TECHNotes

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Efficient Sample Preparation and Analysis of Ferrous Materials

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Introduction

Many thousands of years ago ferrous materials were mainly found in naturally occurring forms, such as debris from meteorites. The first known working of iron was in c.1500 BCE by the Hittites (centered in what is now Turkey). It took another 500 years before the process of early steel manufacture in simple furnaces was discovered, and that quenching could improve hardness. This sparked the growth of steel and the end of the Bronze Age.

As late as the 1800's, processing of steel was arduous. Bulk processing was possible, mainly using the "cementation" process - heating bars of iron with charcoal for days on end, a process that was both inefficient and inconsistent. In 1856 Bessemer developed his converter, closely followed by the Siemens Open Hearth process, allowing the removal of carbon and other elements from the iron and the true control of the elements in steel began.

Today, there are several hundred varieties of steel with as many applications. To meet modern demands, the chemistry and properties of the steel need to be tightly controlled. Metallographic processes and analysis techniques have needed to develop in line with these increasingly demanding applications.

This paper discusses sample preparation and analysis on a variety of today's often used ferrous materials 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 reliability, flexibility, elasticity or durability depending on the application.

Heat treatment is generally defined as the controlled heating and cooling of solid metal or alloys to obtain specific properties by changing the microstructure. There are many different components to heat treatment, including normalizing, annealing, quenching, tempering and stress relieving. These treatments can affect many properties simultaneously, but improving one can be detrimental to others.

In some cases components need to have varying properties in different areas and in these cases localized techniques can be used. For example, gear wheels need to be hard and wear-resistant at the surface, but maintain the ductility and toughness to endure high loads without breaking. Surface treatments to create this hard layer involve using heat and exposure to chemical elements such as carbon or nitrogen to modify the surface properties. These include processes such as carburizing, nitriding and induction hardening.

Induction hardening uses a high frequency alternating current to rapidly heat the surface of the work piece. Quenching results in a martensitic structure being maintained in that area, which is significantly harder than the core material. The depth of the hardened layer depends on the operating frequency used.

Carburizing and nitriding involve heating the sample in an environment rich in the desired element, changing the chemistry at the surface, and as a result modifying the microstructure. In these cases the hardening depth depends on time, temperature and the concentration of the required elements.

Checking that surface treatments have been correctly performed is a vital quality control step. Both hardness testing and optical examination of the microstructure are used. Both of these require proper metallographic preparation to ensure accurate results.

Sample Preparation

Sectioning

The first step in the preparation of most samples for metallographic examination is the removal of a representative sample from a larger component. It is important to ensure that the sample is not exposed to excessive heat or mechanical damage during sectioning. Saws designed specifically for metallographic purposes should be used. The machine itself is usually selected on the basis of capacity and level of automation. Specialist abrasive wheels are normally used, as they provide excellent cutting speeds with minimal damage.

The selection of the correct abrasive wheel is critical to ensuring an undamaged specimen, as well as optimizing speed. The abrasive type should be selected to suit the material being cut - alumina is most effective for ferrous materials. In addition, the strength of the bond holding the abrasive in the wheel is very important. Hard materials require a sharp abrasive in order to cut effectively. If the abrasive is held too strongly in the wheel, it will not wear and expose fresh particles. This can lead to glazing of the wheel, extended cutting times and excessive heat or deformation in the part. Conversely, too soft a bond can lead to excessive wheel wear. To simplify selection, Buehler provides a range of wheels from which the user can readily select. If cutting components with a variable properties, such as surface treated steels, a wheel suited to the hardest material present should be selected. Table 1 shows recommended wheels for different ferrous materials and corresponding parameters for the AbrasiMatic™ 300 using 12in [305mm] abrasive wheels.

Table 1: Recommended Wheels and Cutting Parameters, based on 12in [305mm] AbrasiMatic 300 Cutter.

