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# SENSITIZATION AND CORROSION IN STAINLESS STEEL FILTERS

## ABSTRACT

Sensitization and other corrosion problems common to stainless steel filters are discussed. Basic metallurgy and corrosion chemistry is outlined and applied to these problems. Solutions based on normal good metallurgical practice are suggested.

## 1. INTRODUCTION

1.1 Stainless steel construction is specified by filter manufacturers or buyers to achieve necessary mechanical properties and corrosion resistance. These filters are fabricated from stainless steel wire mesh which may be woven from wires smaller than one thousandth of an inch in diameter. In many applications the fine wire mesh must provide reliable filtration at differential pressures of several thousand lb. per sq. in. and withstand repeated pressure surges without deformation or fatigue failure. Cleaning procedures used on cleanable filter elements often require corrosive reagents and solvents or intensive ultrasonic cleaning to remove impacted contaminants from the filter. Failure of only a few tiny wires may result in failure of the filter and ultimate failure of the system it is designed to protect. Properly specified and processed stainless steel wire mesh can be made to reliably meet the stringent requirements imposed by these rigorous service conditions.

1.2 Reliability is not automatically achieved by specifying "stainless steel" however. Strength, ductility, and corrosion resistance inherent in stainless steel depend on the chemistry and metallurgy of the alloy which may be strongly affected by manufacturing processes. Nearly all fine stainless steel wire mesh is presently imported from Western Europe because it is less expensive due to lower labor costs. Control of alloy chemistry balance and carbon content requires careful specification and testing for selection from the available stocks of imported mesh. Mesh which is accepted for fabrication into filters must be carefully processed to avoid metallurgical damage such as carbide precipitation or sensitization and subsequent intergranular corrosion (sometimes called intergranular attack) which can result in catastrophic disintegration of the wire mesh and failure of the filter. The ultimate reliability of the completed filter depends largely on the manufacturer's technology and quality control practice in selecting and processing raw mesh.

1.3 Metallurgical damage, unlike dimensional deviations and mechanical defects, cannot easily be detected by the filter user. Nondestructive test methods for alloy chemistry and metallurgy are not available. Because of a natural reluctance to destructively test an expensive stainless steel filter, most filter users must rely on the filter manufacturer for metallurgical quality assurance. Since metallurgical requirements are not controlled by most filter procurement specifications, perhaps because of the relative difficulty in testing the fabricated filter, most defective filters are not detected until inspection after cleaning or replacement. Such failure is often described as "low bubble-point," "brittle mesh," or "rusting." Unless metallurgical examination is employed, the failure may be blamed on "flow fatigue," "poor rinsing" after cleaning, or "rough handling." Many such failures are actually due to metallurgical defects caused by improper wire mesh selection and processing by the filter manufacturer.

1.4 Metallurgical quality control, like the control of physical dimensions or functional performance, requires systematic testing based on a sound understanding of the necessary technology. A basic understanding of stainless steel chemistry and metallurgy applied to fine wire mesh is a prerequisite to achieving reliability in stainless steel filters. When such understanding is properly applied, the problem of sensitization and related corrosion can be completely controlled.

## 2. STAINLESS STEEL

2.1 Stainless steel is the generic term for all iron alloys containing more than about 10% or 12% chromium. Dozens of different stainless steel alloys are in commercial use and contain varying amounts of different alloying ingredients in addition to the required chromium to achieve particular engineering properties. Stainless steel alloys are conveniently divided into three major classes based on their crystal structure and resultant properties.

2.2 Martensitic stainless steels contain from 12 to 17% chromium as the major alloy addition and substantial amounts of carbon as a necessary addition. Heat treatment and subsequent quenching increases hardness by forming martensite, a hard metallurgical phase super-saturated with carbon and having a body-centered tetragonal space-lattice. The transformation is analogous to the hardening of ordinary carbon steel, and like iron or steel the resultant structure is strongly ferromagnetic. Corrosion resistance is greatly increased by the chromium content. Filter construction utilizes martensitic stainless steel for integral valves, bearing surfaces, and similar parts which require great hardness and good corrosion resistance. Wire mesh and other filter media are rarely made from these alloys.

2.3 Ferritic stainless steels typically contain more chromium (usually a total of 18 to 30%), than martensitic types and much less carbon. They are unable to harden by martensite formation and have a microstructure consisting of softer ferrite, a body-centered cubic lattice like mild steel or iron. These alloys have excellent corrosion resistance and are strongly ferromagnetic. Ferritic stainless steels are rarely used for filter construction.

