

Neutral salt pot alloy life: maintenance is the key

Based on several failures in the field, the author observes that some heat treaters may not realize that operating practice and maintenance are critical to the life of alloys used in neutral salt environments.

BY James Kelly

Maintenance is key to satisfactory performance of alloy heat treating equipment. As heat-resistant alloy specialists, Rolled Alloys is accustomed to emphasizing the role of alloy selection, but with metallic salt pots we have found that operation and

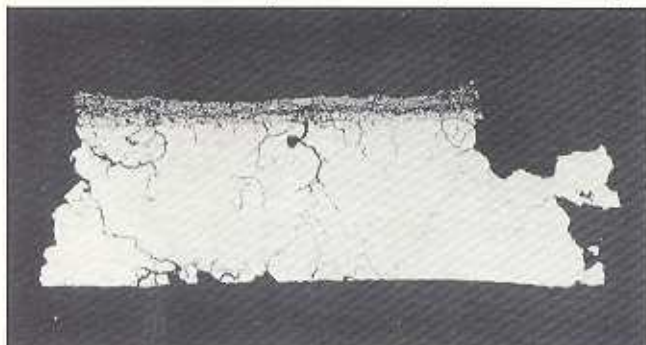


Fig. 1: 1/4 inch RA330 from salt pot. Inside surface is dark band at top of photo. 4X

maintenance are much more important than alloy selection. Neutral salt pot operating procedures literally determine whether life expectancy is two days or a year.

In other words, a heat treater may find that a change of alloy can improve upon already good life; but there is no alloy solution to the problem of erratic life measured in days or weeks. If salt pot life is a serious problem, the user must look first to his operating procedures, or, when fixtures are concerned, to fabrication practice.

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The following points are important for good alloy life in a salt heat treat environment:

- 1) Ensure that there is no salt whatsoever in the combustion chamber of a gas-fired pot or about the elements of an electrically-heated pot. This is crucial.
- 2) Rectify and desludge neutral chloride salts at least daily.
- 3) In both pots and fixtures, all welded joints must be full penetration welds.
- 4) Idle the pot with salt still molten, rather than shutting down completely and letting the salt freeze solid.
- 5) Do not put oily work or any foreign matter into the pot.
- 6) Only after

these five points are addressed should alloy selection be reviewed.

Let us examine these points in detail:

Mixtures of potassium, sodium and barium chlorides are widely used as heating media that neither oxidize nor decarburize steel. Regarded as "neutral" salts, they are, in practice, oxidizing to the Ni-Cr-Fe alloys often used for pots and fixtures.

When these salts are heated, the vapors mixed with air can destroy an alloy pot in short order.^{1,2} A common scenario is to get perhaps a year's pot life in a new furnace, then have the replacement pot fail

in two days. Some of these failures can be quite spectacular. In one shop we saw an RA253MA pot turn bright green. Nominal operating temperature was 1850°F, the upper limit for metallic pots. Later, an RA330 pot in the same furnace formed a loose "shroud" of oxide all over the outside, after 33 days. Chromium, originally 18-20%, had been depleted to no more than

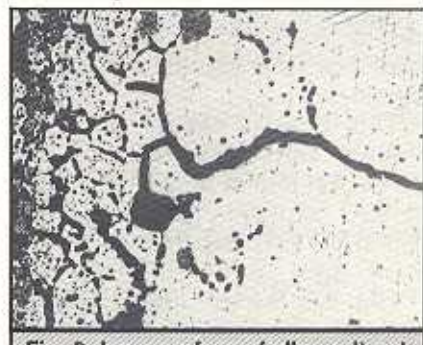


Fig. 2: Inner surface of alloy salt pot showing typical porous surface and deep intergranular attack. 100X

4.5% in the alloy. Most of the missing chromium was in the salt, judging from its green color.

Another supplier then had the misfortune to have a pot of its advanced nickel alloy tried in this furnace. It lasted only a few days, a casualty of excessive scaling on the outside surface and attack on the inside from the molten salts.³ At Rolled Alloys, we use this same Ni-Cr-Al-Y alloy for oxidation test racks. We have found no significant scaling in air even at 2200°F.

