Metallographic preparation of High Alloy Tool Steels

Struers

Application Notes

Steels can broadly be classified into three categories based on chemical composition:
- Carbon steels
- Low alloy steels with small amounts of alloying elements
- High alloy steels with >6% alloying elements

In addition to carbon, high alloy steels contain large amounts of alloying elements such as chromium, nickel, vanadium, tungsten and molybdenum. Wear resistance, toughness, strength and hardness are the most important characteristics of tool steel, and the alloying elements mentioned improve and optimize these mechanical properties, and, if added in sufficient amounts, provide specific properties such as corrosion and heat resistance, retention of hardness at high temperatures, and retention of strength at low temperatures etc.

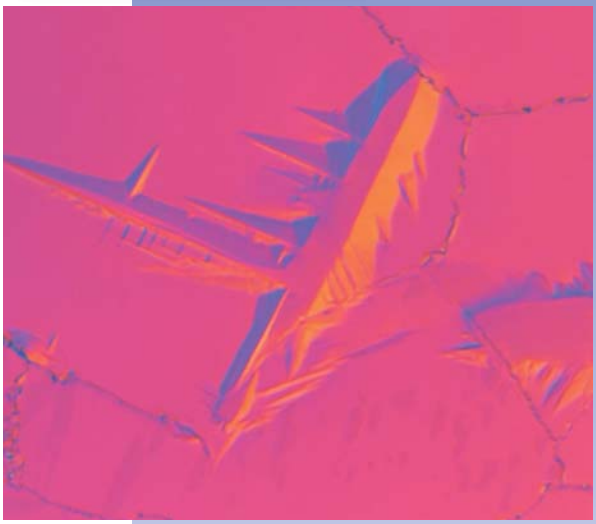
The increasing demands of advanced production technologies and economic pressures in all industries require steel manufacturers to constantly improve the quality of high performance tool steel alloys for special and demanding applications. For example, steels for making punches, dies or cutting tools require very specific properties such as high strength and hardness combined with toughness. In addition, these steels require a high standard of cleanliness. Such properties can only be achieved by carefully controlling all stages of steelmaking, and subsequent forging/ rolling and heat treatment processes.

The main demands on the metallography departments of steel producers of high quality alloyed tool steels are the following:
- To handle high sample volumes efficiently,
- To use, if possible, one standard procedure for all steel qualities,
- And to deliver well polished surfaces with undamaged carbides and inclusions.

This is particularly important for evaluating structures with carbides and inclusions of ultra clean steel.

The metallographic sample evaluation includes distribution and size of carbides, detection of decarburization of hardened and then tempered steels, detection of micro-segregations and inclusion ratings.

Cold work tool steel. Tool for punching



Plastic mould steel etched with 5% Picral, revealing some singular needles and plates at high magnification in an otherwise amorphous martensite — *1000x, DIC*

Difficulties during metallographic preparation

Cutting:
Efficient cutting without overheating.

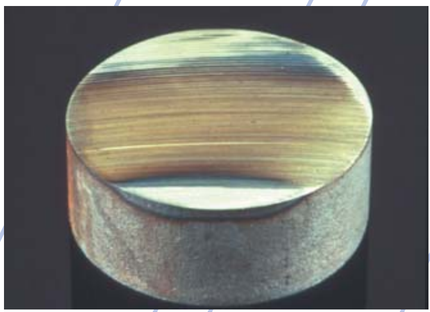


Fig.1: Thermal damage due to faulty cutting conditions

Grinding and polishing: Handling large sample volumes. Very fine carbides and inclusions can be pulled out of the soft matrix; large carbides can be cracked during plane grinding.

