Metallographic preparation of stainless steel

Struers

Application Notes

Corrosion resistant steels contain at least 11% chromium and are collectively known as “stainless steels“. Within this group of high alloy steels four categories can be identified: ferritic, martensitic, austenitic, and austenitic-ferritic (duplex) stainless steels. These categories describe the alloy’s microstructure at room temperature, which is largely influenced by the alloy composition.

The main characteristic of stainless steels is their corrosion resistance. This property can be enhanced by the addition of specific alloying elements, which have a further beneficial effect on other characteristics such as toughness and oxidation resistance.

For instance, niobium and titanium increase resistance against intergranular corrosion as they absorb the carbon to form carbides; nitrogen increases strength and sulphur increases machinability, because it forms small manganese sulphides which result in short machining chips. Due to their corrosion resistance and superior surface finishes stainless steels play a major part in the aircraft, chemical, medical and food industries, in professional kitchens, architecture and even jewelry.

High performance stainless steel parts for the aircraft industry

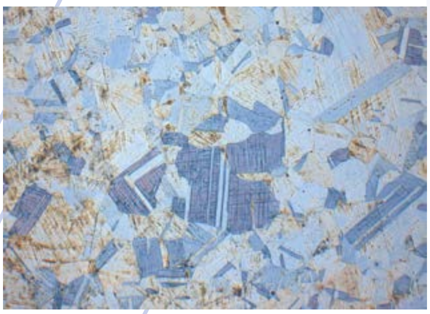
Metallography of stainless steels is an important part of the overall quality control of the production process. The main metallographic tests are grain size measurement, identification of delta ferrite and sigma phase and the evaluation and distribution of carbides. In addition, metallography is used in failure analysis investigating corrosion/oxidation mechanisms.

Fig.1: Duplex steel etched electrolytically with 40% aqueous sodium hydroxide solution, showing blue austenite and yellow ferrite 150x

Difficulties during metallographic preparation

Grinding and polishing: Deformation and scratching in ferritic and austenitic stainless steels. Retention of carbides and inclusions.

Surface of stainless steel after 3µm polish, showing deformation from grinding DIC 25x



Insufficiently polished stainless steel after colour etching (Beraha II), showing deformation 100x

Solution:

Thorough diamond polishing and final polishing with colloidal silica or alumina.

Production and application of stainless steel

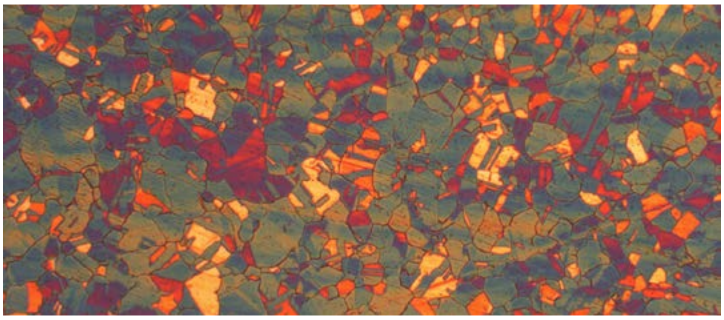
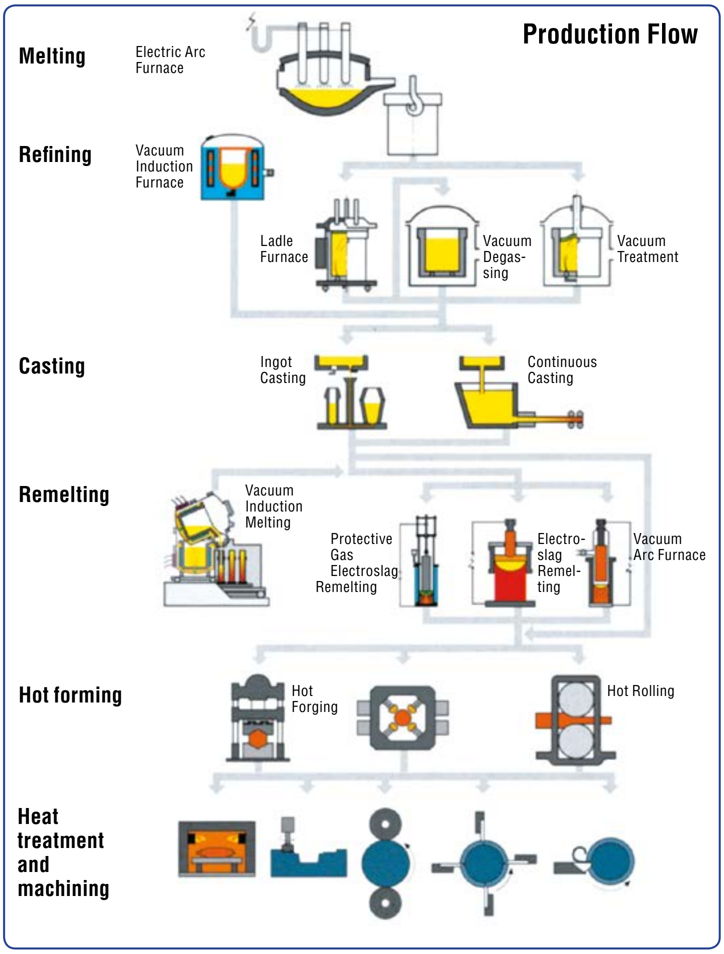


