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Selection and performance of stainless steels and other nickel-bearing alloys in sulphuric acid

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INTRODUCTION

Sulphuric acid, H₂SO₄ counts among the most important industrial chemicals. Nonsocialist world production approximates 100 million metric tons per annum. Although highly corrosive, concentrated sulphuric acid at ambient temperatures is customarily handled in carbon steel equipment. The aggressiveness of the acid varies with its concentration and temperature, with its velocity relative to exposed surfaces and with the nature of possible contaminants.

This paper examines the behaviour of several stainless steel and nickel-bearing alloy compositions, taking account of the variables enumerated in the preceding paragraph. It begins with a discussion of alloys suitable for equipment used in the manufacture of sulphuric acid and in the storage and handling of cold, concentrated commercially-pure acid; the text then presents guidelines for the selection of corrosion resisting materials, including typical applications which involve:

Dilute acid	0 to 20% concentration
Intermediate acid	20 to 70% concentration
Strong acid	more than 70% concentration

This paper does not cover concentrations in excess of 98%, e.g. anhydrous, fuming sulphuric acid or oleum.

The various alloys interact with sulphuric acid in a complex manner. A single process may involve not only a wide range of sulphuric acid concentrations but also the presence of such contaminants as ferric and cupric ion, nitrates, chlorides, ammonia, sulphur dioxide and metallic oxides. While the hydrogen ion activity of sulphuric acid attains its maximum at about 5N, 25% concentration, its activity diminishes above and below this level. The foregoing variables exert significant influence not only on the oxidizing capacity of sulphuric acid solutions but also on the performance of stainless steels and nickel-bearing alloys in contact with them.

Table 1 lists the alloys considered herein.

	Stainless steels								High-nickel alloys			
Generic designation	304L	316L	317LM	2205	904	904hMo	28	20	825	G-3	C276	625
UNS designation	S 30403	S 31603	S 31725	S 31803	N 08904	N 08925	N 08028	N 08020	N 08825	N 06985	N 10276	N 06625
Nominal analysis	%	%	%	%	%	%	%	%	%	%	%	%
C max	0.03	0.03	0.03	0.03	0.02	0.02	0.02	0.03	0.025	0.015	0.015	0.1
Cr	18.0	17.5	18.0	22.0	20.5	20.5	27	20.0	21.0	22.0	16.0	21.5
Ni	10.0	13.5	13.0	3.0	25.0	25.0	31.0	37.5	42.0	41.0	57.0	51.0
Mo		2.5	4.5	3.0	4.7	6.0	3.5	2.5	3.0	7.0	16.0	9.0
Cu					1.5	1.0	1.0	3.5	2.5	2.0		
N			0.14	0.15		0.12						
Cb/Ta								0.3		0.3		3.65
Others								Cb	Al,Ti	Co,W	Ti	Al,Ti,Co

Table 1 Representative stainless steels and high-nickel alloys for sulphuric acid service

Sulphuric acid manufacture

Technical and industrial development of a nation is often gauged by its per capita consumption of sulphuric acid because the acid is so widely used in chemical processes, in fertilizers and in petroleum refining.

The past 15 years have witnessed major changes in sulphuric acid manufacture. Plants are growing steadily larger and more energy-efficient; they are designed to minimize process energy consumption while maximizing energy recovery and utilizing steam to generate electricity. The Claus process is rapidly being supplanted by new technology. *Figure 1*, a simplified flow diagram, presents three alternative routes for the generation of SO_2 . The first of these is combustion of sulphur; the second is recovery of SO_2 from metallurgical processes, such as roasting of pyrites and other sulphide ores. The third alternative route is regeneration from spent acid.

Because such acid tends to be contaminated, this process sequence customarily begins with thermal degeneration, followed by scrubbing of the resultant sulphur dioxide prior to cooling and drying.

