

Choose Materials for High-Temperature Environments

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High temperatures, stresses, and the presence of elements such as oxygen, sulfur, or the halogens can add up to trouble. Here is how to screen candidate alloys for such service.

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Materials are selected on the basis of service requirements, notably strength, so corrosion resistance (stability) may not be the primary design consideration. Assemblies need to be strong and resilient to the unique loads and stresses imparted on them, which can include significant temperature changes and thermal gradients for many high-temperature applications.

In making a choice, it is necessary to know what materials are available and to what extent they are suited to the specific application. The decision is quite involved and the choice is significantly affected by the environment and the intended use, be it a reactor vessel, tubes, supports, shields, springs, or others. Some problems may occur because of distortion and cracking caused by thermal expansion/contraction; typically, a high-temperature alloy might change ¼ in./ft from ambient to 1,000°C (1,832°F).

The user or designer needs to properly understand that the environment dictates the materials selection process at all stages of the process or application. For example, an alloy that performs well at the service temperature may corrode because of aqueous (dew point) corrosion at lower temperatures during off-load periods, or through some lack of design detail or poor maintenance procedures that introduce local air draughts that cool the system (e.g., at access doors, inspection ports, etc.).

To provide as optimum performance as possible, it is necessary for a supplier to be aware of the application, and for the user to be aware of the general range of available materials. Otherwise, severe problems can result. For example, a catastrophic failure occurred within weeks for an ignitor, made with Type 304 stainless steel (UNS S30400, iron, 19% Cr, 9% Ni, 0.08% C). Type 304 stainless steel would have been suitable for clean oxidizing condi-

Materials

tions to about 900°C (1,650°F) in continuous service, or 845°C (1,550°F) in intermittent (temperature cycling) service (1). The failure occurred because of overheating with contributions from sulfidation (hot corrosion). The true cause of failure was a material mix-up, because Type 304 was not specified, but was inadvertently used.

Mechanical limits of materials

In considering traditional alloys, it is important for the designer and user to be fully aware of the mechanical limits of a material. For example, the ASME Pressure Vessel Codes advise that the maximum allowable stress shall not exceed whichever is the lowest of: (i) 100% of the average stress to produce a creep rate of 0.01% in 1,000 h; (ii) 67% of the average stress to cause rupture after 100,000 h; and (iii) 80% of the minimum stress to cause rupture after 100,000 h.

These recommendations may be better appreciated by extracting typical data for Type 304 intended for use in a pressure vessel up to 815°C (1,500°F). Based upon ASME tables, for a load of 17 MPa (2.5 ksi) at 760°C (1,400°F), the expected design life would be 24 yr; at 788°C (1,450°F), the life falls to 7 yr; and at 815°C (1,500°F), it is only 2.2 yr. Thus, a short-term temperature excursion can have a significant effect on equipment life. Also to be noted is that a small increase in loading, for example, from 2.5 to 3 ksi at 760°C (1,400°F), can markedly reduce the life expectancy, here, from 24 to 9 yr.

Overheating is the most common cause of high-temperature corrosion failure, but the temperature influence on mechanical properties is of equal or even more significance in that many failures occur because of creep deformation (creep voids) and thermal fatigue. Overheating can arise for various reasons, including an unexpected accumulation of tenacious deposits that can foul tubes in a heat exchanger.

High-temperature alloys and uses

High-temperature alloys are typically iron-, nickel- or cobalt-based alloys containing >20% chromium (or 30% for cobalt), which is sufficient to form a protective oxide against further oxidation. The basic alloys include various additional elements that aid in corrosion resistance, notably aluminum (typically >4% to develop an alumina scale), silicon (up to 5% to develop an amorphous (glass-like) scale that is complementary to chromia), and rare earth elements (typically <1%, e.g., yttrium, cerium, and lanthanum, that improve scale adhesion). Other additions, such as the reactive metals, the refractory metals, and carbon, primarily improve mechanical properties. The beneficial and detrimental roles of common alloying elements on the anticipated performance of alloys at high-temperatures is covered by Agarwal and Brill (2).

