Metallographic preparation of Cast iron

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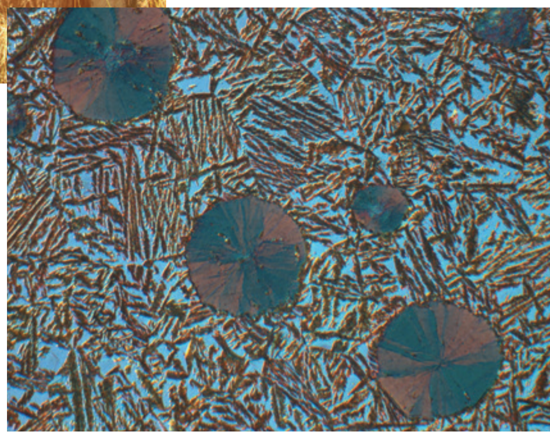
Application Notes

Iron is one of the most diverse metals and alloyed with carbon and other elements it offers an enormous variety of cast iron and steel alloys. Cast iron has been produced in China as early as 600BC and in Europe it was first known in the 14th century. With the development of coal fired blast furnaces the properties of iron improved and a better castability opened new fields of applications for products in every day life. With the industrialisation cast iron became an important construction material as buildings from the 19th century show: the domes of train stations, market halls, and green houses of botanical gardens, bridges and the Eiffel Tower still document the substantial application of cast iron during that time.

The term cast iron refers to those iron-carbon-silicon alloys which contain 2.5% - 4% carbon and usually 1-3% silicon. Cast iron is an important engineering material with a number of advantages, mainly good castability and machinability and moderate mechanical properties.

Because of its economical advantages cast iron is used for many applications

in the automotive and engineering industry. In addition, specific cast irons are the material of choice for sea water pump housings, rolling mill rolls and parts for earth moving equipment.
As the morphology of graphite has a major influence on the mechanical properties of cast iron, metallographic quality control of grey iron is an integral part of its production process. Using standard reference comparison charts and/or image analysis techniques, the morphology, size and distribution of the graphite is determined on an unetched, polished sample. Depending on the specification, the sample is then etched to check the structure of the matrix.



Austempered ductile iron, Beraha color etch, DIC, 500x

Difficulties during metallographic preparation

Cutting: White cast iron is very hard and therefore difficult to cut.
Grinding and polishing: Graphite is soft and retaining it in its true shape and size can be difficult. The matrix of ferritic and/or austenitic cast irons is prone to deformation and scratching.

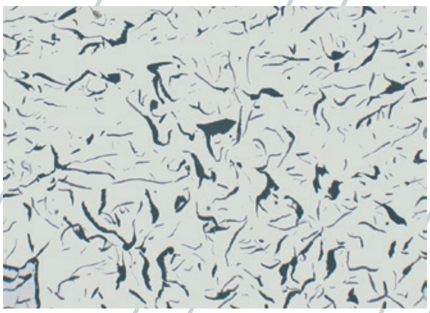


Fig.1: Grey iron with flake graphite, insufficient polish 200x

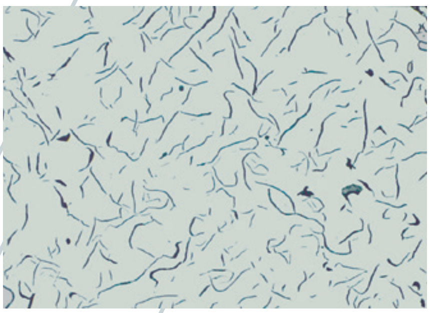


Fig. 2: Same as Fig.1, showing correct polish 200x

Solution

- Cubic boron nitride cut-off wheel
- Thorough diamond polishing on hard polishing cloths and final oxide polishing.

Production and application of cast irons

Fig. 6: Exhaust manifold, compacted graphite iron

Production

Cast irons are melted in a cupola- or induction furnace charged generally with pig iron, cast iron scrap, steel scrap and various additions. The alloy composition and the cooling rate will influence whether the iron will solidify grey or white.
A fast cooling rate results in a white solidification and the formation of iron carbide (Fe_3C or cementite). At the eutectoid transformation a fast cooling rate promotes the formation of pearlite, whereas a slow cooling rate promotes the formation of graphite and ferrite.
The microstructure of grey cast irons can have either a pearlitic and/or ferritic matrix with free graphite in the shape of flakes, nodules or temper carbon respectively. Through alloying and heat treatment the properties of cast iron can be adjusted for certain applications, for instance, alloying with molybdenum and nickel improves their heat and corrosion resistance.
In the following the individual cast irons will be briefly described and their major fields of application mentioned.