A catalytic converter represents the most important part of the plant. SO_2 at about 425°C (800°F) reacts with excess air in the presence of vanadium pentoxide catalyst during several passes through the catalyst beds inside the converter. Since the reaction is highly exothermic, the gases traverse a cooling chamber after each pass through the catalyst bed. The SO_3 then enters the absorbing towers where it reacts to form sulphuric acid. About 35% of the heat generated during the process serves to raise the temperature of the sulphuric acid produced. It has been found that, in the temperature range $155\text{-}190^\circ\text{C}$ ($310\text{-}370^\circ\text{F}$), relatively low corrosion rates prevail for stainless alloys such as

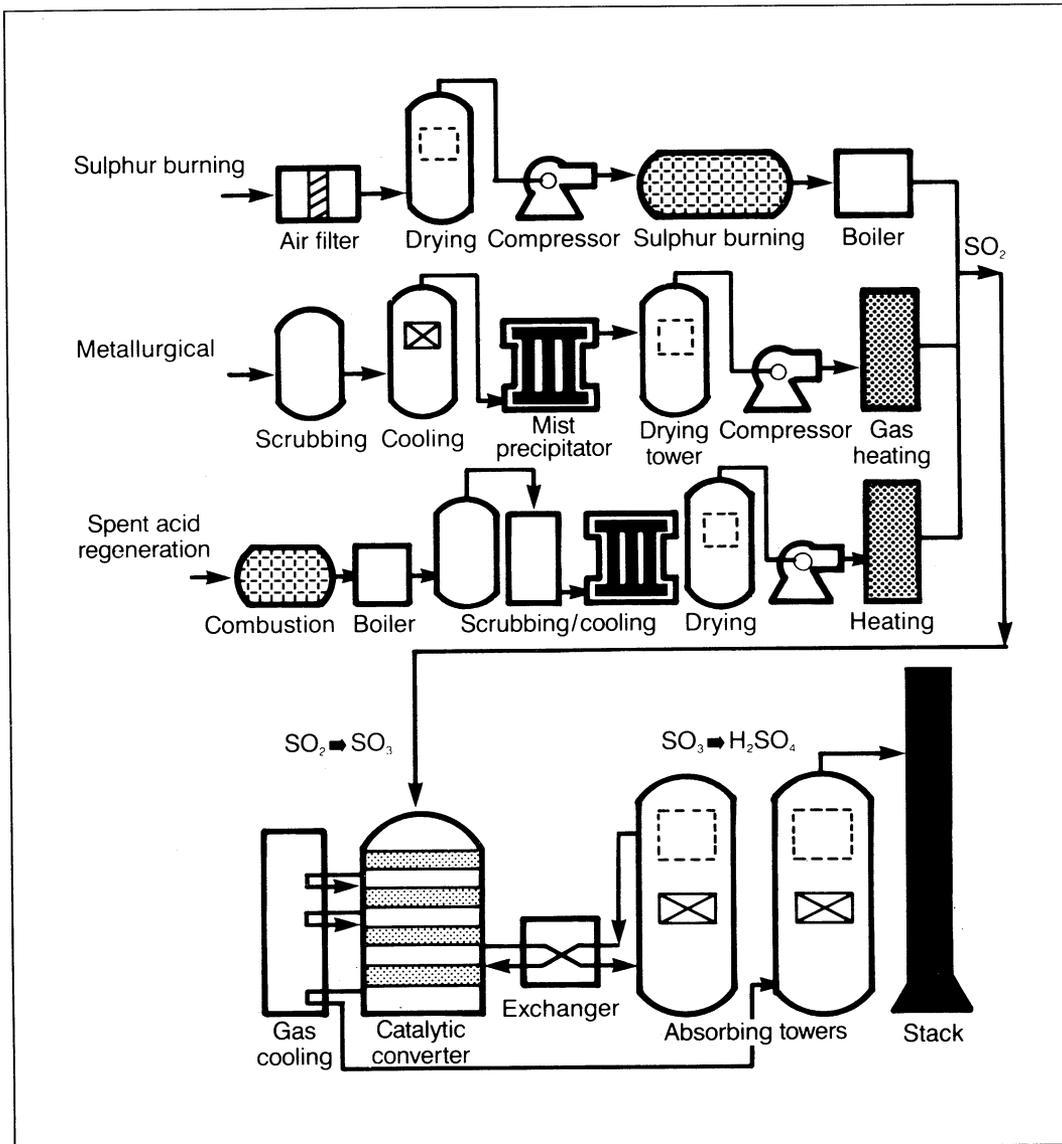


Figure 1 The new technology of sulphuric acid

Handling of cold, concentrated sulphuric acid

At concentrations of 93% and 98%, commercial sulphuric acid can be stored in carbon steel tanks, provided that iron contamination does not present a problem. The term *concentrated sulphuric acid* generally refers to the concentration range from 90% to 100%. Carbon steel provides satisfactory corrosion resistance in the concentration range between 80% and 100% at ambient temperatures, in static conditions or at low velocity. In this service, carbon steel depends for its corrosion resistance on a ferrous sulphate film that is easily removed by erosion or turbulence. Rapid corrosive attack takes place in the absence of that film. Therefore, the maximum permissible design velocity in carbon steel is set at two feet per second. Turbulent flow occurs in elbows, tees, valves and rough or uneven internal pipe surfaces, e.g. steps or ledges created by improper pipe alignment during welding. Turbulence so created may extend over a distance of several pipe diameters downstream. Moreover, the solubility of the ferrous sulphate layer is directly proportional to temperature. For carbon steel, 50°C (120°F) is considered the upper limit in acid of 92% to 98% concentration; between 78% and 90% concentration, the corresponding temperature is only 25°C (80°F).

The remainder of this section suggests materials that are suitable for equipment and components in contact with concentrated sulphuric acid:

Storage tanks These may be carbon steel, provided that a corrosion allowance of between 1/8 and 1/4 inch, 3mm and 6mm, above the design wall thickness can be accommodated. Discharge nozzles should be AISI type 316L, UNS S 31603.