Refractory metals — Molybdenum, which is a beneficial addition for resisting aqueous chloride-induced pitting corrosion (found in Types 316 and 317 stainless steels, and the 6%-Mo alloys), is prone to catastrophic oxidation as temperatures exceed about 700°C (1,292°F), the point above which MoO₃ forms eutectic mixtures with iron, nickel, and chromium oxides. The oxide MoO₃ melts at 795°C (1,462°F°).

Catastrophic oxidation rapidly renders a metal into a useless powdery oxide. Damage is worse in stagnant conditions and appears to be exacerbated when sodium oxide is present (e.g., from insulation). All of the refractory metals (tungsten, tantalum, niobium, and molybdenum) may experience catastrophic oxidation. Silicide coatings have shown some to offer some resistance to this catastrophic ("pest") oxidation.

Coatings — High-temperature coatings or surface modifications are generally based on chromium, aluminum, or silicon, which, at high temperatures, form protective oxides

rich in chromia, alumina, or silica, respectively. In more recent years, there have been developments in applying so-called alloy coatings, for example, the use of MCRALY (metal, chromium, aluminum, and yttrium) on steels or other high-temperature alloy substrates. Efforts have also continued in weld overlay work, where a strong base metal can support a corrosion-resistant surface-coated layer.

Applications — In considering materials options, a thorough knowledge of the service applications (stress-bearing service; cyclic loading or not; frequency of cycling; impact or erosion effects; thermal expansion and contraction) is needed.

Different high-temperature corrosion processes are simultaneously involved in many common service applications. Some of these are synergistic, which creates a formidable challenge for users and alloy producers. Some examples of the forms associated with various applications are given in Table 1 (3, 4).

Types of high-temperature corrosion

There are certain distinguishing features about the morphology of high-temperature corrosion that aid in deciding upon the cause of damage. Some typical indications include thick scales, grossly thinned metal, burnt (blackened) or charred surfaces, molten phases, deposits of various colors, distortion and cracking, and magnetism in what was first a non-magnetic (e.g., austenitic) matrix.

Damage varies significantly based upon the environment, and will be most severe when a material's oxidation limits are exceeded, notably when an alloy sustains breakaway attack by oxygen/sulfur, halogen/oxygen, low-melting fluxing salts, molten glasses, or molten metals, especially after fires.

Oxidation

Many industrial processes involve oxidation, i.e., a metal reacts in air to form and sustain a protective oxide.

Table 1. Typical process conditions causing corrosion.

Process or Components	Temperature	Type(s) of corrosion							
		O	S	C	Cl	F	N	Slag	Melt
Steam reforming tubes	To 1,000°C	•		•					
Steam cracking tubes: ethylene	To 1,000°C	•		•					
Vinyl chloride crackers	650°C				•				
Hydrocrackers: heaters	550–600°C		•	•					
Coke calcining recuperators	815°C	•	•	•					
Cat cracking regenerators	To 800°C	•							
Flare-stack tips	950–1,080°C	•	•		•				Cl ₂ ; marine corr.
CS ₂ furnace tubes	850°C		•	•					Deposits
Melamine/urea reactors	450–500°C						•		
Reactors in Ti production	900°C	•			•				
Nitric acid: catalyst grids	930°C	•	•				•		
Linings for Al pyrohydrolysis	To 1,000°C	•				•	•		
Nuclear processing reactors	750–800°C	•				•			
HTGR* (gas-cooled) reactors	750–950°C	•							
Oil-fired boilers/superheaters	850–900°C	•	•					•	•
Gas-turbine blades	950+°C	•	•		•		•	•	•
Waste incinerators	470–500°C	•	•		•	•		•	•
Fluidized-bed combustors	>600°C	•	•	•	•			•	•
Glass: recuperators	1,090°C	•	•		•				•
Hot-dip galvanizing	455°C								•

* HTGR is high-temperature gas reactor.

There can be several oxide products, some of which are less desirable, for example, wustite, a defective oxide of iron that forms rapidly at about 540°C (1,000°F) on steel.

Most high-temperature alloys are oxidation resistant, so price, availability, experience, and the type of application usually dictate choice. There are no significant problems up to 400°C (750°F), few up to 750°C (1,380°F), but the choice of successful alloys becomes somewhat limited above about 800°C (1,470°F).