Wheel Type	Material	Avg. Feedrate	Max. Feedrate
Soft ferrous metals 50-350HV (5-35HRC)	1.25in [32mm] Soft steel 20HRC	0.019in [0.5mm]/ sec.	0.031in [0.8mm]/ sec.
Medium soft ferrous metals 350-500HV (35-50HRC)	1.25in [32mm] Medium hard steel 30HRC	0.019in [0.5mm]/ sec	0.047in [1.2mm]/ sec.
Medium hard ferrous metals 500-700HV (50-60HRC)	1.25in [32mm] hard steel 50HRC	0.019in [0.5mm]/ sec	0.031in [0.8mm]/ sec.
Hard ferrous metals 600-700HV (55-60HRC)	1.25in [32mm] Very hard steel 60HRC	0.015in [0.4mm]/ sec	0.023in [0.6mm] / sec.
Soft ferrous metals 50-350HV (5-35HRC)	1.25in. [32mm] Stainless Steel	0.019in [0.5mm]/ sec	0.031in [0.8mm]/ sec.
Medium soft ferrous metals 350-500HV (350-500HRC)	1.25in. [32mm] Cast iron	0.011in [0.3mm]/ sec	0.023in [0.6mm] / sec.

Standard Metallographic

Mounting specimens prior to metallographic preparation can improve the quality in many ways. Standardizing the sample size improves reproducibility and allows multiple samples to be held quickly and easily in the preparation machine. In addition, mounted samples significantly reduce consumable usage during preparation. When examination is to be done at the edges of the specimen, mounting is even more critical. High quality edge retention mounting media is recommended, as this provides the best protection to the specimen while minimizing shrinkage gaps that can otherwise lead to relief and contamination issues. The use of compression mounting machines such as the SimpliMet range, and mineral filled epoxy mount material such as EpoMet, is recommended for all surface hardened applications. When compression mounting is not an option, use a minimal shrinkage high hardness castable mount such as Varidur 3003.

For initial coarse grinding stages, CarbiMet™ SiC grinding paper can be used, to achieve planarity and remove any residual deformation from sectioning. Normally, one SiC paper lasts for not more than two minutes (dependent on sample material). Sometimes several papers have to be used before the sample is ready for subsequent steps. These will sequentially reduce the level of mechanical deformation in the sample, until a reflective damage-free surface is revealed.

Improving Efficiency for Regular and Heat Treated Steels using Planar Grinding

Efficient Sample Preparation Using Planarmet™ 300 for Ferrous Materials

In some environments, especially high throughput production, rapid processing of samples is critical. Process cycle time can be significantly improved by tailoring preparation more closely to the sample type, and by the use of specialized machines in addition to a traditional grinder-polisher.

The use of a planar grinder allows initial grinding processes to be done far more rapidly than with SiC paper, by using a high speed abrasive wheel. The speed allows for high removal rates, and deformation is controlled by optimizing wheel speed, effective cooling, and 'dressing' the stone to keep it at maximum cutting efficiency.

After sectioning, the samples are fixed in a central force holder (Figure 3). This type of holder can be used in both the PlanarMet and EcoMet Automet, so no need to lose time switching holders between stages. Holders are available for mounted and unmounted samples in a range of sizes.

Table 2: Generic Contemporary Preparation Method for Many Metals and Alloys

Surface	Abrasive/ Size	Load lb. (N)/ Specimen	Base Speed (RPM)	Relative Rotation*	Time (min:s)
CarbiMet	120 [P120] 240 [P280] grit SiC water cooled	6 (27)	300	>>	Until plane
UltraPad	9µm MetaDi Supreme Diamond*	6 (27)	150	><	05:00
TriDent	3µm MetaDi Supreme Diamond*	6 (27)	150	>>	04:00
ChemoMet	0.02-0.06µm MasterMet colloidal Silica	MasterMet colloidal Silica	150	><	02:00

**Plus MetaDi Fluid as desired*
>> = Complimentary (platen and specimen holder rotate in the same direction)
>< = Contra (platen and specimen holder rotate in opposite directions)

Samples that do not require mounting can be fitted directly into the specimen holder. If mounting, however, the samples should be degreased to ensure proper contact between the mounting compound and sample surface, to help ensure there are no gaps.