Piping Ductile iron for large diameter piping. AISI type 304L, UNS S 30403 or AISI type 316L for piping having an internal diameter of 3 inches, 7.6 cm, or less.

Valves and pumps Type CF-3M, UNS J 92800

(19% Cr, 11% Ni, 2.5% Mo) and CN-7M (20% Cr, 29% Ni, 2.5% Mo, 3.5% Cu) are usually selected to cope with velocity effects. CN-7M and CD4-MCU (26% Cr, 5% Ni, 2% Mo, 3% Cu) can be specified for valves and pumps at little additional cost. These alloys also offer resistance to dilute acids up to specific limiting temperatures.

Tank cars and road tankers An iron sulphate sludge tends to accumulate in these tanks and must be removed periodically. Because handling and brushing of the internal surfaces is unavoidable during sludge removal, the tanks should be coated internally or preferably specified in AISI type 304L. Lightweight road tanker construction favours stainless steel, either in AISI type 304L or AISI type 316L. Tanks made from these alloys can carry a wide range of products, are easy to clean and reduce the hazards of product contamination.

When concentrated sulphuric acid is fed into an aqueous stream, the temperature of the diluted acid rises. In order to prevent attack by the hot acid, the mixing section is usually a spool of 14.5% silicon iron, or steel lined with Kynar or Teflon. A check valve is provided to prevent backup of the intermediate-strength acid. Corrosion rates inside a dilution device usually exceed the capabilities of the various stainless steels and Alloy 20, UNS N 08020, 20% Cr, 37.5% Ni, 2.5% Mo, 3.5% Cu. Cast or wrought control valves could be made from Alloy C-276, UNS N 10276, 16% Cr, 57% Ni, 16% Mo.

In summary, the major problems associated with handling and storage of concentrated sulphuric acid relate to:

1. Hygroscopic effects
2. Exothermic temperature rise on dilution
3. Velocity effects that erode or otherwise damage protective surface layers

Selecting corrosion-resistant materials

Corrosion is a complex and baffling phenomenon. Seemingly unimportant variables, such as minute amounts of impurities, can materially change the performance of alloys. Nevertheless, concisely summarized information is useful in that it permits a bird's eye view of a situation, a preliminary screening that helps to minimize the number of materials to be considered and evaluated. *Figure 4*, an iso-corrosion chart based on pure sulphuric acid, presents such a screening tool. It summarizes the limits of usefulness for some of the alloys shown in *Table 1*. *Figure 4* also indicates the inhibiting effect which metal sulphate corrosion products exert on the performance of stainless steels.