Simple iron-chromium (or iron-chromium-molybdenum) alloys become less useful as service temperatures increase, which is where the Type 300 series austenitic stainless steels, (304, 309, 310, 314, 330, 333, etc.) and certain ferritic stainless steels (410 and 446) find many applications. For more arduous service conditions at higher temperatures, these alloys are surpassed by nickel- or cobalt-based formulations, including many of the more robust alloys that are mechanically alloyed to im-

prove strength and to control (that is, minimize) grain growth at elevated temperatures.

Certain alloys (usually those with rare earth additions) are more resilient to oxidation under thermal cycling (shock) conditions. Some applications do not allow an alloy to fully develop its steady-state condition, thus, performance is dictated by the transient (not-so-protective) surface scales. Transient effects will become apparent should failure analysis be performed.

Caution should be given to iron-chromium-nickel alloys that can be prone to sigma-phase formation between 540–800°C (1,000–1,470°F), which results in premature brittle failure. Molybdenum-containing alloys (Types 316 and 317 stainless steels and the 6%-Mo alloys) can be prone to catastrophic oxidation above about 680°C (1,256°F).

Sulfidation

Sulfurous gases are common to many applications, including fuel

combustion atmospheres, petrochemical processing, gas turbines, and coal gasification. Sulfides (e.g., sulfur vapor, hydrogen sulfide) can be very damaging, because metal sulfides form at faster rates than do metal oxides. Sulfides have low melting points and produce voluminous scales (scale spallation).

With mixed corrodant environments (oxygen and sulfur), alloy performance is based upon a subtle interplay between oxide and sulfide formation. Oxides are more stable; sulfides form more rapidly (due to kinetics). Thus, oxides, sulfides, or both may form. If deposits are also present, then conditions at the metal surfaces are reducing compared to areas external to the deposits. Damage can be extensive.

Mixed sulfur-and-oxygen gases can invoke very high corrosion rates due to breakaway attack, typically above about 600°C (1,110°F) for nickel-based alloys, 920°C (1,688°F) for cobalt-based, and 940°C (1,724°F) for iron-based formulations. Break-



Above: Tube failure due to local overheating.

Left: Burst tube walls due to overheating.



away attack is commonly associated with sulfur and excess air. Once the first-formed oxide is lost or destroyed, sulfides can invade the chromium-depleted substrate, thus, causing accelerated attack to occur.

Stainless steels and iron-based alloys are preferred over high-nickel alloys, because nickel is prone to forming the low-melting nickel-nickel sulfide eutectic, $\text{Ni-Ni}_3\text{S}_2$, which melts at 635°C ($1,175^\circ\text{F}$). Eutectics of cobalt and iron occur at higher temperatures, 880°C ($1,616^\circ\text{F}$) and 985°C ($1,805^\circ\text{F}$), respectively.

Alloys can be weakened by internal corrosion, most noticeably when mobile species are present, such as low-melting sulfides, which are typified by localized dull uniform gray phases within the alloy matrix. At times, liquid-appearing phases are found in the metallurgy.

Alloys containing aluminum, silicon, and cobalt are useful in sulfidizing environments. Many alloys classified as candidates for sulfidation do well only if oxides are first able to form. Preoxidation can be of value.

Halogenation

Halogen attack is commonly manifested as a combination of scale spallation with internal alloy damage including voids that form as a result of highly volatile species (5). Material

performance is dictated by the unique properties of the halides, including high vapor pressures, high volatility (vaporization), low melting points, mismatched expansion coefficients with metal substrates, and the effects of displacement reactions whereby oxide or sulfide are thermodynamically favored over the halides.

Alloy performance is greatly affected by oxidizing or reducing conditions. For oxidizing atmospheres or for vapors jointly present with oxygen (or air), there is an opportunity for reduced corrosion rates (kinetics) associated with oxide formation, although the scale may later be disrupted by the volatile halides, especially if iron-based alloys are used. Nickel alloys are generally favored for halogen atmospheres, since iron-based alloys are more vulnerable, due to their volatile products, *e.g.*, FeCl_3 . Silicon additions are useful if oxidizing environments prevail, but not for reducing conditions. Preoxidation is not normally a benefit for reducing halogen attack.