Figure 1: PlanarMet™ 300 stone grinder.

Dependent on the properties of the material being prepared (hard, soft, and unmounted) the preparation method was applied as stated in the corresponding tables 3. A 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 to clean the samples during the steps and as a final cleaning step.

Figure 2: Highly durable grinding wheel for initial grinding (alumina).

Tables 3–5 illustrate the preparation method for soft/medium and medium/hard ferrous materials, as well as a method for unmounted samples.

Table 3 shows the preparation method for soft/medium ferrous materials. For all the preparation methods, the first step after sectioning was to grind the samples for one minute on the PlanarMet™ 300. Using an alumina wheel with 120 [P120] grit size (Figure 2) Parameters, such as load, head speed (base speed is fixed to 1500rpm), the relative rotation of head and base, dressing cycle and dressing depth can be adjusted to enable steady and continous removal, and optimize consumables use. Dependent on the hardness of the material the removal rates achieved (MRR) varied between 250 and 800µm/min with a uniform scratch pattern.

Table 3: Improved Preparation Method for Soft/Medium (~35HRC) Ferrous Materials Using PlanarMet 300.

Surface	Abrasive/ Size	Load lb. [N]/ Specimen	Base Speed (RPM)	Head Speed (RPM)	Relative Rotation*	Time (min.s)
Alumina Grinding Stone	120 [P120] grit	7 [30]	fixed	120	>>	1:00
UltraPad	9µm MetaDi Supreme Diamond*	7 [30]	150	60	><	4:00
VerduTex	3µm MetaDi Supreme Diamond*	7 [30]	150	60	>>	3:00
ChemoMet	0.05µm MasterPrep Alumina	7 [30]	150	60	><	1:30

**Plus MetaDi Fluid as desired*
>> = Complimentary (platen and specimen holder rotate in the same direction)
>< = Contra (platen and specimen holder rotate in opposite directions)

The first step consists of grinding for one minute on the PlanarMet™ 300. After the grinding step, the samples were even and the main part of the deformation zone induced by cutting was removed. The following 9µm polishing step on UltraPad™ polishing cloth refined the scratch pattern and reduced the deformation zone. The durable VerduTex™ silk cloth used with 3µm diamond suspension removed the deformation induced by the previous polishing step, whereas the final polishing step on ChemoMet™ with MasterPrep™ polishing media provided a deformation-free sample surface. Until now, the samples were clamped in the sample holder continuously.

Table 4: Improved Preparation Method for Medium/Hard (>35HRC) Ferrous Materials Using Planar Met 300.

Surface	Abrasive/ Size	Load lb [N]/ Specimen	Base Speed (RPM)	Head Speed (RPM)	Relative Rotation*	Time (mins)
Alumina Grinding Stone	120 [P120] grit	7 [30]	fixed	120	>>	1:00
UltraPad	9µm MetaDi Supreme Diamond*	7 [30]	150	60	><	4:00
VerduTex	3µm MetaDi Supreme Diamond*	7 [30]	150	60	>>	3:00

**Plus MetaDi Fluid as desired*
>> = Complimentary (platen and specimen holder rotate in the same direction)
>< = Contra (platen and specimen holder rotate in opposite directions)

The mechanical sample preparation is completed and the sample can be further investigated by microscopy.
For medium to hard ferrous materials, like heat-treated steels, the corresponding preparation method in Table 4 decreased to only three steps. After the initial grinding step using the PlanarMet 300, the next step was a 9µm coarse polish carried out on the UltraPad polishing cloth. The UltraPad polishing cloth is suitable for steels and provides excellent flatness, as well as high removal rates. It 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 5: Improved Preparation Method for Unmounted Medium/Hard (>35HRC) Ferrous Materials Using PlanarMet 300.

