TECHNotes

Published by Buehler, a division of Illinois Tool Works

Volume 8, Issue 1

Metallographic Preparation of Fasteners; Microscopic de/carburisation assessment and analysis of steel fasteners

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Introduction

The addition of carbon to iron results in steels of varying carbon content, the concentration of which has a bearing on the strength, and its relative hardness compared to an unalloyed steel variant. Carbon can also be added to steels through a heat treatment process, in which carbon is diffused into the surface to increase the carbon content producing a hard and wear resistant outmost layer. The process, referred to as carburising, results in the formation of martensitic microstructure after quenching on the surface regions of a steel component, imparting high hardness with typical Vickers hardness values of around 600-700HV. The resultant case or hardened region offer good wear and fatigue resistance with the core offering the component good toughness.

After carburising, the fasteners are subsequently quenched and tempered in a furnace with controlled atmosphere to ensure that the carbon content remains at the desired level for the steel being heat treated. Furthermore, measures are always taken to prevent presence of oxygen within the furnace to prevent formation of oxides that would be deleterious to fastener components. Therefore, furnace conditions must be monitored and carefully controlled to ensure decarburisation, the loss of carbon content on the surface layers, does not occur as this will be detrimental to the performance of a steel threaded fastener.

Materials and Methods

Standards do specify the steels desirable for fasteners and related components. The steels can be low carbon, medium carbon, various alloy steels containing molybdenum, chromium, nickel and manganese to a controlled weight percentage. Standards are also available for guidance on how to assess fasteners manufactured from these materials with respect to decarburisation phenomena. Carburisation and decarburisation assessment can be investigated microscopically or through hardness testing of components as prescribed in EN ISO 898, ASTM F2328M or ASTM E1077 and ISO 3887. It's worth noting that too much of either process is not desirable to the performance of a threaded fastener component. The standards specify how to assess de/carburisation as schematically illustrated in Figure 1

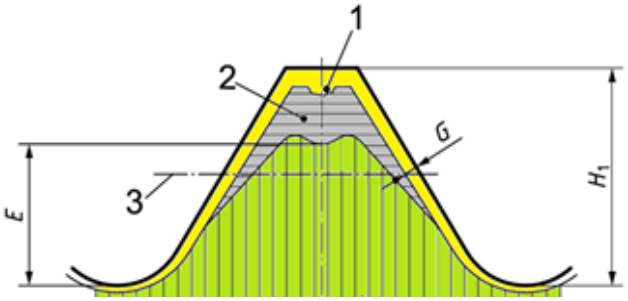

***Figure 1**. schematically illustrates decarburisation zones on a fastener with 1. showing complete decarburization, 2 - partial decarburization or ferritic decarburization, and 3 - the pitch line. E - Height of the non-decarburised thread zone, G - depth of complete decarburization in the thread and H_1 - External thread height in the maximum metal condition*

Before de/carburisation assessment is carried out, metallographic preparation of the threaded component must be carried out correctly and as close as possible to the longitudinal threaded axis. This involves sectioning, mounting in an appropriate resin to preserve the edges or outermost surface regions, then through a series of grinding and polishing stages before final optical and/or hardness testing analysis is carried out. For optical microscopical analysis, the polished components are generally etched using a 2-3% Nital solution (nitric acid in ethanol) to reveal the different zones on the threaded regions.

This application article will highlight metallographic preparation approach that would ensure correct and routine de/carburisation assessment of steel fasteners using microscopical technique. Microscopical techniques are ideal for measuring the depth of decarburisation of either a hot rolled, forged, annealed or normalised steel samples or where decarburisation phenomena is observed.

Sample preparation techniques

Sectioning

Fasteners specification do state that the specimens should be longitudinally sectioned to reveal the profile for the head, thread and shank regions and as close to the thread axis as possible. The most effective way to achieve this is to section the fastener slightly off-centre, Figure 2, to allow for material loss attributable to grinding and polishing stages.