These exasperating failures occur when a pot leaks and is replaced without *all* the old salt being cleaned out of the furnace brick

work. Chloride vapors and air form loose, porous scales that are completely non-protective.⁴ It makes no practical difference whether the chlorides are mixed with products of combustion in a gas-fired unit or quietly vaporized by electrical heating elements.

Fig. 1 is the cross-section of one such expensive failure. This ¼-inch-thick RA330 salt pot leaked after six weeks at 1500°-1525°F, holding neutral salt desludged twice daily. The previous pot, also RA330 from the same fabricator, had been installed when the furnace was new. It lasted 14 months. When it failed, however, all of the leaked salt was not cleaned out of the gas-fired furnace before the new pot was installed. This oversight cut life by 90%.

We cannot emphasize enough that failure to clean out all spilled salt from the previous pot is the direct cause of life as short as two days with the next pot.

Salt leaking between the pot flange and furnace top also gets down into the combustion chamber; the results are just as troublesome.

This type of failure has been known and described since the 1930s. Yet Rolled Alloys has 55 salt-related failures in its Investigation File from 1957 through 1983, with more since.

Hot salt vapors attack metallic pots, over-the-top electrodes and fixturing at the salt-air interface. For the most part, there is little to be done about this except to replace the corroded parts. Fixturing is one area in which alloy selection can directly affect life. However, there seems to be no general agreement regarding the most cost-effective grade, and everything from mild steel to alloy 600 is used.

Rectifying the salt

In the course of normal operation, molten salt baths continuously pick up oxygen from the atmosphere and from contaminants carried into the bath with the work. The oxygen is present as oxides of barium, sodium, potassium and iron, as well as various chromates. To prevent the steel workpieces

from decarburizing, the salt must be rectified. That is, the oxygen content of the bath must be reduced to low levels. Usually this is done by introducing methyl chloride (well away from electrodes and metallic pot sidewalls), which converts the metal oxides back to chlorides. Solid rectifiers such as powdered silicon, silica, ferrosilicon or dicyandiamide are also used. The inorganic rectifiers form a metallic sludge in the bottom of the bath.

The normal failure mode of an alloy pot is by salt permeating the metal until it begins to leak through to the outside. This occurs because even salt neutral to steel is selectively oxidizing to chromium, with little effect on the alloy's iron and nickel content. As fast as chromium oxide forms it is stripped away by the chlorides, forming alkali and alkaline earth chromates. More chromium diffuses along grain boundaries to replace that lost to the salt. Eventually, diffusion voids

BaO to double or triple the corrosion rate of commonly used heat resistant alloys.⁵

We urge users of salt baths to contact salt suppliers for advice on how best to rectify their particular salt. The various methods of rectification affect alloy life in different ways. It would be wise to solicit opinions from the technical representatives of two or three different salt manufacturers.

Sludge accumulates in the bottom of salt baths due to various contaminants, and as a normal product of inorganic rectifiers. It should be removed frequently, perhaps twice daily; otherwise it may cause overheating of metallic pots and shorten electrode life. Alloy Casting Institute studies of salt baths show that sludge deposits increase corrosion rates by 25% to 100%.⁶

Welded joints

All welded joints must be full penetration welds. Lack of weld penetration is perhaps the single