What makes halogens different from other oxidants is their high mobility and diffusivity into a metal, resulting in internal damage of the alloy matrix. Fluorine can penetrate twice the distance of chlorides, which means that the predominant mode of damage in fluorine-contain-

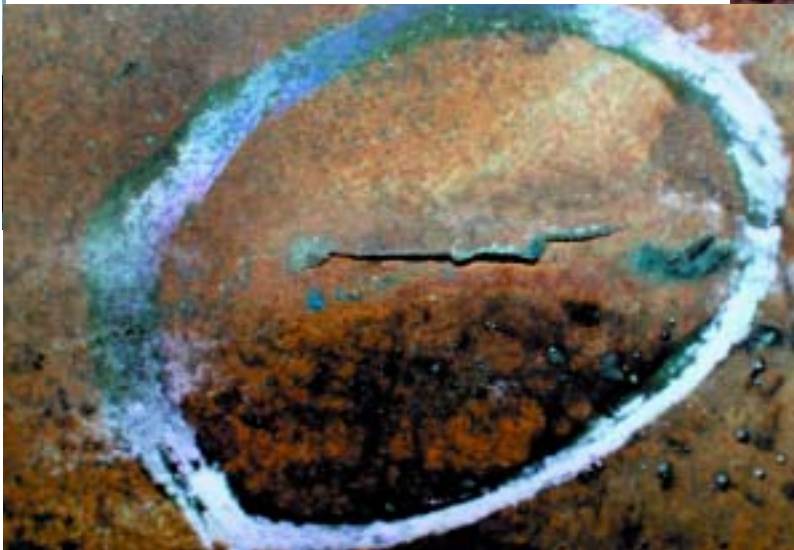
ing gases is by means of internal attack.

Halide products are also hygroscopic (3), so it is not unusual to discover local protrusions on a metal that have been removed during service. In laboratory studies, it is common to find that a surface apparently free from chlorides (removed during metallographic preparation) is later found to show them. This is because the chlorides have been leached out from deep under the voided areas in the metal.

Carburization

Several environments are synonymous with carburization, including pyrolysis and gas-cracking processes, reforming plants, and heat-treating facilities that involve carbon monoxide, methane, and hydrocarbon gases. Damage is usually manifested as internal carbides, notably in grain boundaries and is generally worst above $1,050^\circ\text{C}$ ($1,922^\circ\text{F}$). When carburizing conditions alternate with oxidizing ones, carbides can become oxidized to oxides, which yields carbon monoxide that can weaken the grain boundaries in an alloy. Such an alloy fails by "green rot," a name that describes the green fractured surface that results (chromium oxide).

Strongly carburizing atmospheres (*i.e.*, those that have a carbon activity >1) can cause a metal to form coke-like layers, often of a dusty form. This form of attack, termed metal dusting, commonly occurs between $425\text{--}800^\circ\text{C}$ ($790\text{--}1,470^\circ\text{F}$) and can be very rapid (in days not months). Damage is either general or localized (pitting), as dictated by the ability of the alloy to form a surface oxide (6).



Above: Thermal fatigue crack in boiler tube.



Top: Tube fouling in an incinerator plant due to carryover of deposits.



Bottom: Through-metal perforation in tubing from a carbon black plant.

Carbon steels and alloy steels are normally uniformly thinned by metal dusting; more highly alloyed materials usually display local outgrowths of coke emerging through small pits that broaden with time.

Cast iron-nickel-chromium alloys are widely used for carburizing applications, including the more recent alloys containing 1–2% silicon and 1.5% niobium (the HP Mod alloys) (4, 6). High-nickel alloys (with low solubility for carbon) find many applications for carburizing conditions. Stronger nickel-based alloys with high chromium and silicon contents are useful in more demanding environments. Highly alloyed ferritic stainless steels (that are able to more rapidly form a thin oxide film) tend to outperform austenitic steels.

Nitriding

Relatively little is reported about nitridation other than material performance is weakened (embrittlement) as a result of the formation of internal nitrides in the alloy (4). It is common to expect damage with nitrides at 700–900°C (1,290–1,650°F). Nitrides appear generally as needle-like precipitates in the alloy matrix.

Nickel- and cobalt-rich alloys appear to be first-choice candidates for resisting nitride attack, because of the low solubility of nitrogen in these

base metals. Iron tends to be detrimental, as do aluminum and titanium in low concentrations. Silicon forms a brittle intermetallic compound with nitrogen and can contribute to scale spallation, especially in applications at low oxygen concentrations (potentials), where thin oxides can form, and during thermal cycling.