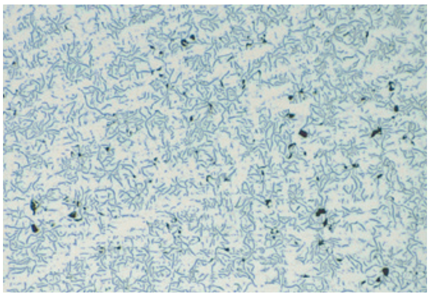


Fig. 3: Grey iron with fine flake graphite, unetched 100x

Grey iron with flake graphite (FG) has between 2.5-4% carbon, 1-3% silicon and 0.2-1% manganese. Carbon and silicon promote the formation of graphite flakes and ferrite. Phosphorus in small amounts increases the fluidity of grey iron. It also forms a ternary phosphorus eutectic called “steadit”, which constitutes a web like structure increasing the wear resistance. In the flake form, graphite provides notches within the metallic matrix and consequently lowers the tensile strength, especially when the flakes are very large. In unalloyed grey iron the best mechanical properties can be achieved with fine and

Fig. 4: Grey iron with flake graphite in pearlitic matrix 200x

evenly dispersed graphite flakes in a pearlitic matrix (see Figs. 3 and 4).
Grey iron has a high damping capacity, excellent sliding properties and thermal conductivity, which makes it suitable for machine bases, damping plates for pianos, engine blocks, flywheels, piston rings, brake discs and drums.

Ductile iron with spheroidal graphite (SG), also called nodular or spheroidal iron, is made from the same raw material as grey iron but requires higher purity. The melt should be free of Pb, As, Sb, Ti, and Al and have very little phosphorus and sulphur. By adding trace amounts of magnesium to the melt before casting, the graphite forms in a spherical shape instead of flakes.
Ductile iron has greater strength and ductility than grey iron of similar composition. Ductile iron has good machining qualities and is used for heavy duty gears, pistons, rolls for rolling mills, gear cases (Fig.10), valves, tubes and door hinges. Pearlitic ductile iron is the initial material for cam- and crankshafts which are surface hardened for wear resistance (Fig. 8).

Fig. 5: Filter head of ADI cast iron for the hydraulic system of a pressure die casting machine for plastics

Austempered ductile iron (ADI) is a ductile iron austenitized at 840-950°C and then quenched to 250-400°C where it is held until the matrix is changed to ausferrite. This is a mixture of needle-like ferrite and a carbon saturated retained austenite, which gives the ADI iron a high strength and ductility. The microstructure looks like bainite but has no carbides.
High-strength ADI irons are mainly used for wear resistant parts for heavy trucks, farm and earth moving equipment. Applications of ductile ADI irons are for parts with dynamic stress such as axle journals, gear drives, crankshafts, pull hooks and wheel hubs.

For making **Compacted graphite iron (CG)** the same raw material is used as for making ductile iron. By carefully controlling the amount of magnesium added to the melt for nodulizing approx. 80% of graphite is formed as compacted graphite, the rest as nodules.
The quality control of compacted iron is very important as the formation of graphite is critical. A slightly higher percentage of nodules can be tolerated, but the formation of flakes has to be avoided as they would lower or even eliminate the beneficial properties of the compacted iron.
Compacted graphite iron has better strength, ductility, alternating stress fatigue strength and higher resistance to oxidation than grey iron; and it is better to cast, easier to machine, has better damping qualities