Effect of contaminants

Oxidizing contaminants include sulphur dioxide, dissolved oxygen, nitrite or nitrate ion, chromates or vanadates and oxidizing metallic cations — notably ferric and cupric ion. If these are present in sufficient quantity, they can passivate stainless steel. *Figure 5* demonstrates the relevant effect for AISI type 304 and AISI type 316. The effect of ferric ion is not significant in highly-concentrated acid, 96% concentration and beyond. Great care must be exercised if oxidizing agents are to be relied on as corrosion inhibitors to passivate alloys. This practice can be very dangerous, especially in the presence of chlorides or in equipment that contains undercuts or

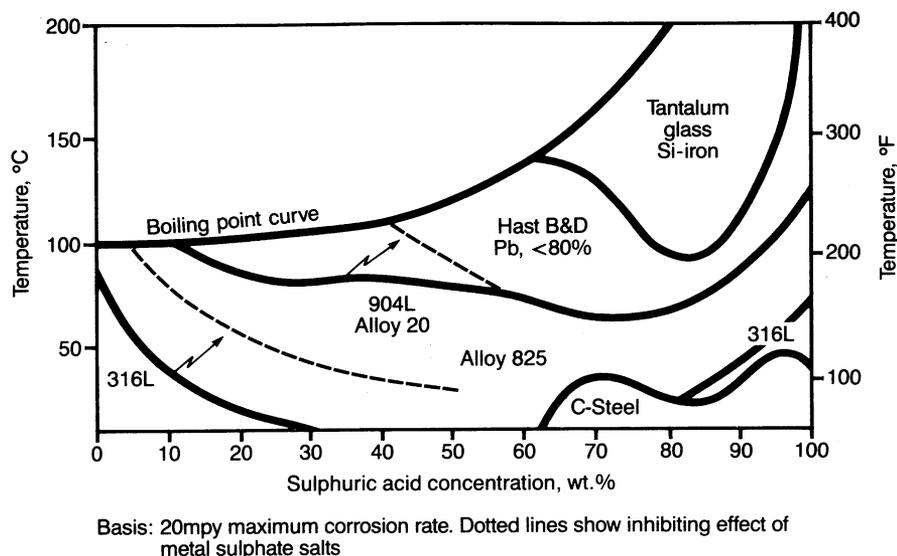


Figure 4 Materials selection chart for sulphuric acid

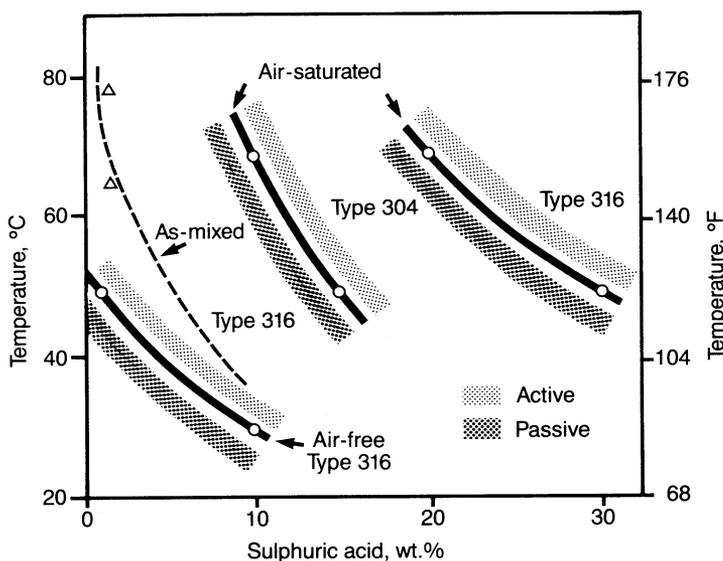


Figure 5 Active-passive regions for AISI types 304 and 316 stainless steel

crevices wherein the inhibitors may be locally depleted.

Reducing contaminants include the halides — i.e. chlorides, fluorides, bromides and iodides — various compounds of arsenic and antimony as well as hydrogen sulphide. They adversely affect the performance of stainless steel.