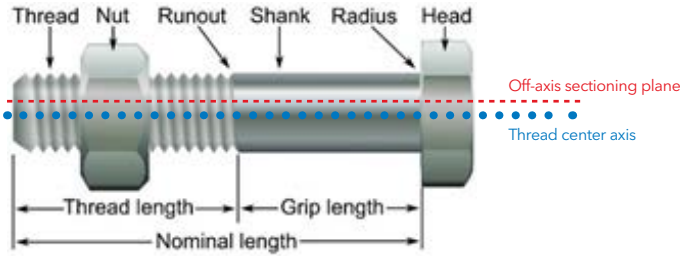

***Figure 2**. Schematic illustration of threaded component with thread axis, and off-centre sectioning plane.*

An additional sectioning consideration is to always use thin abrasive wheels for abrasive cutters as these offer smaller kerf loss during sectioning as well as allowing one to section as close to the threaded component center axis. For smaller sized fasteners, using a precision sectioning machine is an alternative that also offers superior surface finish ideal for fine grinding and polishing. The selection of either an abrasive or precision sectioning machine depends on two main factors; the size of the finished components and the speed of processing the samples, which is underpinned by whether the laboratory is processing a relatively small number of samples, or a much higher volume.

***Figure 3**. Illustrate typical fasteners sizes*

Clamping consideration

Incorrect clamping will have a major bearing on proper lengthwise sectioning near the thread center axis as shown in Figure 2. To ensure a precise, flat longitudinal sectioning, ensure dual clamping, Figure 4, is carried out or using clamping devices suitable for fasteners as illustrated by a fastener chuck in Figure 5 below.

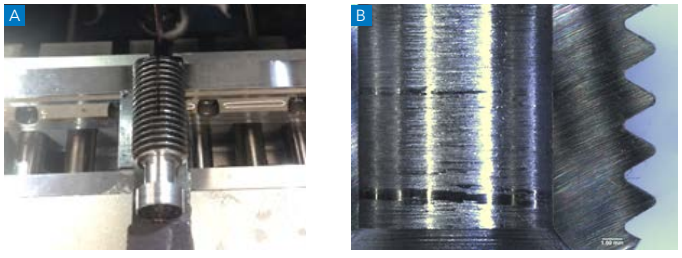

***Figure 4**. (a) shows dual clamping of a fastener, and (b) showing the thread region after sectioning.*

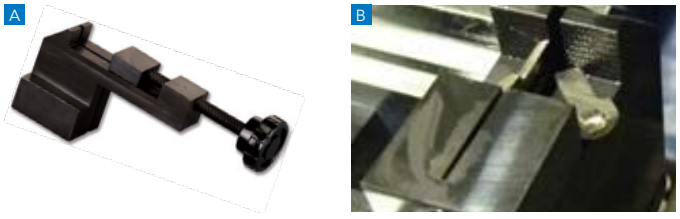

***Figure 5**. (a) showing a fastener chuck, and (b) showing fastener sectioning using the chuck on an abrasive cutter*

Once sectioned, the test pieces are subsequently mounted using a suitable resin/media to preserve all features present on the fastener.

Mounting

Selection of mounting media that has minimal or no shrinkage at the resin component interface whilst ensuring good edge retention of the components is key to fastener preparation. Additional consideration is whether one uses cold or hot mounting techniques. Cold mounting has the advantage of speed when using fast curing resins, however, these have health and safety considerations during use. Examples of resins include VariDur 3003 and VariDur 200. For compression or hot mounting process on the other hand, this has a limit on how many samples one can process in one cycle, however, it has the advantage of simplicity and also results in resin mounts with better shore hardness offering better abrasion and polishing rates, examples of resin used are EcoMet F/G for edge and backed-up to make full mount with Phenocure resins.

***Figure 6**. (a) SimpliMet 4000, and (b) showing dual mounting on same cycle.*

Grinding & Polishing

After mounting, samples can be prepared using two methodologies. For fast preparation ideal for high throughput in the laboratory, a procedure involving planar stone grinding followed by polishing steps can be adopted or following a more traditional method of grinding and polishing. The methods highlighted in this section will illustrate both methodologies and how the procedures have been carried out for steel fasteners.