Fig.7: Part of a wheel cassette of austempered ductile iron

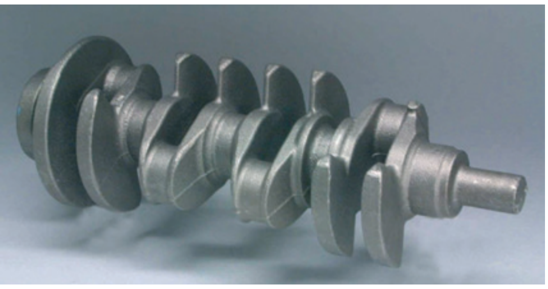


Fig. 8:
Crankshaft, ductile iron

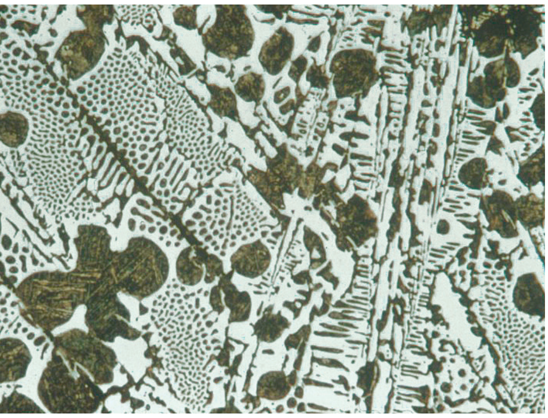
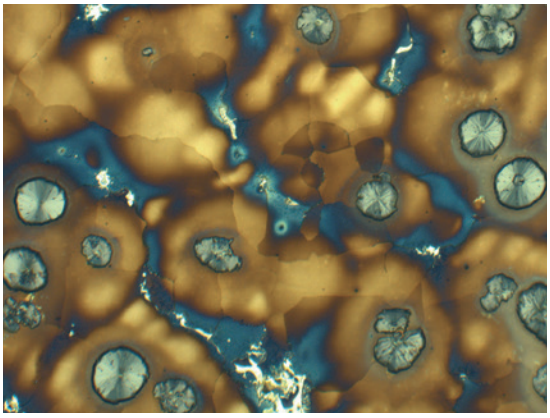


Fig. 9: White cast iron, pearlite with ledeburite 200x



Austenitic cast iron, etched with 3% Nital + modified Beraha's reagent 200x

and thermal conductivity and retains the shape better under temperature changes than ductile iron.
Applications: cylinder heads for high turning diesel motors, axle- and gear cases, exhaust manifolds (Fig. 6), housings of turbo chargers.

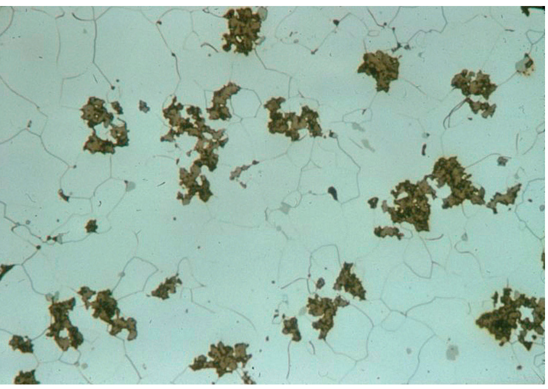
White cast iron contains 1.8-3.6% carbon, 0.5-1.9% silicon and 1-2% manganese.
A fast cooling rate prevents the precipitation of carbon as graphite. Instead the carbon, which is in solution in the melt, forms iron carbide (Fe_3C, also called cementite). The structure of white cast iron consists of pearlite and ledeburite (Fig. 9), a eutectic of pearlite, converted from austenite, and cementite. Ni-hard alloys (8-9% Cr, 5-6% Ni) have a martensitic matrix with chromium carbides.

White cast iron has a high compressive strength and alloyed versions have a good retention of strength and hardness at elevated temperatures. Due to its large masses of carbides, especially when alloyed, white cast iron has an excellent resistance against wear and abrasion. It is used for shot-blasting nozzles, rolling mill rolls, crushers, pulverizers and ball mill liners.

By chilling grey or ductile iron on the outside and letting it cool slowly inside, it is possible to produce parts with a hard surface of white cast iron with a ductile core (chilled cast).

Malleable iron with tempered graphite (TG)
Malleable iron is made by heat treating white cast iron. Through a two stage, long time heat treatment (tempering) white cast iron is converted to ferritic or pearlitic malleable iron. The carbon of the iron carbide first goes into solution, and through slow cooling then precipitates in irregular nodules called temper carbon. Pearlitic malleable iron can be hardened.
Increasingly malleable iron is replaced by nodular iron for economical reasons, especially since the fields of application are very similar.

