphite having the lowest electrical conductivity of all microstructure constituents serves basically as an obstacle for the electrical flux. Thus, the lowest electrical conductivity of the graphite (Eq. (12)) was chosen for the simulation of electrical properties. To estimate the effective graphite properties, the simulations were performed for three different values calculated using $C = 0.2$; $0.1$; and $0$.

The other parameter, which considerably influences not only the correct microstructure characterization, but also the simulation of the material properties, is the resolution of the spatial images. If not a sufficiently large amount of voxels is used to describe a pore or object, the object characterization can be falsified. There will be not enough voxels to generate the reliable field of currents and thus to perform the correct simulation. Thus, analysis is required, in order to determine the sufficient resolution for the simulation.

3.4.1. Influence of the resolution

The influence of the resolution on the simulation of the effective properties was studied on images with artificially reduced resolution, and thus the size of the analyzed volume in voxel. The reduction of the size of the simulated volume is often desired, as the time required for simulation reduces and thus the efficiency rises. Fig. 8 shows that the reduction of the resolution influences considerably the quality of the image; discretization effect is clearly seen on the reconstructed surfaces (Fig. 8a and b). Starting from a certain point, where the fractal dimension of the integral of the mean curvature $d_{mf}$ (see Ref. [12]) exceeds its minimum value 1, the resolution is so low that the thin flakes cannot be correctly represented any more (Fig. 8c and d), which leads to the loss of the microstructural information.

The results of the simulations of the effective conductivities are summarized in Fig. 9 and Table 4. With the help of resampling it was possible to reduce the simulation time already between original image and after one resampling step in more than ten times (from approximately 10 min to approximately 1 min). After exactly one resampling step,

![Fig. 8. Flake graphite particles (a) original FIB image, voxel size 0.18 × 0.24 × 0.5 μm$^3$, (b) 2nd resampling step 5/4/2, voxel size ~ 0.96 μm$^3$, (c) 3rd resampling step 8/6/3, voxel size ~ 1.47 μm$^3$ and (d) 4th resampling step 11/9/4, voxel size ~ 2.06 μm$^3$. Resampling is done by multiplying the original voxel dimensions in μm by the respective coefficients x/y/z.](image-url)
the simulation provides comparable results with the results from the original image. But every further reduction of the resolution (increase of the voxel size) influences the results of the simulation considerably. This effect is higher for the thermal properties than for the electrical conductivities, due to different role of the graphite in the conducting system.

Hence, in order to optimize the simulation time and accuracy, the estimation of the effective properties can be performed on images with reduced resolution as long as all microstructural characteristics do not change. Fig. 9b also shows that the effect of the resolution is higher if the contrast between properties of the matrix and inclusions is higher.

3.4.2. Anisotropy

The certain anisotropy observed on the 3-D images of flake graphite and quantified using area and length of projections in all spatial directions (see Fig. 4 in Ref. [12]) has also influenced the physical properties. Graphite flakes are oriented to the certain extent parallel to the y-axis. Hence, in this direction they provide the best heat transfer and delay least of all the electron transport in the matrix (Table 4).

Comparing the results of the simulation with the literature and experimental values, the suggestion for the thermal and electrical conductivity of the flake graphite can be done. But one has to keep in mind that the simulation was performed on only one limited zone in the middle of the eutectic cell, and is not representative for the bulk volume of cast iron with flake graphite. As the concentration and the complexity of the graphite in the middle of the eutectic cell is the highest, the estimated properties of this local volume should be higher for the thermal and lower for the electrical conductivities. For the analyzed 3-D image of cast iron with flake graphite, this would mean that properties of the graphite approach 500 W m\(^{-1}\) K\(^{-1}\) and 100 S m\(^{-1}\) (i.e. \(C = 0\)).

Using the simulation program GeoDict it was possible with the help of FIB-tomographical images to estimate the local properties of the cast iron with flake graphite. As the real 3-D images were used for the simulation, it was possible to implement all microstructural characteristics: volume fraction (\(V_F\)), specific surface area (\(S_F\)), densities of the integrals of mean (\(M_F\)) and total curvature (\(K_F\)). It was shown that phase connectivity plays an important role for the estimation of the effective properties.