- **Planar grinder preparation**

This is normally carried out using a 3-step procedure for routine analysis, and for an in-depth analysis, a final polishing step is added using alumina suspension. For a 3-step procedure, the samples are finished off on a 3um diamond suspension on a napped surface, such as Microfloc. For a 4-step procedure, a soft woven surface such as Trident replaces the, Microfloc, followed by a final polish on a napped surface, such as MicroCloth. Figure 7 illustrates preparation of steel alloy type of fastener with low magnification showing the thread region (a), and (b) a high magnification image with the outer most edges of the thread showing some oxidation effects.

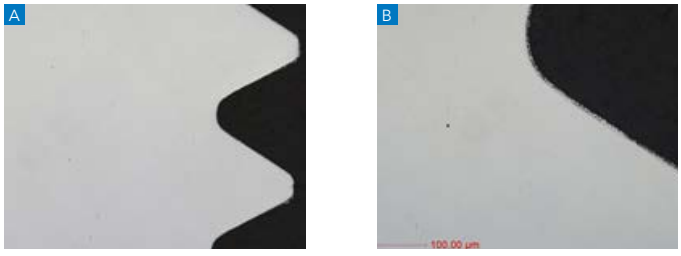

***Figure 7**. (a) and (b) showing final polished surface via planar grinding route on a steel alloy fastener*

- **Traditional preparation**

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Table 1 Planar grinding route - using PlanarMet 300

	Surface	Abrasive	Lubricant/Extender	Force (N) (Per Specimen)	Time (min:sec)	Platen Speed	Head Speed (rpm)	Relative Rotation	Burst
1	Alumina Stone	120	Water	30N	Until Plane	Fixed	120	>>	-/-
2	UltraPad/ Hercules	9 µm Metadi Supreme	MetaDi	30N	2:00	120	60	><	1-2
3	Microfloc/ Trident	3µm Metadi Supreme	Metadi	30N	2:00	150	60	><	3-4
4	MicroCloth	MasterPrep	Water**	30N	2:00	120	60	>>	4-5

>> Comp, >< Contra ** last 20 seconds run water only

Table 2 Conventional method using SiC, DGDs for the planar grinding step

	Surface	Abrasive	Lubricant/Extender	Force (N) (Per Specimen)	Time (min:sec)	Platen Speed	Head Speed (rpm)	Relative Rotation	Burst
1	240-320 grit	58-35 µm	Water	30N	1:00	Fixed	120	><	-/-
2	UltraPad/ Hercules	9 µm Metadi Supreme	MetaDi	30N	5:00	120	60	><	1-2
3	Trident	3µm Metadi Supreme	Metadi	30N	3:00	150	60	>>	3-4
4	ChemoMet	Alumina	Water**	30N	2:00	120	60	><	4-5

>> Comp, >< Contra ** last 20 seconds run water only **Notes:** Step 1, either SiC paper or diamond grinding discs are applicable. Coarse grits can be used for initial stock removal and if finish after section is not good. Incorporate intermediate grinding stage to remove damage from coarser grits.

This involved use of traditional abrasives such as SiC papers and diamond grinding discs for the initial planar grinding step with grit size ranging from 280 to 400 grits. The selection of starting grit value depends on the surface finish after sectioning. After planar grinding, the samples are taken through an integrity stage using a 9um diamond suspension on a rigid composite disc (Hercules) or no napped surface (UltraPad) then followed by a 3um diamond suspension finish on a medium hard woven surface with a final finish on a napped cloth such as Microcloth. For 3 step procedures, the samples can be finished off with Microfloc, a napped surface using 3um diamond suspension.

For both methods, planar grinder and traditional preparation routine, the samples can then be etched either after the 3 or 4 step procedure to reveal microstructural details of the core regions of the fastener, the de/carburised regions, as well as revealing defects due to processing. Similarly, for failure or forensic investigation of components, the same procedures are applicable with the recommendation to do at least a 4-step procedure. It is worthwhile doing an additional 1um diamond polish using a medium hard surface such as Trident or VerduTex to aid with keeping the surface planar with no rounding, smear and deformation artefacts. The same surface should be used for 3um diamond polishing stage.

Optical evaluation of de/carburization

After the final polishing stage, the sample can be analysed in the as-polished condition for defects, degree of internal oxidation, and if conducting micro indentation for decarburisation assessment. For optical evaluation of the de/carburised layers, the samples are etched using 2-3% Nital, for a few seconds and then observed under a microscope. Analysis in this article was carried out using a Nikon LV150 microscope equipped with a 3-megapixel camera and measurements carried out using OmniMet imaging analysis software. Figure 8 illustrates a steel fastener in the original, as-polished and etched conditions.