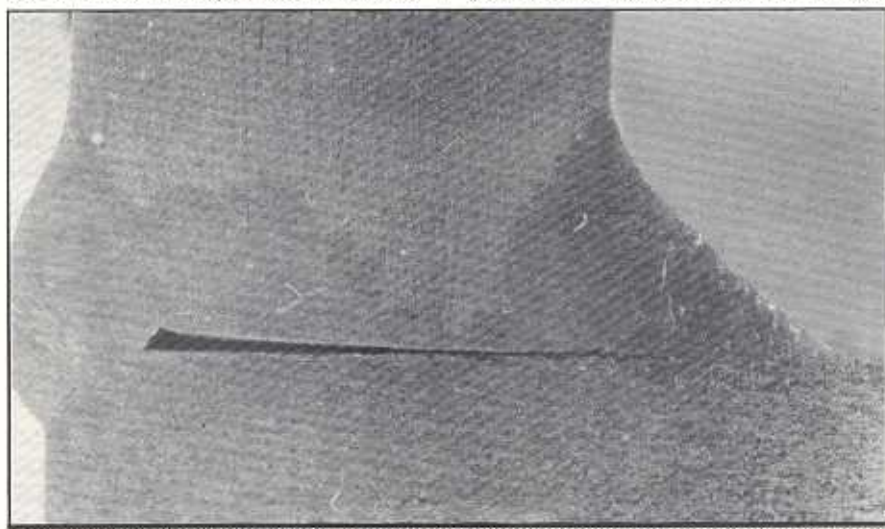


Fig. 3: Lack of weld penetration invites high-temperature failure. 6X

form in the grain boundaries, link up and become a path along which molten salt itself can penetrate the metal. A typical example is shown in Fig. 2.

While this represents the "normal" failure mode, the rate of intergranular attack is strongly dependent upon oxygen content, hence the thoroughness of rectification, of the salt bath. Laboratory tests in a 15% NaCl-bal BaCl₂ salt at 1800°F show the addition of 3%

most important cause of weld failures in high-temperature service. Under thermal or mechanical cycling, the unwelded area acts as a crack, which eventually grows through the weld bead from the inside. Molten salt exacerbates the situation by seeping into the voids and, literally, prying the joint apart.

Fig. 3 shows exactly what not to do, although this particular joint failed in a lead pot, not salt. Weld beads penetrated by salt are shown

in Fig. 4, after 200 cycles at 1650°F, marquenching at 350°F.

Our next point is: Do not let a pot full of salt freeze solid on shut-down. Either idle the pot at a temperature at which the salt is still molten, or ladle out the salt before it freezes. When this solidified salt is again remelted on start-up it goes through a volume increase—about 3/8 to 1/2 inch per foot of pot depth. If the pot is full of solid salt, there are two unpleasant possibilities.

One is that as the salt melts first on the bottom, it expands and cracks open the pot, either in a weld or along the knuckle radius of a dished head. If this doesn't occur, once sufficient salt has melted, the volume expansion may cause it to explode through the remaining frozen layer on top.

Next: Put only clean work into the pot. If machined parts are to be heat treated, wash off the sulfur-bearing machining oil and then rinse off the alkalis from the wash. Otherwise the sulfur will simply attack the nickel alloy pot, the attack being more rapid with the higher

nickel alloys. One of our fabricator customers related an incident in which short life of 309 pots was traced to the practice of disposing of floor sweepings in the heat treat pots at night.

Alloy selection

Finally comes alloy selection. Over the last half century or so, the most popular alloys have been the cast grades HT (17Cr 35Ni) and HW (12Cr 60Ni) or the wrought alloys

	Cr	Ni	Si	C	Other	Fe
CAST						
HT	17	35	1.7	.5	—	44
HW	12	60	1.5	.5	—	24
WROUGHT						
446	25	—	.5	.1	—	73
RA253MA	21	11	1.7	.08	.17N .04Ce	65
RA85H	18.5	14.5	3.5	.2	1 Al	61
309	23	13	.8	.05	—	62
RA330	19	35	1.2	.05	—	43
600	15.5	76	.2	.08	—	8
200	—	99.5	.2	.05	—	.1

Table I: Alloys used in pots, fixtures and electrodes

309, RA330 and 600. (See Table I.) Laboratory work and observations of fixtures are reasonably consistent in showing that the higher nickel grades have better resistance to chloride salts. If that were all there were to it, all metallic salt pots would be HW or 600, and all electrodes in ceramic pots, commercially pure nickel. In fact, the bulk of metallic pots for neutral salts today

are 309 or 330, with 600 in the minority. Almost all heating electrodes are 446, with a very few 600 or 330 users. Almost none are pure nickel. It appears that the various ills that befall metallic pots obscure the benefits of increased nickel to the extent that 13% or 35% nickel grades are considered more cost-effective.