Surface	Abrasive/ Size	Load lb [N]/ Specimen	Base Speed (RPM)	Head Speed (RPM)	Relative Rotation*	Time (mins)
Alumina Grinding Stone	120 [P120] grit	7 [30]	fixed	120	>>	01:00
UltraPad	9µm MetaDi Supreme Diamond*	7 [30]	150	60	><	04:00
MicroFloc	3µm MetaDi Supreme Diamond*	7 [30]	150	60	>>	04:00

**Plus MetaDi Fluid as desired*
>> = Complimentary (platen and specimen holder rotate in the same direction)
>< = Contra (platen and specimen holder rotate in opposite directions)

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, reducing the preparation time, especially at the unsupported edges of the specimen.

Results of Accelerated Metallographic Preparation

Materials Selection

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

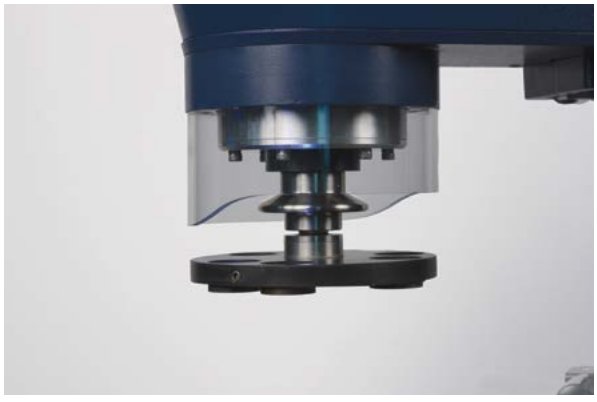

Figure 3: Samples are clamped and aligned in a central force specimen holder.

Table 6: Material Selection and Correspondent Chemical Composition Evaluated by Cast Analysis [4].

Abbreviation	Material No.	ASTM	C [wt.%]	Si [wt.%]	Mn [wt.%]	P [wt.%]	S [wt.%]	Cr [wt.%]	Mo [wt.%]	Ni [wt.%]	Al [wt.%]	Cr+Mo+Ni [wt.%]	Cu [wt.%]	other
100Cr6	1.3505	6440 K (AMS)	0.93–1.05	0.15-0.35	0.25-0.45	≤ 0.025	≤ 0.015	1.35-1.60	≤ 0.10		≤ 0.050		≤ 0.30	O≤ 0.0015
M2	1.1003	Soft-iron	≤0.030	traces	≤ 0.030	≤ 0.010	≤ 0.025	≤ .035			traces			
C35	1.0501	1040 (SAE)	0.32-0.39	≤0.40	0.50-0.80	≤ 0.045	≤ .045	≤ 0.40	≤ 0.10	≤ 0.40		≤ 0.63		
C45	1.0503	1043 (SAE)	0.42-0.50	≤ 0.40	0.50-0.80	≤ 0.045	≤ 0.045	≤ 0.40	≤ 0.10	≤ 0.40		≤ 0.63		
9SMn28K	1.0715	1213 (SAE)	≤ 0.14	≤ 0.05	0.9-1.3	≤ 0.11	0.27-0.33							
42CrMo4	1.7225	A 372	0.30-0.45	≤ 0.40	0.60-0.90	≤ 0.025	< 0.035	0.90-1.20	0.15-0.30					
C100S	1.1274	1095 (AISI)	0.95-1.05	0.15-0.35	0.30-0.60	≤ 0.025	≤ 0.025	≤ 0.04	≤ 0.10	≤ 0.40				
X5CrNi1810	1.4301	304 (SAE)	≤ 0.07	≤ 1.0	≤ 2.0	≤ 0.045	≤ 0.015	17.0-19.5		8.0-10.5				N≤ 0.11
25CrMo4	1.7218	4130 (SAE)	0.22-0.29	≤ 0.40	0.60-0.90	≤ 0.025	≤ 0.035	0.90-1.20	0.15-0.30					(Pb)

Image Analysis Techniques

Contrasting of the Microstructure

Details of the microstructure of ferrous materials can normally not be examined in the polished state. Therefore, for further investigation, metallographic etching is carried out to reveal details such as grain boundaries, alloy constituent, banding or segregation and alloy deformation.