Austenitic cast iron
Cast irons with at least 20% nickel and 1-5.5% chromium have an austenitic matrix with graphite in form of flakes or nodules. Austenitic cast iron can be an economic alternative to stainless steel as it is easier to cast and therefore suitable for precision casting of complicated shaped parts with a narrow wall thickness.
The main properties of austenitic cast irons are: corrosion resistance against sea water and alkaline media and high strength and scale resistance at high temperatures.
They are used specifically for applications in the maritime environment, for instance for large pump housings and other parts of desalination plants, or bushings and linings in chemical plants, compressors for aggressive gases, housings for gas turbines and turbo chargers.



Ferritic malleable iron 200x

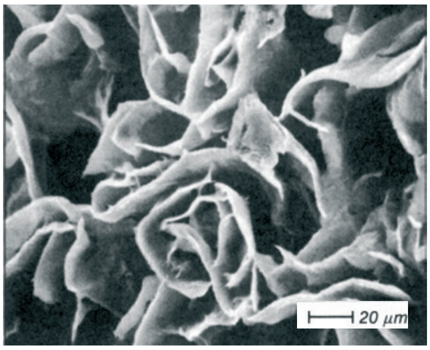
Fig.10: Differential housing of ductile iron

Difficulties in the preparation of cast iron

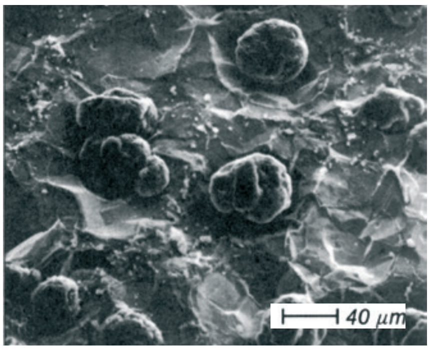
Alloyed white cast irons are very hard (HV 600) and can be difficult to cut, especially large sections. It is important to point out, that despite this hardness diamond cut-off wheels are not suitable for cutting white cast iron.

The main problem when preparing samples of cast iron is to retain the graphite in its original shape and size. Although in the microscope the image of the graphite is viewed as 2-dimensional, it should be remembered that it is actually 3-dimensional. This means that during grinding and polishing the appearance of graphite can slightly change, and that a certain percentage of graphite is cut very shallow with only a weak hold in the matrix. Therefore there is always a possibility that the graphite can not be completely retained. Especially very large flakes or agglomerations of flakes have the tendency to loose the graphite. Therefore graphite nodules can not always be retained or polished well.

In malleable cast irons graphite exists in the form of rosettes or temper carbon. This is a friable form of graphite and can be particularly difficult to retain during the preparation.



SEM image of grey iron with flake graphite



SEM image of ductile iron with graphite nodules

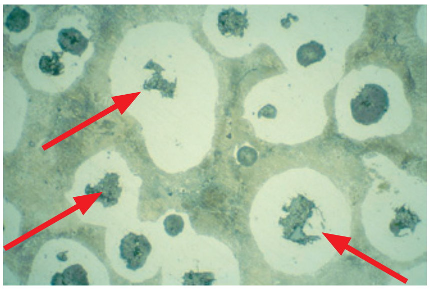


Fig.11: Insufficient polish leaves graphite nodules covered with smeared metal 200x

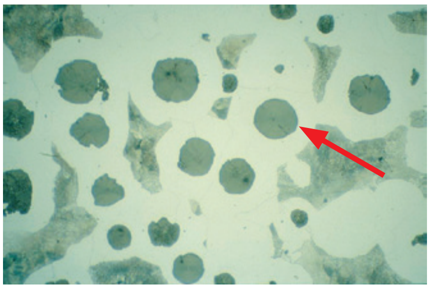


Fig.12: Correct polish shows shape and size of graphite nodules suitable for evaluation 200x

A common preparation error is the insufficient removal of smeared matrix metal after grinding, which can obscure the true shape and size of graphite (compare Figs.11 and 12). This is particularly prevalent in ferritic or austenitic cast irons that are prone to deformation and scratching. For these materials a thorough diamond and final polish is especially important.

The difficulties associated with the preparation of cast irons with graphite can be compounded in situations where metallography is an integral part of the casting line quality system.

Fig.13: Sample holder for semi-automatic polishing of quality control samples in cast line

Time constraints often make it difficult to maintain consistent preparation results using manual methods and often, due to the geometry of the test piece, automatic preparation is not a suitable alternative. However, as the design of the test pieces is usually arbitrary, their dimension and form can be changed in order to fit into an automatic system (Fig.13). This has been successfully carried out by some manufacturers who where then able to make the preparation more efficient and improve the evaluation of the graphite.