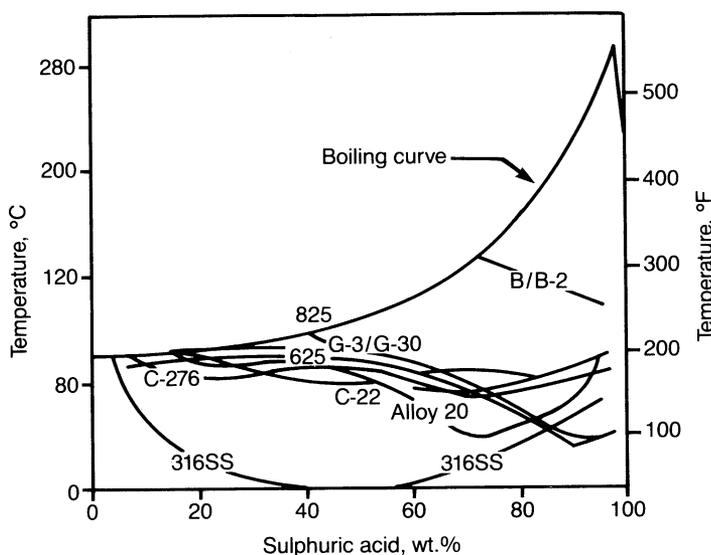
Specialty alloys

Iron- and nickel-base alloys, have been developed to cope with intermediate concentrations of sulphuric acid in conditions beyond the capability of AISI type 316L. These alloys can be specified across the full range of acid concentrations up to a temperature of 65°C (150°F). At concentrations between 65% and 75% they are effective at somewhat lower temperatures because oxygen solubility is reduced in this concentration range. Comparable performance is obtained from the 6% molybdenum alloy 904hMo, UNS N 08925 and Alloy 28 UNS N 08028 stainless steels as well as the high nickel alloys 625, UNS N 06625, G-3, UNS N 06985 and C-276. The per-

formance of some representative compositions in reagent sulphuric acid is compared in *Figure 6*. The differences between their effectiveness are governed by the nature of the impurities that are present and which can, in some cases, extend the passivity of the alloys up to a temperature of 80°C (175°F), or even to the boiling point in the concentration range of up to 40%.

The literature often describes corrosion resistance in terms of iso-corrosion lines for rates of 5mpy, 20mpy or 50mpy. The acronym *mpy* stands for mils per year, 1 mil = 0.025mm. Corrosion rates below 3 mpy are meaningful for passive film-forming alloys; alloys with corrosion rates above 20mpy should not be used.

In nonpassivating nickel-base alloys such as Alloy 400, UNS N 04400 and Alloy B-2, UNS N 10665, corrosion resistance usually depends on the insolubility of the protective surface film of metallic sulphate corrosion product. In the absence of oxidizing species, these alloys perform better than the chromium-bearing grades in acids of less than 25%



The iso-corrosion lines are drawn for .05mm/y, 20mpy, corrosion rate.
Source: N. Sridhar

Figure 6 Comparative behaviour of several Ni-base alloys in reagent sulphuric acid

concentration and temperature ranges up to the boiling point. Under these conditions, Alloy B-2 may be specified in concentrations up to 50%, while Alloy 400 may be used at temperatures up to 65°C (150°F) in the range of 25-50% provided that no accumulation of cupric corrosion products can occur. The resistance of Alloy B-2 diminishes at 160°C (320°F) for acid concentrations of 70%, and at 115°C (240°F) for acid concentrations of 100%. Alloy B-2 and Alloy 400 will also resist chloride ion contamination in the acid.

Casting alloys

Comparable casting alloy specifications are available for many of the alloys mentioned above. Although their composition usually deviates somewhat from the corresponding wrought specifications, the casting alloys offer substantially similar corrosion resistance. Traditional high-nickel casting alloys, such as Illium and Chlorimet, have been largely replaced by compositions typically containing 32% Cr, 35% Ni, 16% Fe, 6% Co, 4% Mo and 3.5% Si. These offer exceptionally good resistance to acids at temperatures up to 125°C (260°F), even under conditions in which erosion or abrasion may occur.

