**Efficient Sample Preparation And Analysis Of Ferrous Materials**

By: Matthias Pascher

**Introduction**

Many thousand years ago, ferrous materials were found to be quite rare and only accessible as debris of meteorites. Back then iron and its natural alloys were used in accessories like jewelry or cultural assets. With the beginning of metallurgical processes, first used for bronze, ferrous materials became more and more important and were used e.g. for agricultural machines.

The industrial revolution accelerated the growth of technical expertise and the knowledge of physicalchemical properties of materials. Blast furnace technology supported the development of metallurgical processes and ferrous materials, now applied for different defined chemical compositions with various alloying additions, like carbon or oxygen.

Today’s ferrous materials comprise a variety of several hundreds of alloys and its corresponding applications. Manufacturer and user need to rely on the materials properties and its use e.g. as a component of a multiple-part complex construction. To control the chemical composition and its mechanical properties or to investigate mechanical sample preparation and analysis was established since the beginning of serial production of ferrous materials.

This paper discusses how to process sample preparation and analysis on a variety of today's often used ferrous materials and compares its methods with state of the art preparation methods using the latest equipment and consumables.

**Background**

**Heat Treatment of Ferrous Materials**

Sample preparation and analysis was established for process control, failure analysis and R&D. Highly stressed materials need to have the corresponding mechanical properties to ensure its reliability, flexibility, elasticity or durability dependent on the application.

Heat treated steels are mainly used for high friction applications, like gear wheels, engine components, steering rods. Heat treating is defined as the controlled heating and cooling of solid metal or alloys to obtain specific properties by changing its microstructure locally. There are many different kinds of heat treatment processes, including normalizing, annealing, quenching, tempering or stress relieving. For example, gear wheels need to be hard and wear-resistant on the surface of gear teeth but elastically at the core to compensate irregularities or high wear during start-up processes. A common heat-treatment procedure is the so-called induction hardening where the surface of the work piece is heated up by alternating voltage induced eddy current. Direct quenching afterwards results in maintaining a martensitic microstructure which is significantly harder than the core material (ferrite/perlite) when using e.g. carburized steel.

Thus, heat treatment processes are important for a variety of materials ranging from automotive, aircrafts, house constructions, razor wheels and many more. Practically all steels can be strengthened by at least one type of heat treatment process. Non-ferrous alloys of Al, Cu, Ni, Mg or Ti can also be strengthened but not to the same degree and techniques as steels.

Another technique to enhance mechanical properties is a chemical-thermal surface treatment, such as nitriding or carburizing. In case of nitriding the work piece is held in a gas-atmosphere containing ammonia and hydrogen. Defined partial pressures in the process atmosphere lead to the formation of nitrides within the surface layer, resulting in a significant change in hardness from the surface to the core material. To control such hardness depths (diffusion zones of nitrides to be formed), optical measurements on cross-sections can be evaluated as mentioned above, with e.g. Micro-Vickers hardness tests. For both cases the sample needs to be prepared in a proper way to make such examinations possible.

**Efficient Sample Preparation Using Planarmet™ 300 for Ferrous Materials**

**Sample Preparation Process**

When starting to sample a specific work piece with suitable dimensions, abrasive cutters are widely used for sectioning. These cutters are available in different sizes and with different wheel diameters, dependent on the size of the work piece.

The right selection of the wheel is the first step to begin an efficient sample preparation. Dependent on the hardness of the material, an abrasive cut-on wheel with the right abrasive bonding has to be selected. A rule of thumb is that the bonding strength should decrease with increasing hardness of the material to be cut. Thus, abrasive particles will be released more often in a soft bonded wheel (for hard materials) than in more hard bonded wheels. The right wheel selection is a question of cutting quality, time and money. The abrasive particle removal of softer bonded wheels is higher than of hard bonded wheels. On the other hand, the time-per-cut process depends also on the bonding of the abrasives of the wheel. Therefore, choosing the right wheel is often a matter of quality, time and money. Table 1 shows recommended wheels for different ferrous materials and its correspondent parameters for the AbrasiMatic™ 300 using 12in [305mm] abrasive wheels.