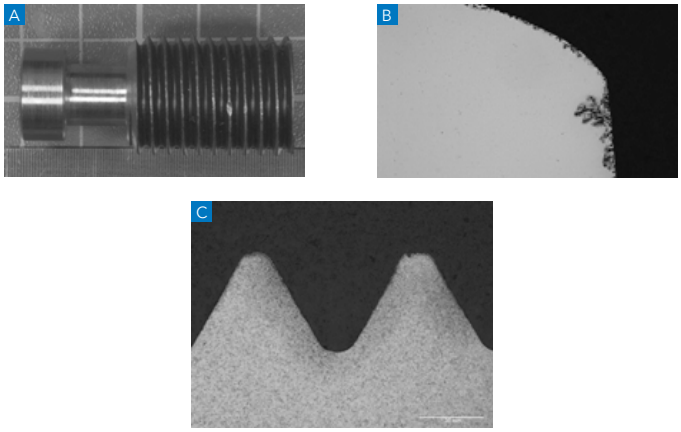


***Figure 8**. (a) illustrates a steel fastener, (b) showing polished tooth region and (c) showing in as etched condition.*

- **Evaluation of core microstructure**

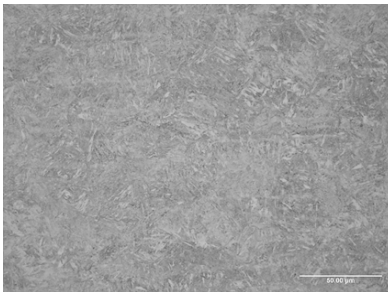


Figure 9. illustrates a tempered microstructure of a steel fastener

Notes:
- Steel fasteners after quenching consist of martensite that is highly unstable in addition to being excessively hard. The fasteners must go through a tempering process to achieve desired hardness and strength.
- Tempering should be carried out immediately after quenching before the fastener cools down
- Microstructure is characteristic of low carbon steel with a tempered martensitic microstructure, Figure 9

- **Evaluation of decarburized area**

Figure 10(a) showing as polished condition with oxide defects observed (internal oxidation) on the crest region of the threads. Figure (b) and (c) showing etched sample illustrating the internal oxidation matrix within the decarburised region and remnant ferritic microstructure adjacent to the bulk tempered microstructure.

The depth of decarburisation is generally easy to discern due to excellent contrast between the ferrite layer and the bulk microstructure. However, this also depends on the steel type, for example, it could be a challenge for heat treated specimens as assessment is based on assessing non-martensitic structures in the partially decarburised regions.

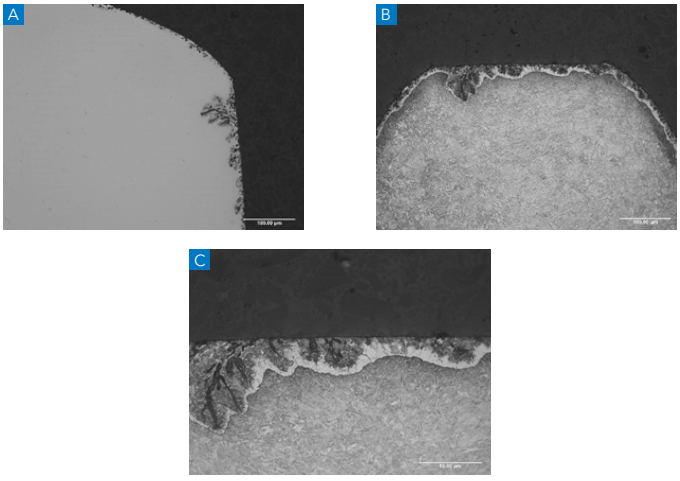
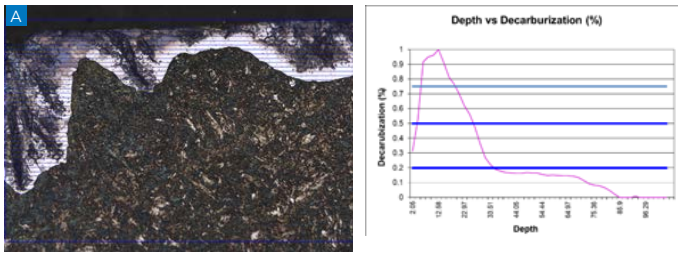