Fig. 2: Fractured primary carbides — *200x*

Solution:

- Selecting the correct cut-off wheel
- Using automatic grinding and polishing equipment
- Sufficient diamond polishing to polish past the mechanical damage of grinding

Production and application

The production process of high alloy steels is a sophisticated process of melting and remelting. A mixture of iron and well sorted scrap is first melted in an electric arc furnace, and cast into an ingot form, or continuously cast into bloom or billet. For many applications these primary products can be further processed into bar, rod or plate form. For steels with higher quality demands, the primary product can be used as feedstock for a secondary steelmaking process. This secondary process can be a double or even triple remelt by vacuum induction melting plus vacuum arc remelting, or electroslag remelting, which can also be done under pressure and protective gases.

The main purpose of this secondary process is to reduce impurities such as oxides, sulphides and silicates so that with successive remelts the degree of cleanliness increases and homogenous ingots with excellent mechanical and physical properties are produced.
The high cost of these energy intensive remelting techniques is reflected in the price of high temperature and corrosion resistant martensitic and hot work tool steels for special applications.

The range of high alloy steels is very wide and some products are even tailor-made for especially demanding applications. Following are some examples of high alloy tool steels and their applications, with the approximate content of main alloying elements:

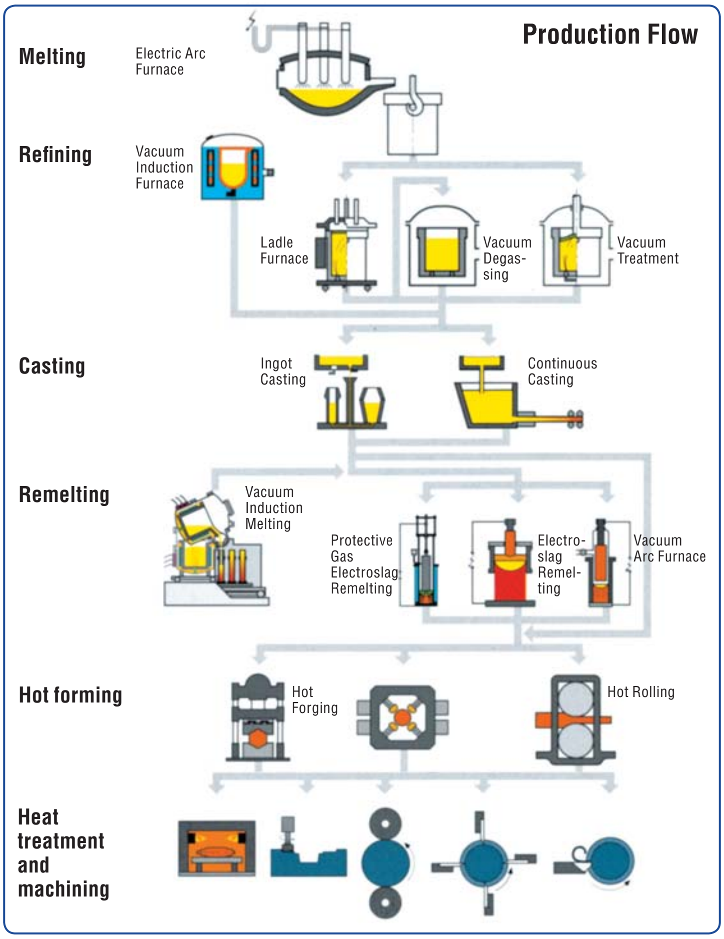


Fig. 3: Production flow for high alloy tool steels

Cold work tool steel: 1.6-2% carbon, 5-12% chromium, for punching, stamping, deep drawing, thread rolling tools, shear blades.
Properties: high toughness, high compressive strength and wear resistance, good nitrideability.