The preparation method (in Table 2) shows the grinding and polishing consumables and parameters used for many metals and alloys.
When using a semi-automatic grinder and polisher the load per sample can be adjusted as well as head and base speed. Generally, 300rpm for the grinding and 150rpm for polishing stages is suitable for the base speed.

For initial coarse grinding stages, CarbiMet™ SiC grinding paper can be used which removes most of the deformation zone that was imposed during the cutting process. The second advantage is to achieve best edge retention for further preparation steps. This ensures grinding and polishing media is homogenous and evenly distributed across the sample surface when carrying out coarse-fine polishing stages. Normally, one SiC paper lasts for approximately not more than two minutes (dependent on sample material). Sometimes several papers have to be used to prepare the sample for the following steps. It has to be noted that soft materials are prone to embedment of loose SiC particles on the surface, therefore pressure and grinding times should be be adjusted properly when handling soft ferritic materials.

The relative rotation of specimen to base plate can also be adjusted. Generally, contra mode is more aggressive than complementary, thus leading to higher material removal rates. The subsequent polishing stages will reduce the deformation zone to a minimum and after the final polishing step the sample surface should be shiny and ready for investigation or etching.

**Improved Method For Regular And Heat Treated Steels In Production Environments**

The above method shows the principle of preparing most metals and alloys. In production environments of steel manufacturers and the correspondent hardening shops, where a high sample throughput needs to be achieved, Buehler has improved the preparation significantly by using latest equipment and consumables. The process cycle time can be significantly improved due to the increased material removal rate after the first grinding step. For the first and only grinding stage, the PlanarMet™ 300 was used (Figure 1) with an alumina stone as the base plate with a grit size of 120 that is capable of achieving removal rates of 400 microns per minute with ease.

After sectioning, the samples are fixed on a central force sample holder, depending on the sample size, they can be used in mounted or unmounted form. This sample holder will be used for all preparation steps, thus, saving time by avoiding taking samples in and out the holder between each step.

The key task is to clean the samples properly after each grinding and polishing step, to ensure that no abrasive particles of the previous step will contaminate the next step. Water and ethanol are mostly used as purifier.
removed. The following 9µm polishing step on UltraPad™ polishing cloth refines the scratch pattern and reduces the deformation zone to a limit. The durable VerduTex™ silk cloth used with 3µm diamond suspension will remove the deformation induced by the previous polishing step, whereas the final polishing step on ChemoMet™ with MasterPrep™ polishing media leads to a mirror-like, deformation-free sample surface. Until now, the samples were clamped in the sample holder. The mechanical sample preparation is completed and the sample can be further investigated by microscopy. For hardness testing above 500g load, the samples are ready for testing after the 3µm polishing step.

**Table 3: Improved Preparation Method for Soft/Medium (735HRC) Ferrous Materials Using PlanarMet 300.**

<table>
<thead>
<tr>
<th>Surface</th>
<th>Abrasive/Size</th>
<th>Load lb [N]/Specimen</th>
<th>Base Speed (RPM)</th>
<th>Head Speed (RPM)</th>
<th>Relative Rotation*</th>
<th>Time (min.s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alumina Grinding Stone</td>
<td>120 [P120] grit</td>
<td>7 [30]</td>
<td>fixed</td>
<td>120</td>
<td>&gt;&gt;</td>
<td>1.00</td>
</tr>
<tr>
<td>UltraPad</td>
<td>3µm</td>
<td>MetaDi Supreme Diamond*</td>
<td>7 [30]</td>
<td>150</td>
<td>60</td>
<td>&gt;&gt;</td>
</tr>
<tr>
<td>VerduTex</td>
<td>3µm</td>
<td>MetaDi Supreme Diamond*</td>
<td>7 [30]</td>
<td>150</td>
<td>60</td>
<td>&gt;&gt;</td>
</tr>
<tr>
<td>ChemoMet</td>
<td>0.05µm MasterPrep Alumina</td>
<td>7 [30]</td>
<td>150</td>
<td>60</td>
<td>&gt;&gt;</td>
<td>1.30</td>
</tr>
</tbody>
</table>