With respect to fixtures, performance often simply follows resistance to chloride salts. Either

Alloy	Intergranular attack, mils
RA253MA	6.9
309	12.5
RA330	13.8
600	7.5
RA85H	4.4

Condition: 210 to 252 cycles in preheat salts 1300°F and 1500°F, high heat salt 2200°F, quench in 1100°F salt, air cool

Table II: Evaluation of baskets used in salt

600 or RA330 is, in our opinion, superior to 309. In some shops, 600 has the advantage over RA330; in others, there is no clear difference. In all cases, full penetration welds are necessary.

In three or four performance trials and one coupon test, it appears that RA85H may perform as well as or better than alloy 600 in neutral chloride salts. Table II is the result of coupon tests on 1/4 inch or 3/8 inch plate samples to 2200°F. These results are encouraging enough that we feel RA85H may be worth evaluating for alloy fixturing, when welded appropriately with matching filler metal. However, we caution users not to place too much faith in anyone's coupon tests. Only actual service trials with your fixturing in your own plant will tell which alloy gives the best service life per fabricated dollar. **HT**

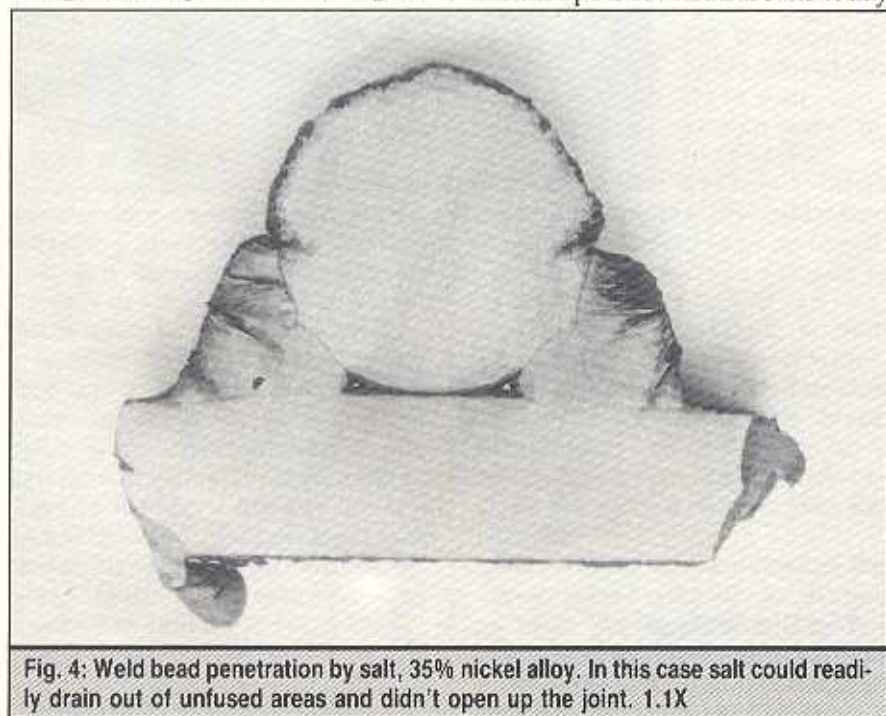


Fig. 4: Weld bead penetration by salt, 35% nickel alloy. In this case salt could readily drain out of unfused areas and didn't open up the joint. 1.1X

REFERENCES

- 1) H. Klouman, Heat Treating Equipment, *The Book of Stainless Steels*, ASM 1935.
- 2) R. C. Stewart, "Characteristics and Uses of Salt Baths," *The Iron Age*, Feb. 14, 1946.
- 3) D. F. Fluck, R. B. Herchenroeder, G. Y. Lai and M. F. Rothman, *Metals Progress*, September 1985, p. 40.
- 4) Peter Elliott, "Practical Guide To High-Temperature Alloys," *Materials Performance*, Vol. 28, No. 4, 1989.
- 5) Jon R. Bryant and Donald E. Wenschol Jr., Improving Salt Bath Equipment Life, *Metals Progress*, February 1979.
- 6) J. H. Jackson, Cast Alloys For Salt-Bath Heat Treating, *Alloy Casting Bulletin*, No. 16, The Alloy Casting Institute, November 1952.