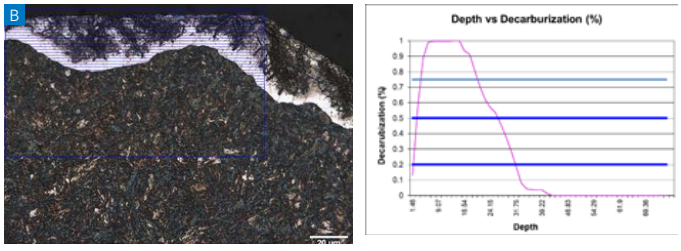
Figure 10. (a) as polished, (b) etched with 2% nital showing the decarburised regions around the flat crest and (c) showing a high magnification region around the crest with internal oxides and a ferritic microstructure adjacent to the bulk microstructure.

For decarburisation assessment, OmniMet image analysis decarburisation script was employed to quantify the degree of decarburisation as per ASTM E1107. Before carrying out decarburisation measurement, the sample is given a slightly longer etch, at least 20 seconds to cause a deeper etch, as illustrated in Figure 11. The decarburised layer after etching will show or exhibit a light etching appearance as shown, however, this might not appear light after etching, and depends on the steel alloy type being investigated. The sample is then placed under the microscope to capture the microstructure and automatically calculate the percentage decarburisation from the surface. The software will also plot a depth profile from the surface of the thread to the bulk microstructure.

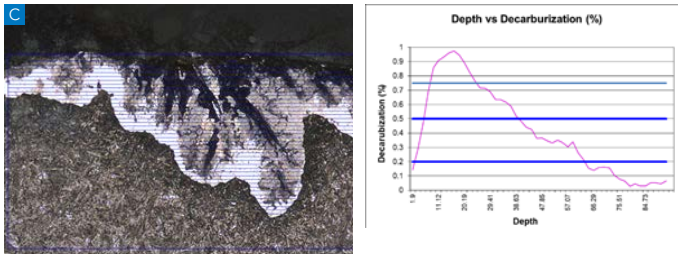
The key consideration for decarburisation assessment is the evaluation of the maximum depth of complete decarburisation G, Figure 1, and the height of the non-decarburised zone, E. with automated image analysis the different regions can be evaluated with ease on the pitch of the thread as specified in ASTM/ISO standards..



Percent Decarburization	Depth (µm)
20	34.89
50	26.58
75	19.40



Percent Decarburization	Depth (µm)
20	31.54
50	26.36
75	20.41



Percent Decarburization	Depth (µm)
20	63.04
50	39.56
75	24.61

Figure 11. (a) to (c) showing decarburisation assessment of different regions along the thread region.

Appendix

Terminologies – ISO/ASTM

Carburisation – process of increasing the carbon content of the surface layers of a steel fastener product

Decarburisation – is the loss of carbon form the surface layer of the fastener to a level below the solubility limit of carbon in ferrite so that only ferrite is present.

Complete/Gross decarburisation – also known as complete decarburisation where there is sufficient carbon loss from the steel leaving a clearly defined ferritic grains after microscopic analysis.

Partial decarburisation - decarburization with sufficient loss of carbon to cause a lighter shade of tempered martensite and a significantly lower hardness than that of the adjacent base metal, without, however, showing ferrite grains under metallographic examination

Ferritic decarburisation - decarburization with considerable loss of carbon to cause a lighter shade of tempered martensite, with the presence of ferrite grains or grain boundary network under metallographic examination

Depth of decarburisation – The perpendicular distance from the specimen surface to that location in the interior where the bulk carbon content is reached; a sum of the depths of complete and partial decarburisation

Average depth of decarburisation – the mean value of five or more measurement of the total depth of decarburisation.

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ASTM standards; E1077, A370, E3, E384, E407 F2328, F2328M, F606/M

ISO standards; 898-1, 898-1, 4042, 6507, 6506, 6508

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