*Plus MetaDi Fluid as desired

For medium to hard ferrous materials, like heat-treated steels, the corresponding preparation method in Table 4 decreases to only three steps. After the initial grinding step using the PlanarMet 300, the next step is a 9µm coarse polishing carried out on the Apex™ Hercules S rigid grinding disc. The Apex Hercules S disc is suitable for steels and is usually used for polishing with 9 or 6µm for hard steels. After 4 minutes, the initial deformation and scratch pattern of the previous grinding step are removed and the last polishing step can be applied. Due to the higher hardness and resistance of the material to external impurities, the preparation can be finalized with a 3µm polishing step on VerduTex or MicroFloc cloths.

**Table 4: Improved Preparation Method for Medium/Hard (735HRC) Ferrous Materials Using PlanarMet 300.**

<table>
<thead>
<tr>
<th>Surface</th>
<th>Abrasive/Size</th>
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<td>&gt;&gt;</td>
<td>1.00</td>
</tr>
<tr>
<td>UltraPad</td>
<td>9µm</td>
<td>MetaDi Supreme Diamond*</td>
<td>7 [30]</td>
<td>150</td>
<td>60</td>
<td>&gt;&gt;</td>
</tr>
<tr>
<td>VerduTex</td>
<td>3µm</td>
<td>MetaDi Supreme Diamond*</td>
<td>7 [30]</td>
<td>150</td>
<td>60</td>
<td>&gt;&gt;</td>
</tr>
</tbody>
</table>

*Plus MetaDi Fluid as desired

As mentioned above, the samples can be used directly for hardness testing or microstructural investigation. When the samples are unmounted and exhibit sharp edges the MicroFloc polishing cloth is an excellent solution for the final polishing step for hard steels (Table 5). Due to the soft, long napped surface it covers the whole contact area of the sample; edges included, and will lead to an overall shiny surface finish.

**Table 5: Improved Preparation Method for Unmounted Medium/Hard (735HRC) Ferrous Materials Using PlanarMet 300.**

<table>
<thead>
<tr>
<th>Surface</th>
<th>Abrasive/Size</th>
<th>Load lb [N]/Specimen</th>
<th>Base Speed (RPM)</th>
<th>Head Speed (RPM)</th>
<th>Relative Rotation*</th>
<th>Time (mins)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alumina Grinding Stone</td>
<td>120 [P120] grit</td>
<td>7 [30]</td>
<td>fixed</td>
<td>120</td>
<td>&gt;&gt;</td>
<td>01:00</td>
</tr>
<tr>
<td>UltraPad</td>
<td>9µm</td>
<td>MetaDi Supreme Diamond*</td>
<td>7 [30]</td>
<td>150</td>
<td>60</td>
<td>&gt;&gt;</td>
</tr>
<tr>
<td>MicroFloc</td>
<td>3µm</td>
<td>MetaDi Supreme Diamond*</td>
<td>7 [30]</td>
<td>150</td>
<td>60</td>
<td>&gt;&gt;</td>
</tr>
</tbody>
</table>

Materials Selection

The new preparation methods were applied on the following ferrous materials, as shown in Table 6.

**Sampling And Preparation Procedure**

As previously mentioned, the right wheel selection ensures a clean and smooth cut surface. For the ferrous materials investigated three different abrasive wheels were used to achieve the best cut surface. After sectioning out representative pieces, sharp edges and possible burr from sectioning can be removed with a medium coarse SiC paper like 280 [P320] grit.

For the unmounted samples, these can be positioned and fixed directly in the sample holder. When the samples have to be mounted, a compression-mounting compound such as EpoMet™ is suitable due to its perfect edge retention. Before mounting, the parts should be degreased with ethanol to ensure proper contact between mounting compound and sample surfaces to avoid possible cracks or gaps between resin and sample. Note it is best practice to select a mounting compound that has similar removal rates as the sample. After the mounting, the specimens are fixed in a central force specimen holder (Figure 3).