Miscellaneous materials

At acid concentrations below 80%, lead has been a traditional material of construction. However, its use has decreased because the metal is toxic and because other more cost-effective materials have become available. In weak sulphuric acid, titanium is of interest only if powerful oxidants are present, as — for example — in high-pressure leaching of copper ores. Zirconium can be useful in acid concentrations up to about 60% although care is required at approximately 60% and 80°C (180°F) because, in these conditions, the corrosion products are pyrophoric and pose an ignition hazard. Tantalum may be used at all acid concentrations up to the atmospheric boiling point; however, in concentration ranges between 77% and 100%, it should not be specified at temperatures above 190°C (375°F). Similar performance may be expected of glass although neither it nor tantalum should be used in the presence of fluorides where glass suffers direct chemical attack while tantalum undergoes rapid deterioration as a result of hydriding phenomena. The 14.5% silicon-iron alloy can handle the full range of sulphuric acid concentrations up to the boiling point but is also attacked by fluoride contamination that removes the protective siliceous film.

Major markets for sulphuric acid

In the United States, the largest single end use for sulphuric acid is the manufacture of phosphoric acid for the production of fertilizers and detergents. Between 2.6 and 2.9 tons of H_2SO_4 are consumed per ton of P_2O_5 , calculated on a 100% basis. The reaction between sulphuric acid and phosphate rock generates large quantities of calcium sulphate as a waste product.

The organic chemical industry is the second largest user of sulphuric acid. Production of plastics and synthetic fibres is only one example of the numerous chemical processes in which sulphuric acid plays an important part as a reactant.

Production of titanium dioxide consumes large quantities of sulphuric acid. TiO_2 is one of the most important white

pigments incorporated in paints and plastics. Spent acids are produced in the concentration range between 20% and 23% with metallic sulphate contents of 7% to 15% by weight. In the metals industry, sulphuric acid plays an important part in pickling of ferrous and nonferrous materials and in the recovery of copper, nickel and zinc by hydrometallurgical extraction from low grade ores. Significant quantities of sulphuric acid are consumed in the petroleum industry where the acid acts as a catalyst for alkylation, combining olefins and isobutanes to make high-octane blending stock. Acid treatment of distillates and extraction of ethanol from refinery gases represent further outlets for sulphuric acid in the petroleum and petrochemical industries.

Typical applications of stainless steel and nickel-containing alloys in sulphuric acid service

Acid pickling Hot-rolled, forged or heat-treated steel parts are pickled in hot sulphuric acid solutions in order to remove oxide scale. Acid concentrations range from 5% to 15% at temperatures varying between 60°C and 95°C (140°F and 200°F). Alloy 400, containing 66.5% nickel and 31.5% copper, is the accepted material of construction for crates, racks, baskets, hooks, chains and other components that support the steel during the pickling sequence. The pickling reactions appear to consume any oxygen which may have been dissolved in the acid. Hydrogen evolved by reaction of the acid with the steel helps to keep the metallic ion contaminants in a reduced state. Moreover, Alloy 400 may become galvanically protected by the steel parts with which it comes in contact. Alloy 400 racks are also chosen for pickling brass and copper and for the racks that hold the product to be pickled prior to enameling or carburizing.

Alloy 825, UNS N08825 may be specified for most pickling applications. In one case it replaced Alloy 400 in a hook that conveyed wire bundles through an acid bath. The first stage was a dip into H_2SO_4 at a concentration of 12% and a temperature of 70°C (160°F) in the presence of sodium dibromate. This treatment was followed by a dip into a mixture consisting of 60% H_2SO_4 , 25% HNO_3 and 0.2% HCl by weight. Alloy 825 achieved excellent results not only in the hook but also the heating coils in both pickling baths.

Hydrometallurgy AISI type 316L is satisfactory for the tanks, piping and other equipment in a copper refinery. Acid concentrations vary between 13% and 15%; the temperature in the electrowinning bath varies from 50-65°C (120-150°F). Cupric sulphates inhibit corrosion at the surface of the alloy. However, AISI type 316L is not suitable for the heating coils wherein steam at 100psi causes the metal to attain temperatures beyond the limit permissible for AISI type 316. Alloy 20 or 904L would be more appropriate. In zinc refineries, extensive applications for stainless steel include AISI type 304 for cathode starting sheets and AISI type 316 for evaporators and crystallizers that handle byproducts such as copper sulphate. The finished crystalline product is conveyed and stored in equipment made from AISI type 304. In *Figure 4*, dotted lines show the inhibiting effect of metal sulphate salts on the performance of AISI type 304 and AISI type 316.