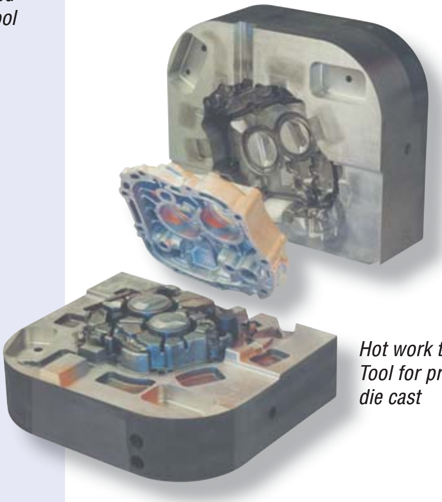
Hot work tool steel: 0.38% carbon, 5% chromium, 1.5-3% molybdenum and 0.5% vanadium, for pressure die casting tools.
Properties: high hot strength, toughness and wear resistance, high thermal fatigue and shock resistance.

High speed tool steels: 0.75-1.3% carbon, 4.5% chromium, 2% vanadium, 6-18% tungsten, 4-9% molybdenum, for taps, turning and milling tools.
Properties: retention of hardness and toughness at elevated temperature.

Plastic mould steel: 0.3% carbon, 12-17% chromium, for molding plastic parts for the automotive, medical and consumer goods industry.
Properties: Can be polished to high surface finish, exceptional toughness and hardness, good corrosion resistance.

For tool making the steel is used in the as tempered condition. After the tool is machined it is surface hardened by nitriding or induction hardening. The conditions under which tools have to operate are varied and sometimes extreme. Therefore, the variations in alloys for tool steels are wery wide in order to accommodate the best possible selection for specific difficult and demanding tool applications.

High speed cutting tool



Hot work tool steel. Tool for pressure die cast

Tool for moulding plastic

Difficulties in the preparation of high alloy tool steels

As heat treatability of tool steels is a quality criterion, thermal influence during cutting has to be avoided in order to give a true representation of the actual structure. When cutting larger sections and failure analysis samples this preparation step has to be carried out with great care.

The main difficulty of grinding and polishing high alloyed tool steels is the retention of carbides and non metallic inclusions. In cold working tool steels primary carbides are very large and fracture easily during grinding. In the fully annealed conditions, secondary carbides are very fine and can easily be pulled out from the softer matrix. (See Fig. 2 front page, micrograph with cracked carbides).
Processing large sample volumes of different high alloy tool steels during various stages of the production can be a challenge, which requires a very efficient organisation of the workflow, automatic equipment and standard procedures.

Recommendations for the preparation of high alloyed steels

Cutting
The majority of samples are usually sectioned by rough mechanical means from slabs and blooming mill material into standard sizes. Critical cuts for heat treatment samples or failure analysis are always carried out with a metallographic cut-off machine. High alloy tool steels are extremely sensitive to thermal damage. Therefore special care must be taken to select the appropriate cut-off wheels and secure sufficient cooling for cutting.

Soft aluminium oxide or resin bonded cubic boron nitride cut-off wheels are recommended.

Mounting
Depending on the size and volume of the samples and the information that is needed from them, the specimens can be unmounted, hot or cold mounted. Surface treated samples that need good edge retention should be hot compression mounted using fibre-reinforced resins (IsoFast, DuroFast). Samples that do not require edge retention can be left unmounted if their dimensions are suited for sample holders. For standardizing sample sizes, which can be an advantage when handling large volumes, cold mounting in rectangular silicon or polypropylene mold cups (UnoForm) is recommended. It is important that the cold mounting resin has little shrinkage to avoid contamination due to gaps between sample and resin.

Grinding and polishing
The main requirements on the preparation of high alloy tool steels are a true representation of form, amount and size of carbides, and the retention of nonmetallic inclusions in an undeformed matrix. Large volumes of samples of

high alloy steels are best processed on fully automatic grinding and polishing machines, which guarantee a fast and efficient workflow and reproducible results. As tool steels are hard, fine grinding with diamond is more efficient and economical than grinding with silicon carbide paper. Sometimes a final oxide polish after the diamond polishing step can be useful for contrasting and identifying carbides.

Following are suggestions for preparation methods with fully automatic grinding and polishing equipment, and semi-automatic equipment respectively.

These methods are based on experience and offer excellent reproducible results. Small changes may have to be made to accommodate specific requirements or personal preferences.