Most of the standard microscopic checks of cast irons are done with a magnification of 100x, which makes the graphite appear black. Only with higher magnifications can it be verified if the carbon is completely retained. Well polished graphite is grey (Fig.14).

Note: cast irons with graphite are not suitable for electrolytic polishing as the graphite is washed away by the electrolyte. However, if only a quick identification of the microstructure of the matrix is required electrolytic polishing and etching can be used (Fig.15).

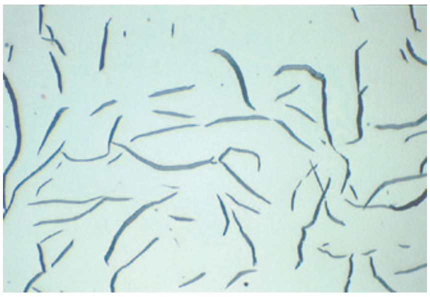


Fig.14: Well polished graphite flakes 500x

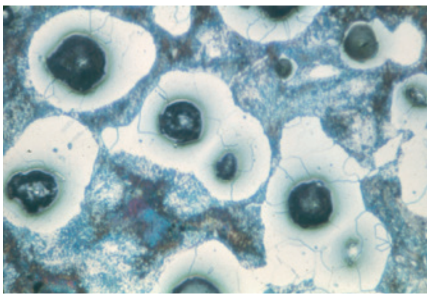


Fig.15: Ductile iron electrolytical polished and etched shows the pearlitic matrix and ferrite surrounding graphite. Graphite is washed away

Grinding

Step		PG	FG
Surface		MD-Piano 220	MD-Allegro
Abrasive	Type	Diamond	Diamond
	Size	#220	9 µm
Suspension/ Lubricant		Water	DiaPro Allegro/Largo 9
rpm		300	150
Force [N]/ specimen		30	30
Time (min)		Until plane	5

Polishing

Step		DP 1	DP 2
Surface		MD-Dac	MD-Nap
Abrasive	Type	Diamond	Diamond
	Size	3 µm	1 µm
Suspension/ Lubricant		DiaPro Dac 3	DiaPro Nap B 1
rpm		150	150
Force [N]/ specimen		40	30
Time (min)		4	1-2

Table 1: Preparation method for white cast irons

Alternatively DiaPro diamond suspension can be replaced by DP-Suspension, P, 9 µm, 3 µm and 1 µm respectively, applied with blue lubricant.

Grinding

Step		PG	FG
Surface		Foil/Paper	MD-Largo*
Abrasive	Type	SiC	Diamond
	Size	#220	9 µm
Suspension/ Lubricant		Water	DiaPro Allegro/Largo 9
rpm		300	150
Force [N]/ specimen		30	30
Time (min)		Until plane	5

Polishing

Step		PG	FG	OP**
Surface		MD-Dac	MD-Nap	OP-Chem
Abrasive	Type	Diamond	Diamond	Collodial Silica
	Size	3 µm	1 µm	0.04 µm
Suspension/ Lubricant		DiaPro Dac 3	DiaPro Nap B 1	OP-U NonDry
rpm		150	150	150
Force [N]/ specimen		30	20	10
Time (min)		4	1-2	1

Table 2: Preparation method for cast irons with graphite

**In cases where retention of graphite is very difficult, MD-Plan cloth can be tried for fine grinding.*

***This step is optional*

Alternatively DiaPro diamond suspension can be replaced by DP-Suspension P, 9 µm, 3 µm and 1 µm respectively, applied with blue lubricant

Recommendations for the preparation of cast iron

Cutting: For sectioning hard, white cast irons a cubic boron nitride wheel is recommended. For large sections automatic cutting is more efficient than manual cutting. For cutting cast irons with graphite it is recommended to select an aluminium oxide wheel according to the hardness of the cast iron to be cut.

Mounting: Quality control samples are usually prepared unmounted. For failure analyses samples it is recommended to use hot compression mounting. For soft to medium hard cast irons a phenolic resin (MulitFast) is recommended, for harder types of cast irons a reinforced resin (DuroFast) is more suitable.