Organic Sulphation and Sulphonation Sulphuric acid at 93% concentration and at temperatures between room and 60°C (140°F) is suitable for sulphation of oils to make wetting agents and penetrants. Usually processed in batches, the sulphation product is salted out, washed and neutralized with caustic soda. Frequently, all of these steps take place in the same vessel. Alloy 400 is specified not only for the vessel but also the heating coils, pipe, agitators and pumps. A wide variety of detergents and wetting agents is now made by sulphation of fatty alcohols or fatty esters. Steel and cast iron reaction equipment may be used provided that the sulphuric acid concentration is not permitted to fall below 80% during the reaction. If the concentration of the acid is likely to fall below that limit, Alloy 400 or Alloy 20 should be specified.

Petroleum refining In sulphuric acid alkylation, the hydrocarbons are emulsified in 98% acid and reacted at tempera-

tures in the range of -1°C to +10°C (30°F to 50°F). The acid remains fairly concentrated, diluting to about 88%. Mild steel offers satisfactory corrosion resistance except in areas where high velocity may be encountered, such as in pumps, valves or return bends for which AISI type 304 or AISI type 316 should be specified.

Acid treatment of lubricating oils and other distillates is generally carried out in 83% acid at 65°C (150°F) although some processors may operate at temperatures as high as 105°C (220°F). The acid is diluted with water in order to facilitate separation of the sludge which arises after treatment. Batch operations commonly utilize Alloy 400 which can also be specified for the high-speed centrifugal equipment that accomplishes the separation of oil from sludge. Because acid and oil remain mixed in this equipment, corrosion is usually not severe at moderate temperatures.

Ammonium sulphate Ammonia-rich gases pass upward through a tower countercurrently to a stream of ammonium sulphate solution that contains sulphuric acid at a temperature of 50°C (120°F) and concentrations between 4% and 10%. The exiting solution is pumped to a crystallizer and thence to centrifuges or filters. In this process sequence, both Alloy 400 and AISI type 316L have performed well for the scrubber, crystallizer, mother liquor mixing tank and the settling tanks. In an alternative process sequence, ammonium sulphate is generated by reaction of synthetic ammonia gas with concentrated sulphuric acid in a mother liquor of concentrated ammonium sulphate. Operating temperatures range between 80°C and 105°C (180°F and 220°F). While Alloy 400 is unsuitable in this temperature range, AISI type 316L may be used for reactor and crystallizer equipment.

Phosphate fertilizers Phosphate rock feed material enters the system as a slurry that reacts with a mixture of concentrated sulphuric acid and partially spent acid that has been recycled from the filtering operation. The agitator paddles and shafting are commonly made from Alloy 20, Alloy 825 or Alloy 904L. The same alloy selection applies to a slight variant of the foregoing process sequence wherein the sulphuric acid is prediluted with filter acid prior to entering the reactor. However, the service life of the foregoing alloys has proved to be significantly shorter in the newly-designed isothermal reactors that are characterized by higher velocities and more severe agitation and abrasion by the gypsum solids. In these settings, Alloy G-3 has proved to be successful for the draft tube, shafting and impeller. Depending on the type of feedstock, corrosive attack in these units can become exceptionally severe. Some phosphate rocks contain high concentrations of fluorides and chlorides in addition to oxidizing metal salts, chlorates and manganese peroxides. Occasionally, only alloys such as Alloy C-276 or Alloy 625 will suffice in this environment.

Flue gas scrubbers After the effluent gases from power-generating plants have been scrubbed free of sulphur dioxide, these wet gases are routinely mixed with some 10% by volume of nonscrubbed hot gases. The object of this routine is to raise the temperature of the scrubbed gases from 60°C to 70-80°C (135°F to 160-180°F) in order to increase stack efficiency. This practice has brought about extremely severe corrosion in the outlet

ducting, the stack breeching section and the stack liner. The attack is particularly virulent during startup and abnormal operating conditions when a higher-than-normal percentage of sulphur dioxide-bearing flue gas may bypass the scrubbers. Since the mixed gases will be at a temperature below the dew point, they will throw off condensate. The adiabatic saturation curve for sulphuric acid, *Figure 7*, helps to determine the approximate concentrations of sulphuric acid

to be expected in the condensing droplets. Under normal operating conditions, these concentrations range from 25% to 55% sulphuric acid but they can rise to as high as 80% under full bypass conditions. Several plants have successfully applied Alloy 625 and Alloy C-276 for the exhaust ducting, breeching section and stack liners. High-alloy cladding and thin-gauge alloy liners have been employed as an added economy measure.