A wide variety of examination techniques are available to the metallographer, and their use depends on both the material being examined and the detail being looked at.

Some important optical techniques are available to metallographers that assist greatly in the analysis. These include:

- Reflected light microscopy: standard technique for metallography that shows differences in reflectance of the surface as contrast in the image
- Darkfield Microscopy: only scattered light is observed: recommended for crack detection and highlighting edges
- Differential Interference Microscopy: highlights the topography of a surface, allowing exceptionally fine details to be observed
- Polarized light: uses two polarizers in the light path to create contrast through interference effects at the specimen surface. It can be used on unetched specimens of many non-cubic materials, and is necessary for analysis of color etching such as used in this report. A waveplate is necessary to introduce color, otherwise contrast is seen in grayscale.

In this work, both chemical etching and surface interference techniques (color etching) have been used to generate images of the microstructure. The advantage of color etching is that individual phases can be color coded, making identification easier. However, color etching is particularly sensitive to any deformation remaining in the surface of the specimen, and so it will only work properly when a sufficient quality of sample preparation is used.

It is important to realize that different etchants will contrast different parts of the microstructure, and not necessarily all of them. Therefore, etchants should be selected that are suitable for both the material being investigated and the structure to be observed. The Buehler SumMet handbook [1] lists many of the common etchants used in metallography, but multiple references to good etchant selection are available [2]
Table 7 gives some examples of etchants that may typically be used for the materials in this report.

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].

Material	Etchant	Application
Low alloyed steels, cast iron, welds, diffusionzones	Nital (1-10% nitric acid, ethanol)	Seconds to minutes, wet etching mostly used
High alloyed steels, austenitic castings, ferrite	V2A etchant (15-20% hydrochloric acid, 1-5% nitric acid)	Seconds to minutes, can be heated up to 70°C max., wet etching
Stainless steel, austenitic CrNi steels	Beraha II (32% hydrochloric acid, ammonium biflouride, potassium metabisulfite)	Coloring, wet etching
Cementite, diffusionzones	Klemm I (sodium thiosulfate, potassium metabisulfite)	1-2 minutes, wet etching, coloring

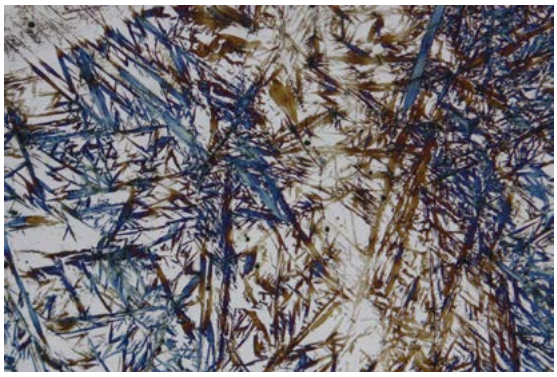

Figure 4: Martensitic Steel with retained austenite (white) etched with Klemm I. Magnification 200x viewed with polarized light and sensitive tint..

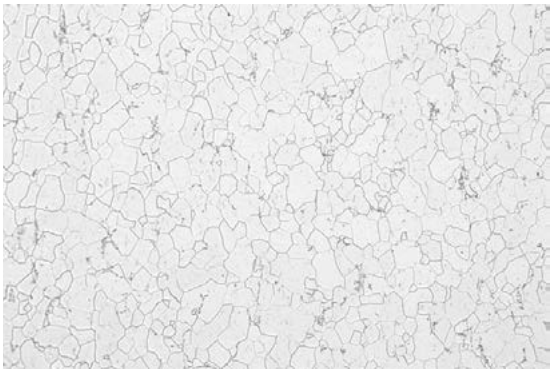

Figure 5: Soft iron, Ferrite grain boundaries with some nodular carbides. Etched with 3% Nital. Magnification 200x.