2.4 Austenitic stainless steels constitute a further modification of the basic chromium-iron alloy and contain from 6 to 20% nickel along with 8 to 30% chromium and a low carbon content. The nickel addition promotes the formation of austenite, a phase basically different from either martensite or ferrite, having a face-centered cubic lattice structure which is non-magnetic. The nickel-bearing austenitic structure provides the ductility and superior corrosion resistance associated with these alloys. Austenitic stainless steels are widely specified for filter construction. Nearly all wire mesh and similar stainless steel filter media are made from austenitic alloys.



## 3. AUSTENITIC ALLOYS

3.1 Austenitic stainless steel compositions are based on the familiar "18-8" alloy with about 18% chromium and 8% nickel with a low carbon content. Many alloy variations have been developed to gain special engineering properties such as improved corrosion resistance or stabilization against sensitization. Several alloys are commonly used in filter construction and are usually specified by AISI (American Iron and Steel Institute) type according to their characteristic chemistry.

Austenitic Stainless Steels

| AISI Type | Analysis, Weight Per Cent | | | |
|---|---|---|---|---|
| | Cr | Ni | C | Other |
| 302 | 17 - 19 | 8 - 10 | .15 max | |
| 303 | 17 - 19 | 8 - 10 | .15 max | sulfur, selenium |
| 304 | 18 - 20 | 8 - 12 | .08 max | |
| 304L | 18 - 20 | 8 - 12 | .03 max | |
| 316 | 16 - 18 | 10 - 14 | .08 max | 2-3 molybdenum |
| 316L | 16 - 18 | 10 - 14 | .03 max | 2-3 molybdenum |
| 321 | 17 - 19 | 8 - 11 | .08 max | 5 x C titanium, min |
| 347 | 17 - 19 | 9 - 13 | .08 max | 10 x C columbium, min |

3.2 AISI Type 302: This steel is the basic 18-8 type, possessing good corrosion resistance and physical properties when properly processed. Cores, heads and bowls are often made from this alloy. Filter mesh, except for some coarse back-up mesh, is rarely constructed from type 302 because of the relatively high carbon content.

3.3 AISI Type 303: Free machining 18-8 stainless steel with added sulfur and/or selenium is called type 303. This alloy is often used for machined details because manufacturing cost is considerably lowered, but corrosion resistance is somewhat reduced by the additives.

3.4 AISI Type 304: This alloy is an "enriched" type 302 with a higher nickel and chromium content and a lower carbon content. Physical properties are comparable to type 302, but corrosion resistance is substantially improved. Most filter meshes are woven from wire of type 304 or 304L alloy which is often used for other filter components as well.

3.5 AISI Type 316: Chemically corrosive environments often require the specification of type 316 alloy for both mesh and other filter parts. The increased nickel and molybdenum content improve corrosion resistance and high temperature properties.

3.6 AISI Type 321: This steel is stabilized against sensitization by the titanium addition which "ties up" harmful carbon. Cold working properties are only slightly reduced so this alloy is used for weaving fine wire mesh, although it is not readily available. General corrosion resistance is good, but the titanium may interfere with sintering and welding or brazing of filter mesh since it is readily oxidized.

3.7 AISI Type 347: Stabilization against sensitization is accomplished by the columbium addition which gives better corrosion resistance without the attendant fabrication problems common to type 321. Unfortunately, type 347 mesh is harder to weave because it work hardens more rapidly. Type 347 stainless steel is often specified for critical applications requiring freedom from sensitization, although good quality mesh is difficult to obtain.

3.8 ELC Grades: Carbon is harmful to achieving good corrosion resistance, and some alloys have been made available in "ELC" grades (extra low carbon) containing less than 0.03% carbon instead of the usual 0.08% carbon maximum. Low carbon is specified by adding an "L" suffix to the AISI type: 304L, 316L. The low carbon helps to control sensitization problems, and is sometimes specified for both mesh and other filter parts. Stabilization by columbium addition (type 347) is more effective in preventing sensitization, but because of lower cost type 304L is more widely used for filter media.



## 4. CORROSION

4.1 Stainless steels are characterized by a phenomenal resistance to most corrosive environments when properly alloyed and processed. The key to understanding "what makes 'stainless' stainless" lies in the role of chromium in promoting passivity. When the chromium content of an iron alloy is increased to about 10 or 12%, corrosion resistance is suddenly and remarkably increased and the alloy is said to have become "passive." The mechanism of this passivity is still in question, but it is usually ascribed to the formation of an impermeable protective film of chromium-iron oxide which acts much like the self-healing oxide mono-layer which protects aluminum from corrosion.