Grinding

Step		PG	FG
	Surface	Stone 150#	MD-Allegro
	Suspension		9 µm
	Lubricant	Water	Blue
	rpm	1450	150
	Force [N]	300	300
	Time	As needed	9 min.

Polishing

Step		DP 1	DP 2
	Surface	MD-Dac	MD-Nap
	Suspension	6 µm	1 µm
	Lubricant	Blue	Blue
	rpm	150	150
	Force [N]	300	150
	Time	6 min.	4 min.

Table 1: Preparation method for high alloy tool steel on large automatic equipment

Grinding

Step		PG	FG
	Surface	MD-Piano 220	MD-Allegro
	Suspension		DiaPro Allegro/Largo
	Lubricant	Water	
	rpm	300	150
	Force [N]	210	210
	Time	As needed	9 min.

Polishing

Step		DP 1	DP 2	OP 1*
	Surface	MD-Dac	MD-Nap	MD-Chem
	Suspension	DiaPro Dac	DiaPro Nap B	OP-AA
	rpm	150	150	150
	Force [N]	210	150	90
	Time	6 min.	1 min.	1 min.

Remark: DiaPro diamond suspensions can be substituted with DP-Diamond suspension P as follows: For FG with 9µm, DP 1 with 3 µm, DP 2 with 1µm.

**Optional*

Table 2: Preparation method for high alloy tool steel on table models semi-automatic equipment

The preparation data in Table 1 are for 6 samples, 65x30 mm, unmounted or cold mounted, using Struers MAPS or AbraPlan/AbraPol.

For smaller sizes and numbers of samples semi-automatic grinding and polishing equipment will give also good, reproducible results.

The data in Table 2 are for 6 samples, 30 mm mounted, clamped into a sample holder, using Struers TegraPol-31/TegraForce-3 with TegraDoser-5.

Etching and structure interpretation

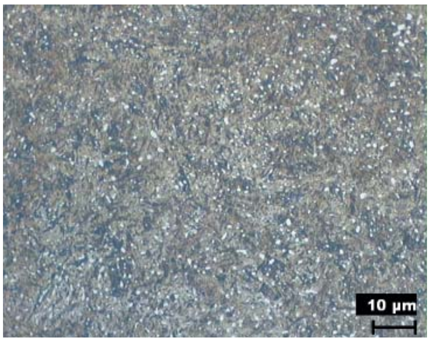
Etching

Usually samples of tool steel are first examined unetched to identify inclusions and carbide size and formation.
For revealing the structure, either Nital or picric acid in various concentrations is used. For instance, to show the carbide distribution in cold work steel a 10% Nital colors the matrix dark while the white primary carbides stand out.
For fine globular pearlite a brief submersion into picric acid followed by 2% Nital gives a good contrast and avoids staining.

For mixing and working with etching solutions standard safety precautions have to be observed.

Fig. 4: Cold work tool steel etched with 10% Nital, primary carbides stand out white *100x*

Fig. 5: Hot work tool steel etched with Picral and Nital, globular pearlite *500x*



High speed tool steel after final heat treatment, very fine structured martensite with chromium carbides

Nital:
100 ml ethanol
2-10 ml nitric acid *(Caution do not exceed 10% solution, explosive!)*

Picric acid etching solution:
100 ml ethanol
1-5 ml hydrochloric acid
1-4 g picric acid

Structure interpretation
Generally high alloyed steels have the same structural phases as regular iron-carbon alloys: ferrite, pearlite, martensite and austenite, but the solid solution can absorb a certain amount of alloying elements. Carbon forms complex carbides with some alloying elements such as chromium, tungsten and vanadium. In addition, the solubility of carbon in iron changes: Adding alloying elements such as silicon, chromium, tungsten, molybdenum and vanadium increases the alpha area of the iron-iron carbon diagram, while adding nickel and manganese will enlarge the gamma area. These characteristics influence the time-temperature transformation which is specifically important for the heat treatment of tool steels.