Figure 6: Soft iron, ferrite grains colored with Klemm I etchant. Magnification 200x viewed with polarized light and sensitive tint..

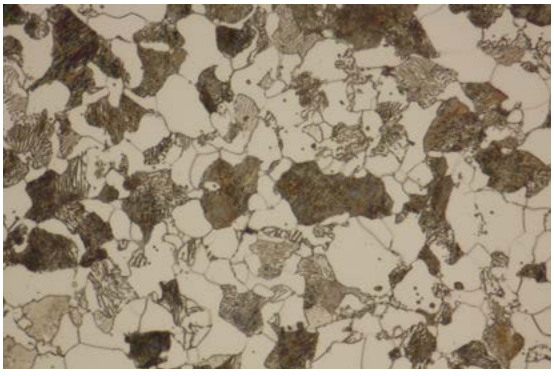

Figure 7: Normalized steel C35/1035, lamellar Perlite with Ferrite. Etched with 3% Nital. Magnification 500x.

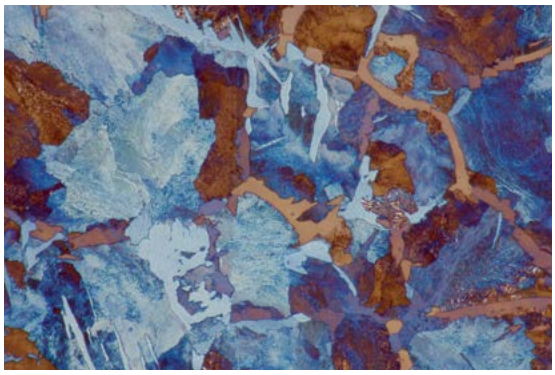

Figure 8: Normalized steel C35/1035, lamellar Perlite with Ferrite. Etched with Klemm I. Magnification 500x viewed with polarized light and sensitive tint..

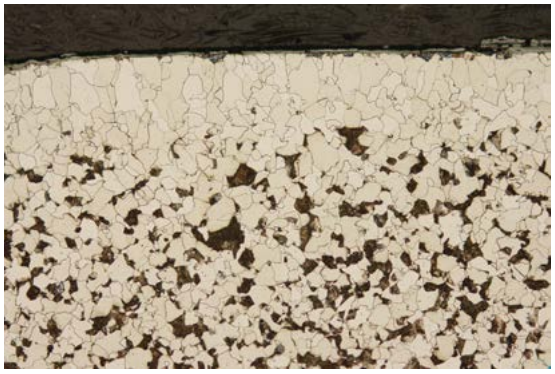

Figure 9: Heat-treated steel C45, hardened, surface decarburization. Etched with 3% Nital. Magnification 200x.

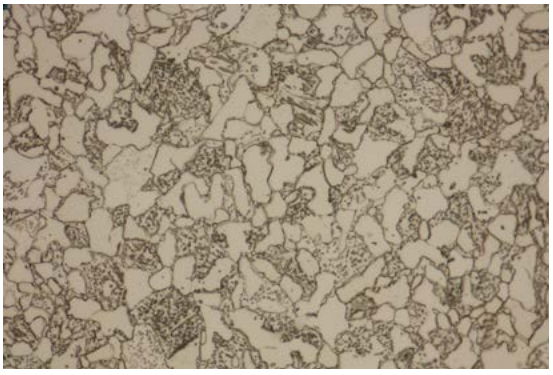

Figure 10: Spheroidised steel C45/1045, Ferrite grains and nodular Cementite. Etched with 3% Nital. Magnification 500x.