Dependent on the sample properties (hard, soft, and unmounted) the preparation method is applied as stated in the corresponding table in section 3.2.

![Figure 3: Samples are clamped and aligned in a central force specimen holder.](image-url)
Common sample preparation methods deal with several steps. The final step consists mainly of a polishing procedure with finely dispersed abrasive particles in the submicron area. After this step, the sample surface is mainly shiny and mirror-like in case of metallic materials. First examination investigates the surface in the as-polished state in bright field for pores, cracks, inclusions and etc. Details of the microstructure can normally not be examined in the polished state. Therefore, for further investigation, metallographic etching is normally carried out to reveal grain boundaries, alloy constituent, banding or segregation and alloy deformation. Etching is broadly classified into two categories, those that change the sample surface is mainly shiny and mirror-like in case of metallic materials. First examination investigates the surface in the as-polished state in bright field for pores, cracks, inclusions and etc.

Some materials can be etched "optically", for example, by adjusting the illumination and reflected light beam, the surface of certain materials exhibit different characteristics. Optical manipulation results in different illumination techniques that affect contrast and subsequent level of detail obtained from the surface.

These techniques include:
- Darkfield microscopy: reflected light of uneven surfaces is detected (e.g. cracks, half-opaque phases can be determined).
- Phase contrast microscopy: mainly used for transmitted light microscopy (e.g. differences of density and refractivity can be determined).
- Differential interference contrast (DIC) microscopy: image can be plastically mapped (e.g. small differences in heights)
- Polarized light microscopy: Determination of anisotropic phases

While the sample surface remains chemically untreated by "optical" etching, quantitative evaluation of the microstructure of ferrous materials (such as grain size determination and area fractions of different phases) can mostly be achieved by chemical etching of the surface with different etchants.

A third possible etching technique is physical etching. Reactive or cathodic sputtering are two examples of physical etching. This is especially useful for weak reflecting samples like certain ceramics. For such methods, special etching chambers and defined vacuum need to be available to ensure proper etching. Thermal etching is also a physical etching method that is often used for ceramics or Titanium. Here, diffusion processes are activated to achieve a state of equilibrium between surface and interface energy, thus, the microstructure will become visible.

In this work, electrochemical etching methods are used for microstructure analysis. Ferrous materials exhibit a huge variety of alloys. Every ferrous alloy has its own characteristics and properties. In R&D, but also in production environments, quality and therefore microstructural investigations need to be accessible, e.g. contrasting of the microstructural detail that is of interest has to be applicable.

For this reason many etchants are available that ensure contrasting of the microstructure but also coloring of distinct crystallographic orientations of grains. Table 7 lists the applied etchants for the material selection.

The micrographs in Figures 4–18 show the results applying the latest preparation method on the different ferrous materials.

### Table 7: Common etchants for ferrous materials [2].

<table>
<thead>
<tr>
<th>Material</th>
<th>Etchant</th>
<th>Application</th>
</tr>
</thead>
<tbody>
<tr>
<td>Low alloyed steels, cast iron, welds, diffusionzones</td>
<td>Nital (1-10% nitric acid, ethanol)</td>
<td>Seconds to minutes, wet etching mostly used</td>
</tr>
<tr>
<td>High alloyed steels, austenitic castings, ferrite</td>
<td>V2A etchant (15-20% hydrochloric acid, 1-5% nitric acid)</td>
<td>Seconds to minutes, can be heated up to 70°C max., wet etching</td>
</tr>
<tr>
<td>Stainless steel, austenitic CrNi steels</td>
<td>Beraha II (32% hydrochloric acid, ammonium bifluoride, potassium metabisulfite)</td>
<td>Coloring, wet etching</td>
</tr>
<tr>
<td>Cementite, diffusionzones</td>
<td>Klemm I (sodium thiosulfate, potassium metabisulfite)</td>
<td>1-2 minutes, wet etching, coloring</td>
</tr>
</tbody>
</table>
Figure 4: Martensitic Steel with retained austenite (white) etched with Klemm I. Magnification 200x.