Molten products

Deposits are a common product in many high-temperature applications, including boilers, waste incinerators, fluidized-bed combustors, and gas turbines. A whole series of reactions is possible should deposits become molten and no single mechanism can be applied generally to characterize such damage (7).

The mechanisms of molten product corrosion are complex. The types of damage include fuel-ash corrosion — sulfates, including acid and basic fluxing reactions (8), and vanadic slag attack (9) — molten salt corrosion (chlorides, nitrates, and carbonates) (4), and molten glass corrosion. Liquid metal attack is yet another special category (4).

Fuel-ash or ash/salt-deposit cor-

rosion stems from high-temperature corrosion processes associated with fuel combustion products in boilers, waste incinerators, and gas turbines. Thus, products can include various deposits (oxidizing or reducing) with active contributions from oxygen, sulfur, halogens, carbon, and nitrogen (4, 7). Typically, alloy matrices display intergranular attack (oxides and chlorides) beneath disturbed oxide layers possibly fused with molten deposits and internal sulfides within the alloy-affected zone.

Hot corrosion is generally regarded as attack in the joint presence of sulfur and oxygen. Typically, attack is considered to be triggered by molten alkali metal salts that melt above 700°C (1,290°F). Sodium sulfate, with a melting point of 884°C (1,620°F), derived from sodium chloride and sulfur from the fuel, is considered to be closely involved in the mechanism of hot corrosion (8). This mechanism is considered to have four stages: oxidation (incubation); mild sulfidation; oxide failure; and catastrophic attack (internal sulfides via a porous voluminous complex oxide/deposit layer). Hot corrosion is an irreversible autocatalytic process.

Table 2. Guide to candidate materials.

Corrosion Mode	Basic Alloy Types	Candidates*	Notes and Cautions
Oxidation	Fe-Ni-(Co) >20% (30%) Cr. Stabilized to minimize sensitization. Al, Si beneficial. Rare earth additions aid scale retention.	304, 321, 309, 310, 800(HT), 803, 430, 446, HR120, 330, 85H, 333, 600, 601(GC), 602CA, 617, 625, 253MA, 353MA, DS, 214, MA956, MA754, X, etc.	Wide choice dictated by application and function; Mechanical properties; Thermal cycle (shock); Transient vs. steady state; Internal oxides. Beware σ ; W, Mo — catastrophic oxidation.
Sulfidation (Reducing gases no oxides)	Fe- with high Cr (Al) alloys.	9–12%Cr steels: 309, 310, 330, 800(HT), 803, HR120, 85H, 253MA, 353MA, MA956, 446, 671, 6B, 188, etc.	Sulfur vapor, H_2S , etc. — no oxides. Beware of Ni/ Ni_3S_2 eutectic; coatings can help.
Sulfidation (Oxidizing gases)	Fe-Cr-based alloys. Oxide formation a benefit. Preoxidization may help.	As above with 153MA, 601, HR160, MA754, MA956, 333, 556, etc.	SO_2 , SO_3 , etc. — risk of breakaway attack with oxides and sulfides; Al coatings. (See hot corrosion.)
Carburization	Wide use of cast alloys. For worse conditions, use high-Ni alloys with Cr, Si. (Low solubility of C in Ni is benefit for Ni alloys.)	HH, HK, HPMo, 309, 310, 330, 333, 85H, 800(HT), 803, DS, HR160, 600, 601, 253MA, 602CA, 617, 625, 690, MA754, MA956, X, 556, 706, 718, 750, etc.	Internal carbides with intergranular attack; Cast tubes benefit from smooth I.D. surfaces. Metal dusting (at lower temperatures). Green rot (with intermittent O_2 -C).
Nitridation	Ni-alloys rather than Fe. Avoid high Cr levels. Use low Al and low Ti levels (nitride formers). Si promotes scale spalling.	309, 800(HT), 330, 446, 188, 230, 600, 602CA, 625, 253MA, etc.	Internal nitrides (e.g., AlN) can weaken alloy; Thin oxide at low oxygen partial pressure reduces nitridation.
Halogenation: chlorination, fluorination, etc.	Ni alloys generally better than Fe. Benefits: Cr (not HF), Al, Si (with oxygen). Preoxidation not beneficial.	800H, 333, 200, 201, 207, 600, 601, 602CA, 214, N, H242, B3, etc.	Volatile products; Internal attack with voids; Hygroscopic products (e.g., chlorides); Scale spallation.
Fuel ash corrosion	FeCrMo alloys at lower temperatures; CRAs† for S, O, C — subject to application. High Cr, Al, Si useful (also as coatings).	309, 310, 800(HT), 600, 601, 602CA, 625, 825, 253MA, 353MA, MA754, MA758, MA956, IN657, 671, etc.	Applications dictate alloy, or coating: Gas turbines (strong + CRA†); vanadic slag (high-Cr + Si).
Molten salts	Ni alloys generally favored; some high-Cr alloys; NiCrMoW alloys for molten chlorides.	As with halogens, sulfidation: depends on nature of salts (acidic/basic).	Intergranular attack, internal voids, and probable embrittlement.
Molten glass	Ni- or some Co-/high-Cr alloys; Some refractories.	600, 601, 602CA, 671, 690, MA758, etc.	Complex fluxing reactions; oxidation; sulfidation; chlorination; fluorination, etc.
Liquid metals	Fe-alloys with Cr, Al, Si usual first choices (subject to liquid metal, e.g., Liq. Na, K, molten Zn, Pb, etc.)	309, 310, 85H, 253MA, etc.	Dissolution or alloying effects; Intergranular attack; Depends on system.
Complex Environments	Synergy of processes.	CRAs† or coatings	Seek input from suppliers; Consider online tests/monitoring.