Fig.2:
Austenitic steel, colour etched (Beraha II).

100x

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 then cast into 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 pro-duct 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.



Application
The corrosion resistance of stainless steels is based on alloying chromium with iron, and is dependant on the formation of a passive surface oxide layer, which rebuilds itself spontaneously when mechanically damaged.

A variety of different types of corrosion can occur, such as pitting, stress, intercrystalline or vibrational corrosion. Improved resistance against any specific form of attack can be provided by adding alloying elements other than chromium, for instance molybdenum, which improves resistance against pitting corrosion. The main alloys, properties and examples of applications of the four types of stainless steels are briefly described:

Ferritic stainless steels: are non heat treatable alloys with a low carbon content and 11-17% chromium.

Properties: magnetic, resistant to atmospheric corrosion, moderate strength and toughness.

Applications: magnetic valves, razor blades, car trim.

Martensitic stainless steels are heat treatable alloys with medium carbon content, 12-18% chromium and 2-4% nickel. Properties: high corrosion resistance, and high temperature and creep resistance. Applications: scalpels, knives, hooks and tweezers in medical applications, drive systems and high performance parts for airplanes.

Austenitic stainless steels are not heat treatable, have 0.03-0.05 % carbon, main alloying elements are chromium (17-24 %), nickel (8-25%) and molybdenum (2-4%); titanium and niobium are added for carbide forming. Properties: high ductility, high corrosion resistance, resistant to oxidizing acids, alkalis, very good cold forming properties, easy to work and machine.

Applications: screws, bolts and implants, low temperature applications, vessels and pipes in the chemical, pharmaceutical and food industries, kitchen utensils.

Austenitic-ferritic steels, (Duplex) have a low carbon content and generally higher chromium (21-24%) and lower nickel content (4-6%) than austenitic steels, and 2-3% molybdenum.

Properties: fatigue resistance in corrosive media, good resistance against stress corrosion.

Applications: equipment for chemical, environmental and offshore industries, architecture.

Difficulties in the preparation of stainless steels

Ferritic stainless steels are soft and austenitic steels are ductile. Both are prone to mechanical deformation. Final polishing usually leaves these steels highly reflective, however, if they are not thoroughly prepolished, deformation can reappear after etching (Fig. 3).

Due to their hardness, martensitic steels are relatively easy to polish. In general, care should be taken to preserve the carbides.