Figure 5: Soft iron, Ferrite grain boundaries with some nodular carbides. Etched with 3% Nital. Magnification 200x.

Figure 6: Soft iron, ferrite grains colored with Klemm I etchant. Magnification 200x.

Figure 7: Normalized steel C35/1035, lamellar Perlite with Ferrite. Etched with 3% Nital. Magnification 500x.

Figure 8: Normalized steel C35/1035, lamellar Perlite with Ferrite. Etched with Klemm I. Magnification 500x.

Figure 9: Heat-treated steel C45, hardened, surface decarburization. Etched with 3% Nital. Magnification 200x.

Figure 10: Spheroidised steel C45/1045, Ferrite grains and nodular Cementite. Etched with 3% Nital. Magnification 500x.

Figure 11: Machining steel 9SMn28K. MnS particles grey. Etched with 3% Nital. Magnification 200x.
Figure 12: Nitrided layer, plated with aluminum foil. The porous layer inside the compound layer is well visible. Magnification 200x.

Figure 13: Nitrided layer after etching with 3% Nital. Magnification 200x.

Figure 14: Nitrided layer. Diffusion zone after etching with 3% Nital. Magnification 100x.

Figure 15: High-grade steel C100. Etched with 3% Nital. Magnification 500x.

Figure 16: Stainless steel X5CrNi18-10. Etched with V2A at 60°C. Magnification 200x.

Figure 17: Stainless steel X5CrNi18-10. Ti (C,N) particles (orange). Magnification 1000x.

Figure 18: Martensite revealed with 3% Nital. Magnification 200x.
Hardness Testing

Hardness testing is widely spread in the field of process control of heat treatment and to evaluate surface hardness and hardness depths of surface engineered materials such as nitrided steels. In case of nitrided or carbonitrided steels, the Nitriding Hardness Depth (NHT, german for “Nitrierhärtetiefe”, acc. to DIN 50190-3) is applied. Therefore, a row of indents with defined indent spacing are placed perpendicular to the sample surface and the hardness of the different formed zones is measured and can be displayed as a function of depth. Typical loads used for this application are below one kilogram, mainly 500grams. Figure 17 shows the principle behind the NHT measurement.

For the NHT evaluation, certain parameters need to be adjusted:
- Limit hardness: measured as core hardness or determined by technical drawings.
- Limit hardness + 50HV: hardness that should be met to interpolate the NHT value.
- Indent spacing and edge distance: should be according to the latest DIN EN ISO 6507-1.

A nitrided surface layer consists mainly of two zones: the compound layer and the precipitation zone. While the compound layer is only few microns in its thickness and consists mainly of iron nitrides (α-N-ferrite, γ′-Fe₄N, ε-Fe₂Nₙ₋₁), the precipitation zone exhibits different precipitated nitrides after cooling or special nitrides formed during the nitriding process [3]. This second zones with its different nitrides is responsible for the hardness increase due to the induced stresses of the lattice mismatch between nitrides and host lattice. Therefore, nitrided materials are used for high wear sensitive parts, like gear wheels in engines.

Hardness evaluation methods, like NHT or CHD, are nowadays highly automated on modern Vickers hardness testing machines. Options such as autofocus and automeasure (sometimes even with autoillumination) algorithms ensure high repeatability and significantly reduced process times. A fully-automated Vickers hardness tester, as shown in Figure 20, was used for this work.

Figure 21 shows a micrograph of Micro Vickers indentations of a typical NHT measurement. The indents were carried out with 300g loads. Two rows where selected to ensure that all indents can be places with respect to the indent distances according to the ISO 6507-1. Increasing indent sizes are correlated to the decrease of hardness.

Conclusion

Efficient sample preparation in production environments needs to be reliable, repeatable and fast to perform. Especially for high sample volume preparation sequences can be improved when changing certain preparation steps within the procedure. After sectioning samples for metallographic investigation, consequently flattening and reduction of the heat affected zone from sectioning need to be performed. Using the PlanarMet™ 300 stone grinder as initial grinding step reduces cycle times while maintaining perfect surface qualities like flatness, homogenous material removal and scratch pattern.

References