Grinding and polishing:
Traditionally cast irons with graphite have been ground with silicon carbide Foil/Paper. In recent years diamond grinding has replaced silicon carbide for fine grinding most cast irons as it keeps the samples very flat and doesn't leave the graphite in relief (compare Fig.16 and 17).
Hard white cast irons and ADI irons can be plane ground with diamond (MD-Piano 220) and also fine ground with diamond (MD-Allegro, see table 1). Soft and medium hard cast irons with a ferritic, austenitic or pearlitic matrix are plane ground with silicon carbide Foil/Paper and fine ground with diamond on MD-Largo, see table 2).
For cast irons that tend to corrode during polishing it is recommended to use water free diamond suspension, A, and yellow lubricant. The preparation data are for 6 samples, 30 mm dia., mounted and clamped into a specimen holder.

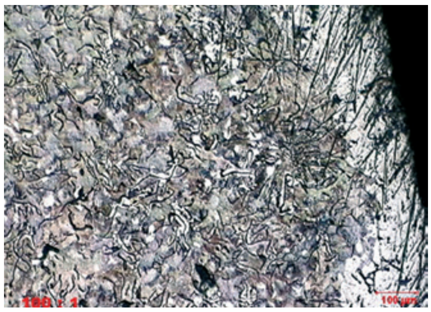


Fig.16: Grey iron prepared with fine grinding on silicon carbide Foil/Paper, still shows scratches

Fig.17: Same as Fig.16, prepared with fine grinding with diamond on MD-Largo, showing good edge retention

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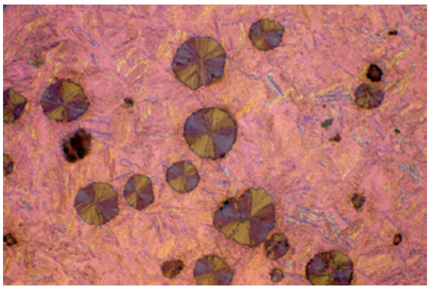
Cleaning: As many cast irons tend to corrode easily the cleaning of samples has to be fast and should always be carried out with cold water. Under no circumstances should the samples be left in contact with water. Thorough rinsing with ethanol and fast drying with a strong stream of warm air is recommended. If corrosion still occurs cleaning and rinsing with water free alcohol only is recommended.

Etching: Initially, the cast iron samples are microscopically examined unetched to evaluate shape, size and distribution of graphite and possible cast porosity. After this initial evaluation the sample is etched for microstructure with 1 - 3% Nital.
The following Beraha reagent can be used for colour etching and can be modified according to the alloy:

1000 ml water
200 ml hydrochloric acid
24 g ammonium difluoride
To 100 ml of this stock solution add 1 g potassium metabisulfite.
Note: *When working with chemicals the standard safety precautions have to be observed!*

Summary
Cast irons are ferrous alloys with mostly 2.5%-4% carbon and 1-3% silicon. The carbon is either present as graphite in grey irons or in form of iron carbide and alloy carbides in white cast iron. The difficulty in the metallographic preparation is to retain the true shape and size of the graphite in its flake, nodular or tempered form. During grinding the matrix is smeared over the graphite and unless it is followed by a very thorough diamond polish, the graphite is not shown in its true form. Especially cast irons with a soft ferritic matrix tend to smear and are prone to deformation and scratching. Plane grinding with silicon carbide Foil/Paper is recommended, followed by fine grinding and polishing with diamond. A brief final polish with colloidal silica is optional.

White cast irons are very hard and a cubic boron nitride cut-off wheel is recommended for sectioning.



Austempered ductile iron, etched with 3% Nital, pol. light *200x*

Note: do not use diamond cut-off wheels! Plane grinding, fine grinding and polishing are carried out with diamond.

Integrated into online casting, semi-automatic preparation equipment can achieve better results for a reliable and reproducible graphite evaluation than manual preparation.

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Acknowledgements

We wish to thank **CLAAS GUSS GmbH**, Bielefeld, Germany, for supplying sample material and giving permission for the reproduction of the foundry photo on page 1 and Figs. 5 and 7. Our special thanks go to Dr. Christine Bartels for her generous support and also to Ute Böhm.

We thank **GF Eisenguss GmbH**, Herzogenburg, Austria, for the permission to reproduce Figs. 6, 8 and 10.

We thank **Zentrale für Gussverwendung**, Düsseldorf, for the permission to reproduce the two SEM photos on page 4.

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28.02.2019 R05 / 62140306