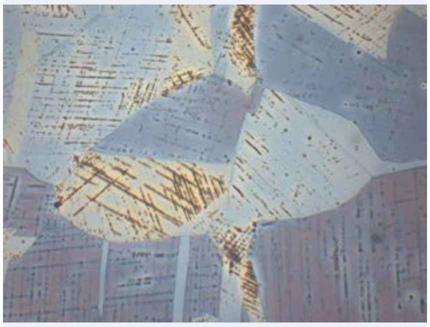


Fig. 3: Austenitic steel insufficiently polished showing deformation after etching (Beraha II) *500x*

Recommendations for the preparation of stainless steels

It is strongly recommended that for the soft and ductile stainless steels the use of very coarse grinding foil/papers and high pressures should be avoided, as this can result in deep deformation. As a general rule, the finest possible grit, consistent with the sample area and surface roughness, should be used for plane grinding. Fine grinding is carried out with diamond on a rigid disc (MD-Largo) or, as an alternative for some types of stainless steels, on a MD-Plan cloth. Fine grinding is followed by a thorough diamond polish on a medium soft cloth, and the final polish with colloidal silica (OP-S), or alumina (OP-A) removes the fine scratches. This step should be very thorough and it can take up to several minutes. A good final polish increases the chance for a better contrast (see "Etching").

Any deformation from the first grinding step, which is not removed by fine grinding, will leave its traces and can not be removed by final polishing.

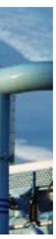


Table 1 shows a preparation method for stainless steel samples, 30 mm mounted, on the semi-automatic Tegramin 300 mm diameter. Table 2 shows a preparation method for 6 stainless steel samples, 65x30 mm, cold mounted or unmounted using Struers MAPS or AbraPlan/AbraPol, 350 mm diameter.

Electrolytic polishing

For research work or fast, general structure check, electrolytical polishing and etching can be an alternative to mechanical polishing of stainless steels, as it does not leave any mechanical deformation.

Electrolytic polishing gives excellent results for checking the microstructure (Fig. 4), but it is not suited to identify carbides. They are washed out or appear enlarged.

Before electrolytic polishing, the samples have to be ground to 1000# on silicon carbide foil/paper. The finer the initial surface the better the results of the electrolytical polish (see preparation method below).

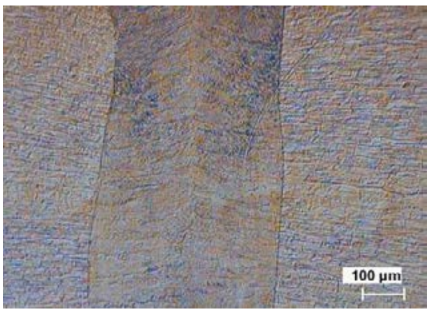


Fig. 4: Stainless steel weld, polished and etched electrolytically, DIC

Electrolyte:	A3
Area:	1cm²
Voltage:	35 V
Flowrate:	13
Time:	25 sec

External etching with stainless steel etching dish:

10% aqueous oxalic acid	
Voltage:	15V
Time:	60 sec

Preparation method for electrolytic polishing and etching of stainless steel. Grinding on SiC foil/paper 320#, 500# and 1000#.

Grinding

Step		PG	FG
Surface		SiC-Paper 220	MD-Largo
Abrasive	Type	SiC	Diamond
	Size	#220	9 µm
Suspension/ Lubricant		Water	DiaPro Allegro/Largo 9
rpm		300	150
Force [N]/ specimen		25	40
Time (min)		As needed	5

Polishing

Step		DP	OP
Surface		MD-Dac	MD-Chem
Abrasive	Type	Diamond	Silica/Alumina
	Size	3 µm	0.04/0.02 µm
Suspension/ Lubricant		DiaPro Dac 3	OP-S NonDry OP-A
rpm		150	150
Force [N]/ specimen		20	15
Time (min)		4	2-3

Table 1: Preparation method for stainless steel samples, 30 mm diameter mounted, on the semi-automatic Tegramin, 300 mm diameter.

As an alternative to DiaPro polycrystalline diamond suspension P, 9 µm, 3 µm and 1 µm can be used together with green/blue lubricant.

Etching

Etching stainless steels requires some experience and patience. The literature for etchants is extensive, and it is recommended to try a variety in order to set up an individual stock of solutions appropriate for the particular material which is regulary prepared in the laboratory.

By virtue of the fact that stainless steels are highly corrosion resistant, very strong acids are required to reveal their structure. Standard safety precautions have to be used when handling these etchants. In many laboratories the etchants mentioned in the literature will be modified according to the material to be etched or even out of personal preference. For good etching results, a sufficient final oxide polishing is essential. Following are some etchants which have proved successful in every day, routine applications.