Figure 11: Machining steel 9SMn28K. MnS particles grey. Etched with 3% Nital. Magnification 200x.

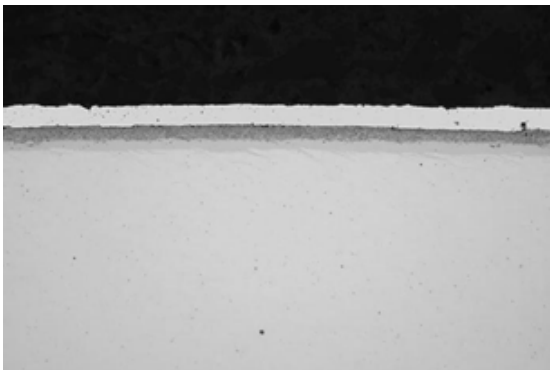

Figure 12: Nitrided layer, plated with aluminum foil. The porous layer inside the compound layer is well visible. Magnification 200x.

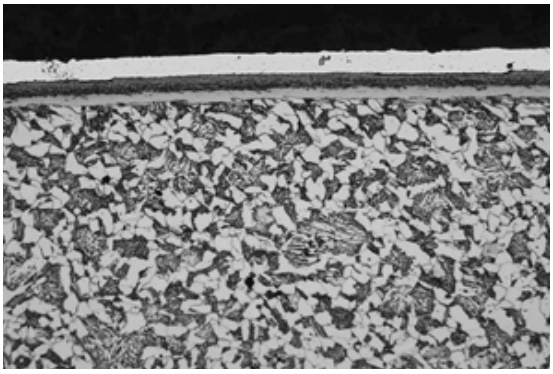

Figure 13: Nitrided layer after etching with 3% Nital. Magnification 200x.

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.

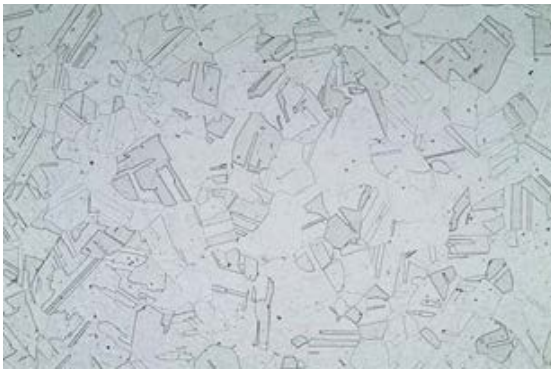

Figure 16: Stainless steel X5CrNi18-10. Etched with V2A at 60°C. Magnification 200x.

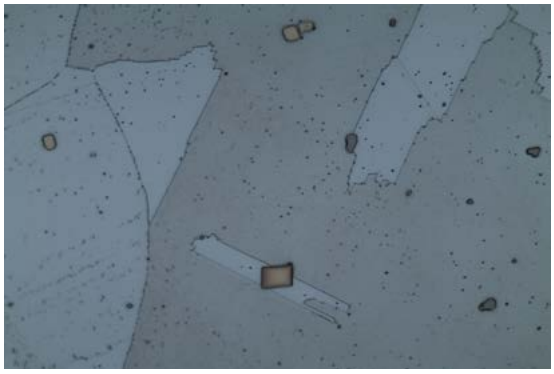

Figure 17: Stainless steel X5CrNi18-10. Ti (C,N) particles (orange). Magnification 1000x.

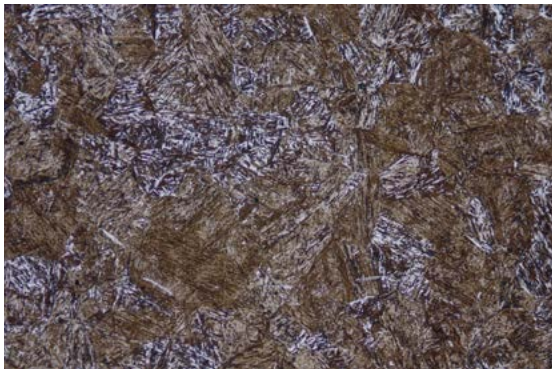

Figure 18: Martensite revealed with 3% Nital. Magnification 200x.