4.2 The protective stainless steel oxide film remains intact under most corrosive conditions, but if the oxide layer is destroyed or is unable to form, the underlying metal is no longer "stainless" and will be attacked as rapidly as ordinary steel. Oxidizing environments cause negligible corrosion to stainless steels because they promote and stabilize the oxide film formation required to achieve passivity. Even strong acid solutions which rapidly attack steel have little effect on stainless steel if an adequate oxidizing potential is preserved. Corrosive conditions which are reducing in nature, or which contain certain reducing acids such as hydrochloric or hydrofluoric acid, may destroy the oxide layer and cause rapid general corrosion of the entire metal surface.

4.3 The corrosion resistance of stainless steel may be impaired by metallurgical deficiencies which interfere with the necessary passive oxide film formation or make it unstable. The most important types of corrosion failure found in austenitic stainless steels are intergranular corrosion, galvanic cell corrosion, and stress corrosion cracking.

4.4 Intergranular corrosion is usually associated with stainless steels which have become sensitized because of carbide precipitation. This form of intergranular attack is characterized by rapid corrosion of the grain boundaries with little corrosion of the metal grain itself. Intergranular corrosion often occurs in mildly corrosive environments which do not attack normal stainless steel. After corrosion begins, it is apparently accelerated by a galvanic effect between the "passive" metal grains and the "active" grain boundaries rapidly resulting in literal disintegration of the metal structure.

4.5 Galvanic cell corrosion is associated with the interaction of two dissimilar metals in a corrosive environment. A "cell" consisting of cathode, anode, electrolyte, and exterior current path is required and nearly always exists, at least on a microscopic scale. The cathode is the relatively noble or passive material or area which is protected while the more active anode is dissolved by electrolysis. A current path is usually provided by the metal structure and the electrolyte may consist of a water solution or even a trace of water in oil or other materials. The "dissimilar" metals may be two different metals used in construction, trace contamination of the metal surface, or even inhomogenieties within the same metal surface.

4.6 Stress corrosion cracking is a special form of galvanic corrosion in which the dissimilar couple is set up by differences in stress within the metal. While theoretical mechanisms are controversial, stressed areas may be considered to contain more residual energy than adjacent areas, resulting in a galvanic cell where the stressed area is dissolved as the anode. Stressed areas may result from pressures or mechanical forces due to the service application, or may be caused by residual stresses from previous manufacturing operations. Stress corrosion is most often found in corrosive environments containing chlorides, which accelerate this type of reaction, and is typified by transgranular cracking of the metal.

5. SENSITIZATION

5.1 Noble passivity is the hallmark of austenitic stainless steels, but sensitization is their Achilles heel. Sensitization is the migration of chromium into the grain boundary forming harmful chromium carbides. This leaves the grain boundary susceptible to rapid corrosion. Intergranular corrosion may later cause catastrophic failure under conditions which do not affect normal stainless steel. Only the grain boundaries are corroded, the bulk of the metal matrix being unaffected. Intergranular corrosion may be compared to dissolving the mortar from a brick wall. The resultant collapse of the structure is also similar.

5.2 Sensitization is caused by heating stainless steel in the temperature range of approximately 800 - 1500 F. Carbides, which were harmlessly dispersed or dissolved within the metal grains, diffuse as carbon along the grain boundaries, combine with chromium there, and precipitate as predominantly chromium carbides. This results in a lowered available chromium content in the grain boundaries so that passivation is prevented and intergranular corrosion may later occur. Corrosion is accelerated by a galvanic cell effect between the anodic chromium-poor grain envelope and the cathodic precipitated chromium carbide phase surrounding the grain. Sensitization requires both an appreciable free carbon content and some time of heating in the critical sensitizing temperature range.



5.3 Carbon is quite soluble in austenite at fairly high temperatures. Austenitic stainless steels are commonly annealed or "solutioned" at 1850 - 2050 F to dissolve previously precipitated carbides. At lower temperatures carbon becomes increasingly less soluble and is virtually insoluble at room temperature. Since sensitization results from precipitated or undissolved carbides, the severity of sensitization depends on the amount of carbon present in the alloy. Higher carbon alloys can become badly sensitized while lower carbon alloys are less affected. Standard stainless steel grades with 0.15% or 0.08% maximum carbon are susceptible to sensitization in proportion to their carbon content. Low carbon grades with less than 0.03% carbon are more resistant, but severe sensitization has been shown to occur on heating for 2 hr in the sensitizing temperature range with as little as 0.003% carbon precipitated.