Grinding

Step			PG	FG
	Surface		Stone 3A36	MD-Largo
	Abrasive	Type	Al_2O_3	Diamond
		Size	#150	9 µm
	Suspension/ Lubricant		Water	DiaPro Allegro/Largo 9
	rpm		1450	150
	Force [N]/ specimen		50	50
	Time (min)		As needed	9

Polishing

Step			DP 1	DP 2	OP
	Surface		MD-Mol APS	MD-Nap	MD-Chem
	Abrasive	Type	Diamond	Diamond	Silica/Alumina
		Size	3 µm	1 µm	0.04/0.02 µm
	Suspension/ Lubricant		DiaPro Mol B 3	DiaPro Nap B 1	OP-S NonDry OP-A
	rpm		150	150	150
	Force [N]/ specimen		50	25	25
	Time (min)		6	4	2-3

Table 2: Preparation method for stainless steel samples, 65x30mm, cold mounted or unmounted using Struers MAPS or AbraPlan/AbraPol, 350 mm diameter.

As an alternative to DiaPro polycrystalline diamond suspension P, 9 µm, 3 µm and 1 µm can be used together with green/blue lubricant.

Structure interpretation

Ferritic stainless steels do not respond to heat treatment. Their properties however can be influenced by cold working. They are magnetic at room temperature. The microstructure in the annealed condition consists of ferrite grains in which fine carbides are embedded. Ferritic steels used for machining contain a large amount of manganese sulfides to facilitate free cutting (Fig. 5).

Martensitic stainless steels respond to heat treatment. Martensite is formed through rapid cooling and properties can then be optimised by subsequent tempering treatment. The alloys are magnetic. Depending on the thermal treatment the microstructure can range from pure martensitic structure to fine tempered martensite.
Different alloys and various dimensions of semi-finished products require complex heat treatment temperatures and times.

Delta ferrite (Fig. 6) is usually an unwanted phase, because long annealing times of steels with high chromium content, at temperatures between 700 and 950°C, can change the delta ferrite into the hard and brittle iron-chromium intermetallic sigma phase.

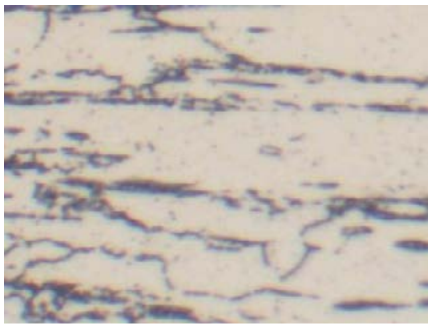


Fig. 5: Ferritic stainless steel with manganese sulfides and strings of small carbides, etched electrolytically with 10% oxalic acid 200x

Fig. 6: Tempered martensitic stainless steel with delta ferrite, etched with picric acid 75x

Chemical etching

Caution: Always follow the recommended safety precautions when working with chemical reagents.

For martensitic steels
925 ml ethanol
25 g picric acid
50 ml hydrochloric acid

For austenitic steels

1) Swab etch:
500 ml distilled water
300 ml hydrochloric acid
200 ml nitric acid
50 ml of a saturated iron-III-chloride solution
2.5 g copper-II-chloride

2) 100 ml water
300 ml hydrochloric acid
15 ml hydrogen peroxide (30%)

3) V2A etchant:
100 ml water
100 ml hydrochloric acid
10 ml nitric acid
Etch at room temperature or up to 50°C

Color etchant Beraha II:
Stock solution
800 ml distilled water
400 ml hydrochloric acid
48 g ammonium biflouride
To 100 ml of this stock solution add 1-2 g potassium metabisulfite for etching.

Electrolytic etching

For austenitic-ferritic steels (Duplex)
40% aqueous sodium hydroxide solution

All stainless steels:
10% aqueous oxalic acid

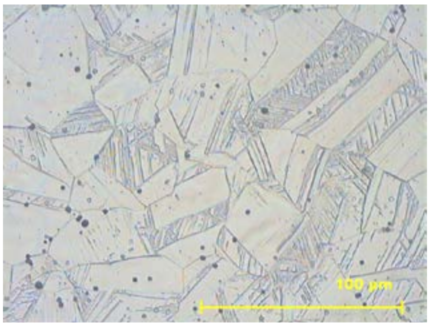


Fig. 7: Cold worked austenitic steel showing twinning, etched with V2A etchant.