Considering the hierarchical character of the microstructure for the estimation of the bulk properties, it is important to know the spatial arrangement of phases and effective properties at each characteristic scale. Cumulative implementation of the different tomographic techniques with varying analyzed volume and resolution can provide complete information about such microstructures and hence the material properties. For example FIB-tomography with high resolution can provide the information about the microstructure at different positions in the eutectic cell and computed tomography the arrangement of the eutectic cells in the bulk volume. To simulate the bulk effec-

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**Table 4**

<table>
<thead>
<tr>
<th>C</th>
<th>Thermal conductivity (W m(^{-1}) K(^{-1}))</th>
<th>Electrical conductivity (10(^5) S m(^{-1}))</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>(x)</td>
<td>(y)</td>
</tr>
<tr>
<td>FG1</td>
<td>0.2</td>
<td>48.4</td>
</tr>
<tr>
<td></td>
<td>0.1</td>
<td>49.4</td>
</tr>
<tr>
<td></td>
<td>0</td>
<td>50.3</td>
</tr>
<tr>
<td>FG2</td>
<td>0.2</td>
<td>34.9</td>
</tr>
<tr>
<td></td>
<td>0.1</td>
<td>35.2</td>
</tr>
<tr>
<td></td>
<td>0</td>
<td>35.5</td>
</tr>
</tbody>
</table>
tive properties of the material, one could use the properties of the sub volumes which were precisely acquired from smaller regions with higher resolution. This procedure opens the way to study the influence of the microstructural changes (which can be simply simulated in the computer models) on the effective physical and mechanical properties of such hierarchical structures.

4. Conclusions

In this study, different graphite morphologies were thoroughly analyzed in order to understand the correlation between properties and microstructure. Graphite morphology is one of the main factors controlling the mechanical and physical properties of cast iron.

Tomographic characterization of the individual graphite inclusions combined with high-resolution analysis of chemical and structural composition of nodular, vermicular and flake graphite provided valuable information about their nucleation, growth and spatial arrangement.

Considering the anisotropic properties of the hexagonal graphite structure and knowing the growth mechanisms of graphite of different morphologies [36], the properties of the graphite inclusions were estimated. The cast iron microstructure was approximated with a simple 2-D model consisting of a pearlite matrix with graphite ellipsoids of different compactness and thus different conductivity. The simulations on this model underestimate the effective thermal conductivity and overestimate the electrical conductivity of the cast iron with vermicular and flake graphite. This is due to the fact that the 2-D model does not consider a very important characteristic of these two graphite morphologies — their connectivity.

The use of real tomographic data for the 3-D simulations of effective physical properties of the cast iron with flake graphite made it possible to implement all important microstructural parameters including connectivity. It was shown that phase connectivity is one of the determining factors for the material properties. Additionally, the influence of the resolution of the tomographic images on the estimation of the material properties was shown. The compromise between reasonable time and accuracy of the simulation can be achieved by the choice of a suitable resolution. The images can be resampled (the resolution decreased) as long as all of the microstructural characteristics do not change. In particular, the parameter: fractal dimension of the integral of the mean curvature should not deviate from its minimum value 1.

The successful implementation of 3-D data from FIB-tomography in simulation procedures offers a way to study the influence of microstructural changes, which can be readily simulated with computer models, on the effective physical properties of such complex microstructures. The ability to reproduce experimentally determined values by computer simulation allows a further interesting step. By modifying the microstructures via computer modeling, one should be able to define ideal structures through its optimization. These optimization procedures could create tailored composite structures which would in turn maximize a specific physical or mechanical property.

Acknowledgements

The research was sponsored by the German Federal Ministry of Education and Research within the project No. 03N3119. The authors would like to thank project partners H. Junk, D. Radebach, Halberg Guss GmbH, and R. Heinenmann, VW AG for kindly providing the sample material, colleagues of ITWM Kaiserslautern for the help with 3-D image analysis and 3-D simulations, and J. Fiscina for the help with electrical measurements.

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