5.4 Heating for a period of time in the sensitizing temperature range is required for harmful carbide precipitation to occur. At temperatures higher than about 1500 - 1650 F carbides are relatively soluble and precipitation is prevented. At temperatures lower than about 800 F carbides are not soluble, but diffusion processes proceed too slowly for carbon to move to the grain boundary at a rate fast enough to cause sensitization. The intermediate temperature range of about 800 - 1500 F represents a compromise between solubility and diffusion rate which results in carbide precipitation which is most severe at about 1200 F. Sensitization also requires time. The actual time required for damaging sensitization depends on the temperature, alloy carbon content, degree of cold work, and many other factors, and may range from a few minutes to many hundreds of hours. Surprisingly enough, short exposure times can be much more damaging than long time heating because thin carbide films which are first formed may agglomerate on continued heating into discontinuous particles which cause less structural damage during corrosion.

5.5 Sensitization depends on time, temperature and carbon content and manufacturing processes must carefully balance these variables to achieve reliable quality in austenitic stainless steel constructions. The carbon content of incoming stainless steel materials, such as bar stock and especially woven wire mesh, must be analyzed and carefully controlled to acceptably low levels. Since sensitization may occur in as-woven mesh, metallurgical examination must also be employed. If the metal will later be heated to high temperatures during welding, brazing, annealing, sintering, or other heat treating operations, surface contamination with carbonaceous materials, such as oil or drawing compounds, must be avoided or carefully removed to prevent diffusion into the metal. Welding processes must be designed to minimize heating time in the sensitizing temperature range or should require annealing after welding. Heliarc or TIG welding processes introduce much less contamination and damaging heat than conventional open arc or torch welding methods. Electron beam welding techniques, which are now commercially feasible, are conducted in high vacuum and require less than 1/25 as much heat as Heliarc welding for an equivalent weld. Silver brazing operations are especially critical since they are conducted near the maximum sensitizing temperature of 1200 F. Special precautions in designing thin brazed sections and placing of chill blocks are required to avoid damaging heat exposure. Acetylene torches can cause carbon contamination from reducing flames; hydrogen-oxygen torches are preferable. Furnace brazing with nickel or other filler alloys also poses special processing problems which must be recognized and controlled. Sensitization can be controlled in standard alloys by observing sound manufacturing practice. Sensitization can be eliminated by specifying columbium stabilized type 347 stainless steel if the additional cost is warranted.

5.6 Stabilized alloys prevent sensitization by the addition of columbium (type 347) or titanium (type 321) which has a greater affinity than chromium for carbon. Essentially all of the carbon is harmlessly precipitated as carbides within the grain and chromium carbide formation is prevented. Subsequent sintering or welding operations may then be performed without the risk of sensitization providing normal good practice is observed. Stabilized stainless steel is specified when sensitization and subsequent intergranular corrosion cannot be tolerated. Stabilized bar stock is widely available, but is more difficult to work than the free-machining grades and is not required for many applications. Stabilized wire mesh is also available but costs about twice as much as regular type 304 mesh because it is more difficult to weave in good quality. Delivery is often slow when the finer weaves are imported from Europe.



## 6. SENSITIZATION TESTS

6.1 Strauss testing is an accelerated corrosion test for detecting harmful sensitization in austenitic stainless steels. Stainless steel samples are boiled in a solution of dilute sulfuric acid and copper sulfate (47 ml/1 concentrated $H_2SO_4$, sp. gr. 1.84 and 13 g/l $CuSO_4 \cdot 5H_2O$ dissolved in distilled or deionized water) for successive 72 hr periods. An air reflux condenser is usually employed to prevent evaporation of water from the test solution. Sulfuric acid acts as a reducing corrosive in this concentration and will rapidly attack grain boundaries which are sensitized and incapable of passivation. Cupric sulfate acts as a protective oxidizing agent and stabilizes normal stainless steel against attack. The net result of this interaction is rapid intergranular corrosion of sensitized stainless steel while normal alloys are unaffected. Large stainless steel parts may not show visible corrosion for many hours or even days, but mesh which has become badly sensitized usually disintegrates into powder within a few hours. This rapid corrosion is due to the large exposed surface area and very small wire section of the fine wire mesh.