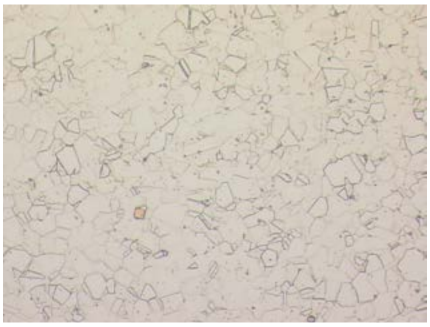


Fig. 8: Austenite with carbides and some titanium carbon nitrides 200x

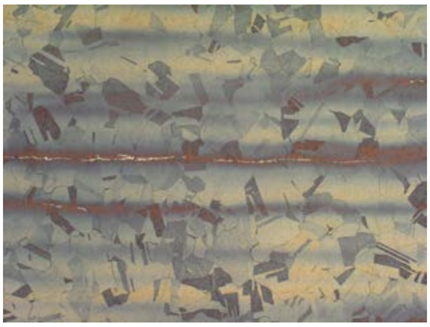


Fig. 9: Austenitic steel with strings of delta ferrite, showing microsegregations. Blue areas: depletion of alloying elements. 125x

Heating up to 1050°C and subsequent quenching removes the sigma phase and with it the embrittlement.

Austenitic stainless steels do not respond to thermal treatment, instead, rapid cooling results in the production of their softest condition. In this state they are non-magnetic and their properties are influenced by cold working. The microstructure of these steels consists of austenite grains which may exhibit twinning (Fig. 7). Exposure of these steels to elevated temperatures in the region of 600-700°C can result in the formation of complex carbides within the austenite grains. This leads to an impoverishment of chromium in the austenite solid solution, which increases the susceptibility to intergranular corrosion or oxidation.

By reducing the carbon to below 0.015% and adding small amounts of titanium or niobium, the risk of intergranular corrosion is reduced, as these elements form carbides in preference to the chrome (Fig. 8).

Delta ferrite can appear due to critical heat treatment conditions in martensitic or cold working of austenitic steels (Fig. 9).

Austenitic-ferritic stainless steels (Duplex) consist of ferrite and austenite. Electrolytic etching in a 40% caustic soda solution reveals the structure and the correct percentage of each phase can be estimated (see Fig.1, and Fig. 10 below). These steels are ductile and are specifically used in the food, paper and petroleum industries.

Summary

Stainless steels are corrosion resistant steels with high chromium and nickel contents. Ferritic and stainless steels are soft, respectively ductile, and are prone to mechanical deformation and scratching during metallographic preparation. In addition, carbides can not always be retained.

For a successful mechanical polish, it is suggested that

- coarse abrasives for plane grinding are avoided.
- fine grinding and polishing with diamond should be thorough and ensure removal of all deformation from plane grinding.
- a final oxide polish with colloidal silica or alumina should be carried out to provide a deformation free surface.

A four step procedure, completed on automatic preparation equipment, gives good and reproducible results. Stainless steels are difficult to etch chemically and the recommended etchants are very corrosive and require careful handling.

Alternatively, electrolytical polishing and etching is recommended, which gives a deformation free surface, but does not retain carbides.

Fig.10: Forged duplex steel showing blue ferrite, white austenite and fine needles of sigma phase, etched electrolytically with 40% aqueous sodium hydroxide.

150x

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Application Notes

Metallographic preparation of stainless steel

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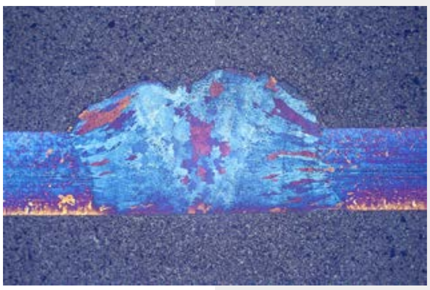

Stainless steel weld etched according to Beraha II.

20x

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