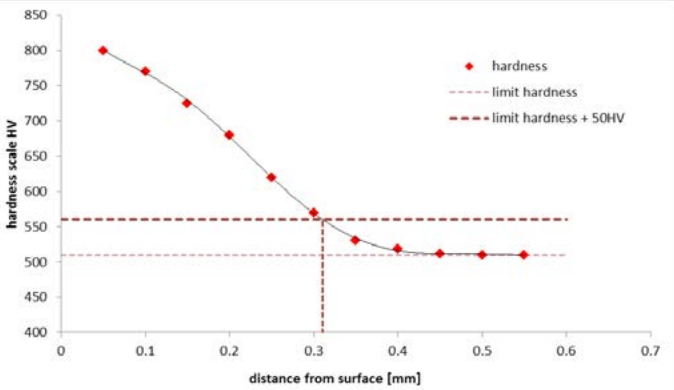

Figure 19: Principle of the NHD evaluation. Hardness is measured from surface to core, leading to a decrease in hardness for surface hardened materials.

Hardness Testing

Hardness testing is commonly used in the field of process control of heat treatment and to evaluate surface hardness and hardness depths of surface engineered materials such as nitrided. Figure 17 shows a typical graph of hardness against depth from the specimen surface.

There are a number of international and national standards to which these tests should be conducted, not all of which can be listed here. As an example we have used the measurement of NHD for nitrided surfaces according to the German standard DIN 50190-3 (NHD is referred to as NHT, German for "Nitrierhärtetiefe").

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 a few microns in its thickness and consists mainly of iron nitrides (α-N-ferrite, γ'-Fe_4N, ε-$Fe_2N_{(1-x)}$), the precipitation zone exhibits different precipitated nitrides after cooling or special nitrides formed during the nitriding process [3]. This second zone 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 case evaluations can now be highly automated, which can represent enormous savings in terms of time and efficiency. These systems can incorporate automated movement, indentation, illumination, measurement and reporting. A fully-automated Vickers hardness tester, as shown in Figure 20, was used for this work.

Figure 20: Wilson VH3300 automated Vickers hardness tester.

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 placed with respect to the indent distances according to the ISO 6507-1 , as indents placed too close together will give erroneous results. Increasing indent sizes are correlated to the decrease of hardness.

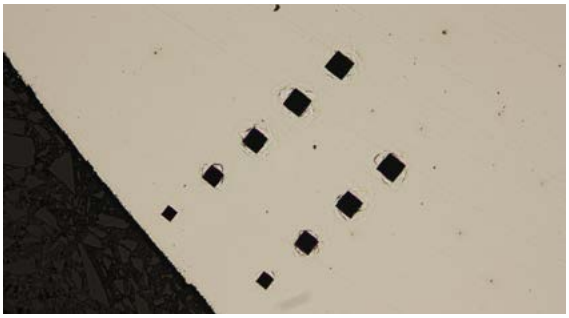

Figure 21: Vickers indents of a NHT measurement. The hardness decrease can be correlated with increasing indent size. Magnification 200x.

Conclusion

Efficient sample preparation in production environments needs to be reliable, repeatable and fast to perform. Especially with high sample volumes of similar materials, the preparation sequence can be accelerated by selecting the optimal equipment and consumables. Using the PlanarMet™ 300 stone grinder for the initial grinding step, as well as aligning the selection of subsequent steps to narrower material groups, can dramatically reduce preparation and analysis times while maintaining a high quality surface finish. Using the PlanarMet™ 300 stone grinder as initial grinding step reduces cycle times while maintaining excellent surface finishes.

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