6.2 Huey testing is also used to detect sensitization in a manner similar to Strauss testing. Samples are boiled for consecutive 48 hr periods in refluxed 65% nitric acid. Concentrated nitric acid passivates normal stainless steel with no attack, but preferentially dissolves the grain boundaries of sensitized alloys. Strauss testing is usually preferred to Huey testing because less obnoxious reagents are employed and test results are often more reliable for some alloys.

6.3 Metallographic examination is also employed to detect sensitization. The stainless steel specimen is sectioned, ground, and polished to prepare a surface which shows the metal grain structure. The polished surface is "developed" by etching with special reagents. Anodic etching with oxalic acid or sodium cyanide solutions is usually employed. The etchants preferentially dissolve the grain boundary carbide envelopes, leaving "holes" which can be observed with a microscope. Semi-quantitative measurement of the severity of sensitization can be obtained by estimating the percentage of the grain perimeter which contains carbides. Usually 50% or more grain boundary carbides will produce rapid catastrophic failure by subsequent intergranular corrosion.

## 7. ANNEALING AND SINTERING

7.1 Austenitic stainless steel wire mesh used for filter construction is often sintered or diffusion welded. Sintering serves the dual purpose of annealing the mesh wires which have been heavily cold worked during previous drawing and weaving operations and bonding or "welding" each tiny wire to its neighbor at points of contact. Annealing is necessary to relieve stresses and soften the mesh for further forming operations and to regain optimum corrosion resistance by dissolving or "solutioning" previously precipitated carbides and by converting the ferrite formed during cold working back into austenite. Sintering is accomplished in a process similar to annealing by holding the mesh for a somewhat longer time at a higher temperature so that diffusion can occur between contacting wire surfaces. Sintering and annealing process details are usually considered to be proprietary and may differ between manufacturers, but common denominators of good metallurgical practice apply to all satisfactory processes.



7.2 Annealing and sintering is best conducted in a pure dry hydrogen atmosphere or in a vacuum furnace. Cracked ammonia atmospheres, which are commonly employed for commercial heat treating operations because of their lower cost, are less satisfactory because the dissociated nitrogen-hydrogen mixture can cause nitride formation resulting in decreased corrosion resistance and lower physical properties such as ductility and fatigue resistance in the mesh. Special decarburization processes using "wet" hydrogen may be employed to lower the mesh carbon content or to eliminate carbonaceous "spot" contamination. Subsequent deoxidation is necessary and the final atmosphere or vacuum must be "clean" and free from contaminants such as oxygen (air), water vapor, or hydrocarbons which can react to cause metallurgical damage.

7.3 Temperature control during annealing or sintering is quite critical. Low temperatures may result in incomplete solutioning or poor sinter bonding while high temperatures may cause alloy segregation, excessive grain growth, or incipient melting. Both cases result in lowered mesh quality and should be avoided. Temperature measurement in atmosphere furnaces is best accomplished with thermocouples located at the actual location of the mesh itself. Since platinum/platinum-rhodium and chromel/alumel thermocouples used to control the furnace temperature are unreliable in hydrogen atmospheres, tungsten/tungsten-rhenium thermocouples are usually employed. Vacuum furnaces may use any suitable thermocouple or radiation pyrometer for temperature measurement. While various temperature measuring techniques may be employed for specific processes, they must all meet the common requirement of providing reproducible and reliable process temperature indication and control.

7.4 Cooling rates after sintering or annealing must be controlled to avoid sensitization in unstabilized alloys with appreciable carbon contents such as common type 304 wire mesh. Freedom from carbide precipitation can be achieved only by very rapid cooling or quenching from the temperature range of about 1650 F down to 800 F within less than 2 or 3 minutes. Since this is often impractical, even in cold-wall vacuum furnaces or continuous-strip annealing furnaces, good control over sensitization is best achieved by regulating the carbon content to a low level and cooling as rapidly as necessary. Atmosphere retorts should be cooled by air blast and water quenching techniques to avoid long hold times in the sensitization temperature range. Slow cooling increases retort life and reduces costs, but plays hob with mesh quality.

7.5 Annealing may be combined with other operations such as furnace brazing if proper engineering practices are specified and observed. Selection of nickel base or other brazing alloys must allow annealing at the required temperature without excessive solutioning or undercutting of the fine wire mesh. Brazing fixtures must be designed with low mass and proper section to provide adequate support and still allow control over heating and cooling rates. Poor brazing practices may produce weak or brittle structures with low fatigue life or inferior corrosion resistance. Proper brazing technology, properly applied, can reliably produce satisfactory filters.