Note: This is a general, not exhaustive, guide.

* Not in any preferred order.

† CRAs are corrosion-resistant alloys.

Vanadic slag corrosion occurs following combustion of certain low-grade or residual fuel oils that are of high vanadium, sulfur, and alkali metals. The molten sodium vanadyl vanadates typically flux away protective oxides and then rapidly dissolve the metal. Many high-temperature alloys cannot survive 100 h at 900°C (1,652°F) in vanadic slags (9). Vanadic attack can be managed by lowering temperatures (if possible), using fuel-oil additives (such as magnesium and calcium oxides), or by specifying high-chromium alloys. Silicon-rich coatings are beneficial and appear to complement the role of chromium (9).

Molten glass typically induces intergranular attack with voids (from volatile halides) and sulfides. Oxides are generally fused into the glass. Attack is commonly rapid, and high chromium-nickel-based alloys are usually employed. Iron-rich alloys can be prone to severe attack due to their ability to form low-melting halides (e.g., FeCl₃).

Molten salts, used for heat treating applications, nuclear engineering, solar cells, and metal extraction, generally promote intergranular attack in alloys, often with voids and internal low-melting products (halides).

A common feature in most high-temperature aggressive environments is the synergy of the reactants one with each other (Table 1). Wastage can be easily measured, but the mechanism(s) are not as easy to determine. As a minimum, a rigorous study and analysis of the morphologies should help to establish the rate-controlling process, which should help to better define the type of alloy that could be considered as a candidate. The broad expertise of the material suppliers should be fully explored in the quest for a suitable choice. Monitoring trials using test spools are recommended wherever they can be used.

Candidate alloys

Choice should be based on careful considerations, including, primarily,

the function of the component or vessel. As might be expected, there are many candidates, yet, from these the choice is often reduced to one of two, once the total range of properties is fully explored. Factors to be considered include mechanical properties (strength, flexibility, fatigue life), physical properties (expansion and contraction, reflectivity, magnetism, etc.), availability (shape and form), and price (economic decision based on overall costs and fabrication, etc.). As a convenience, some generally

recommended alloys and alloy types for various high-temperature environments are summarized in Table 2. This table is intended only as a guide; no order of merit is to be interpreted from the sequence of listings (or omissions) in this table. Also, the alloy lists are not meant to be inclusive, but, rather, merely typical examples of what has worked in the field.

To sum up

Ideally, the material choice is based on known data and experience, which implies communication between a user and a supplier. A better knowledge of anticipated component requirements in addition to corrosion behavior provides for a better choice and the expectation of more reliable service.

Proper identification and recording of damage from prior systems is a positive benefit in deciding upon an alternative alloy or coated system. Wherever possible, and certainly for new and complex environments, testing is to be recommended. ♦

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