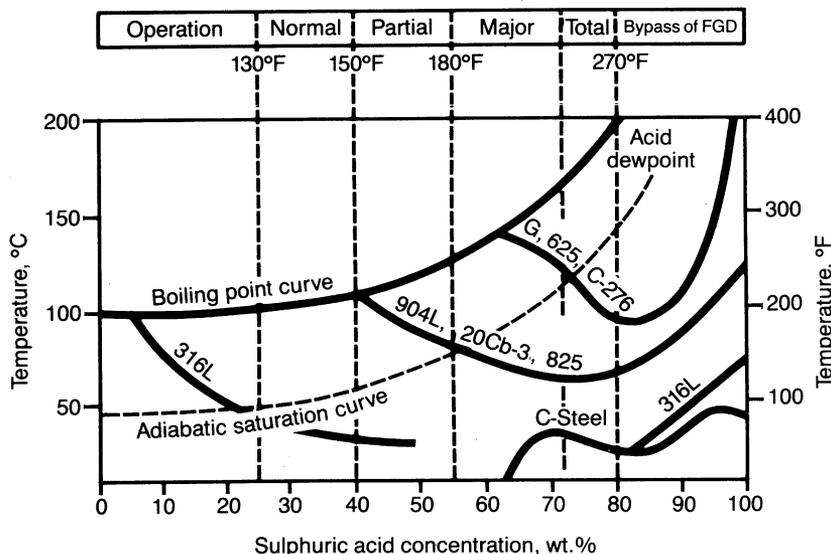


Figure 7 Adiabatic saturation curve showing H_2SO_4 concentration for various temperatures and operating conditions. Basis: 20 mpy maximum corrosion rate, oxidizing conditions.

Summary

Nickel, chromium, molybdenum, copper and silicon are the most important of the elements that enhance the corrosion resistance of alloys in sulphuric acid service. Relatively new alloys, such as Alloy 904L and Alloy 28, have proved to be highly corrosion resistant as well as cost effective. Please refer to *Table 3* for relative cost indications of the various alloys. Many of these alloys resist sulphuric acid both under reducing and oxidizing conditions. Established alloys that have performed satisfactorily during more than 25 years include Alloy 20 and Alloy 825. Under the most severe conditions, Alloy 625 and Alloy C-276 receive consideration. Bronzes and nickel-base alloys containing respectively 28% molybdenum or 14.5% silicon have recently lost ground to the stainless steels and nickel-bearing alloys that rely for their corrosion resistance on a passivated surface layer. New sulphuric acid

manufacturing flowsheets have been developed and promoted by major chemical plant constructors seeking maximum operating economy through energy savings and low maintenance costs. These installations make effective use of AISI type 304, AISI type 316 and Alloy 904.

In general, three strategies may be employed to tackle corrosion problems that arise with equipment in sulphuric acid service:

- Addition of an oxidizing agent
- Application of anodic protection
- Selection of a more corrosion-resistant alloy

The foregoing paper presents only a brief introduction to the subject of metals and alloys in sulphuric acid service. The attached literature references should be consulted for more detailed information.

Generic designation	316L	317LM	2205	904	904hMo	28	20	825	G-3	C-276	625
UNS designation	S 31603	S 31725	S 31803	N 08904	N 08925	N 08028	N 08020	N 08825	N 06985	N 10276	N 06625
Plate	1.0	1.4	1.4	2.0	2.6	2.6	2.8	2.8	4.0	6.8	5.9
Tube, seamless	1.0	1.2	1.2	1.6	2.2	1.8	-	2.2	3.5	5.0	4.3
Tube, welded	0.5	0.6	0.6	1.3	-	-	1.8	1.8	2.1	3.8	3.3

Table 3 Relative cost of stainless steels and high-nickel alloys

316L plate = standard

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