The primary structure of cold work tool steel is a ledeburite. Its coarse structure is transformed through hot rolling or forging into a ferritic-pearlitic matrix with large primary carbides (Fig. 6). A subsequent full annealing shapes the secondary fine carbides (Fig. 7).

Hot work tool steels in the fully heat treated condition show ideally a tempered martensitic matrix containing very fine globular pearlite (Fig. 5). Here it is important that segregations from the primary structure are as much as possible evened out through heat treatment as uneven chemical composition can lead to corrosion problems (Fig. 8).

Plastic mould steel is a corrosion resistant tool steel which before heat treating shows an “amorphous” martensite with strings of carbides (Fig. 9). After annealing it shows finely dispersed carbides (Fig.10).
The even distribution of carbides in tool steel can be improved by powder metallurgical process. Through a powder making process and subsequent hot isostatic pressing, a homogenous, segregation free steel is made, which is especially suitable for unconventional tool geometries that would be expensive to make by mechanical means (Figs.11 and 12).

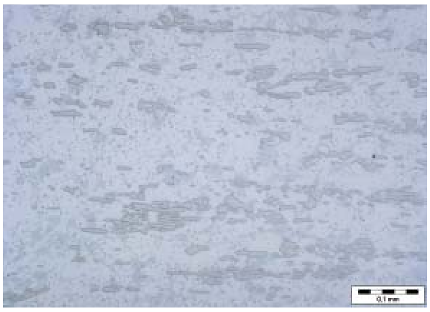


Fig. 6: Cold work tool steel after initial hot forming, slightly contrasted by a short final oxide polish, showing large primary carbides in a ferritic-pearlitic matrix 200x

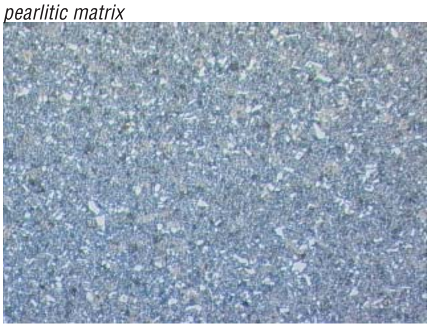


Fig. 7: Fully heat treated cold work tool steel showing very finely dispersed secondary carbides and small white primary carbides 200x

Fig. 8: Hot work tool steel showing segregations 100x

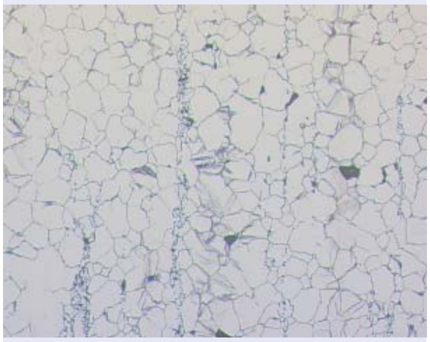


Fig. 9: Plastic mould steel, etched in 5% Picral, amorphous martensite with strings of primary carbides 100x

Fig.10: Plastic mould steel after annealing shows very fine carbides 500x

Fig.11: Carbide distribution in conventionally produced steel

Fig.12: Carbide distribution in powder metallurgically produced steel

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Summary

An ever larger portion of high alloy tool steels are today made to fit customers' applications. This requires the production of very clean material with very specific mechanical, physical and metallurgical properties. Metallographic inspection from the initial casting and first forming stages, to the final semi-finished, heat treated product, is an essential tool for controlling manufacturing and heat treatment processes.

The main challenges regarding metallographic preparation are managing the large sample volume, and producing consistently excellent surface finishes. As size, form and distribution of carbides and inclusion are the main quality indicators of tool steel, it is essential that they are retained during preparation. Automatic grinding and polishing, using diamond for fine grinding and polishing, gives good and reproducible results. Using one preparation method applicable for all the various types of tools steels makes the handling easy and efficient.

Application Notes
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