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Evaluation of Alternate Construction Materials for a Lithium Electrolysis Cell

J. R. Keiser
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Printed in the United States of America. Available from
National Technical Information Service
U.S. Department of Commerce
5285 Port Royal Road, Springfield, Virginia 22161
NTIS price codes—Printed Copy: A03 Microfiche A01

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Metals and Ceramics Division

EVALUATION OF ALTERNATE CONSTRUCTION MATERIALS FOR A LITHIUM ELECTROLYSIS CELL

J. R. Keiser, A. R. Olsen, and J. R. DiStefano

Date Published - May 1988

Prepared by the
OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee 37831
operated by
MARTIN MARIETTA ENERGY SYSTEMS, INC.
for the
U.S. DEPARTMENT OF ENERGY
under contract DE-AC05-84OR21400

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EVALUATION OF ALTERNATE CONSTRUCTION MATERIALS FOR A LITHIUM ELECTROLYSIS CELL*

J. R. Keiser, A. R. Olsen and J. R. DiStefano

ABSTRACT

Electrolytic cells currently used for the production of lithium metal are constructed of A285 Grade A steel (carbon content less than 0.15%). Failures of the cell wall have frequently occurred after 2 to 5 years of service, and these failures have been attributed to corrosion resulting from the reaction of iron carbide with lithium. An examination of a recent failure confirmed this conclusion.

In an effort to identify alternate construction materials with better corrosion resistance for this application, five materials with very low carbon levels and/or carbides more stable than those in A285 were selected for evaluation. Materials were evaluated after exposure in an operating electrolytic cell, after exposure to pure lithium, and after exposure to a moist air-chlorine mixture. Results show that all five of the alternative materials selected are more corrosion resistant than A285, but two were eliminated from further consideration because of other potential shortcomings. Additional long-term corrosion tests are recommended for the remaining three candidate materials. Final selection should be based on considerations of cost, availability, and fabricability in addition to compatibility.

INTRODUCTION

Lithium metal is produced at the Oak Ridge Y-12 Plant using electrolytic cells that operate at a nominal temperature of 440°C with a lithium chloride/potassium chloride eutectic mixture. The cell containment vessel

*Based on work performed at Oak Ridge National Laboratory and Oak Ridge Y-12 Plant, operated for the U.S. Department of Energy under contract DE-AC05-84OR21400 with Martin Marietta Energy Systems, Inc.

contacts a molten-salt mixture, the electrolytically produced lithium metal that floats to the surface of the salt, and a chlorine-air mixture that is produced when moist room air is drawn through the cell to help flush the chlorine gas produced in the process. The material specified for the cell containment vessel is American Iron and Steel Institute A285 Grade A steel. On some occasions, higher carbon grades of A285 have been used, but their actual carbon contents have been in the Grade A range.

The A285 cells have typically developed leaks after about 3 years of continuous service, although failures have been reported in shorter times.[1] A previous analysis of a failed section attributed the failures to reaction of lithium with relatively low stability carbides in the A285 to form lithium carbide that dissolves in the lithium.[1] A Russian study of corrosion of a 0.1% carbon steel by lithium gave similar results, and the investigators proposed that volume changes associated with the corrosion reactions could cause plastic deformation in the corrosion zone.[2] Based on these conclusions, steel plate and welding rod with no more than 100 to 150 ppm carbon were recommended for future cell material. An alternate recommendation was to use A285 Grade A steel in the spherodized condition to put the iron carbides in a geometric arrangement that might be less accessible to tunneling attack by the lithium.

To investigate the problem, we initially performed an analysis of a section of A285 steel plate from a cell that had failed several years ago.[3] The steel plate examined was nominally 25.4 mm (1 in.) thick, had a coating on one side to prevent rapid oxidation by the heating system, and had a crack that penetrated its entire thickness. Metallographic examination in the vicinity of the crack showed that corrosion had proceeded along a fairly uniform front and that cracking had occurred in zones where the carbides appeared to have been leached from the material. Figure 1 shows a cross section of the full plate in the vicinity of the crack, and Fig. 2 shows higher magnification views of the material near the crack. Observations from the metallographic examination are consistent with the theory that lithium reacts with carbides in A285 and makes the material vulnerable to cracking.

To select materials that would be more suitable as the electrolytic cell container, we reviewed literature on the corrosion of materials by

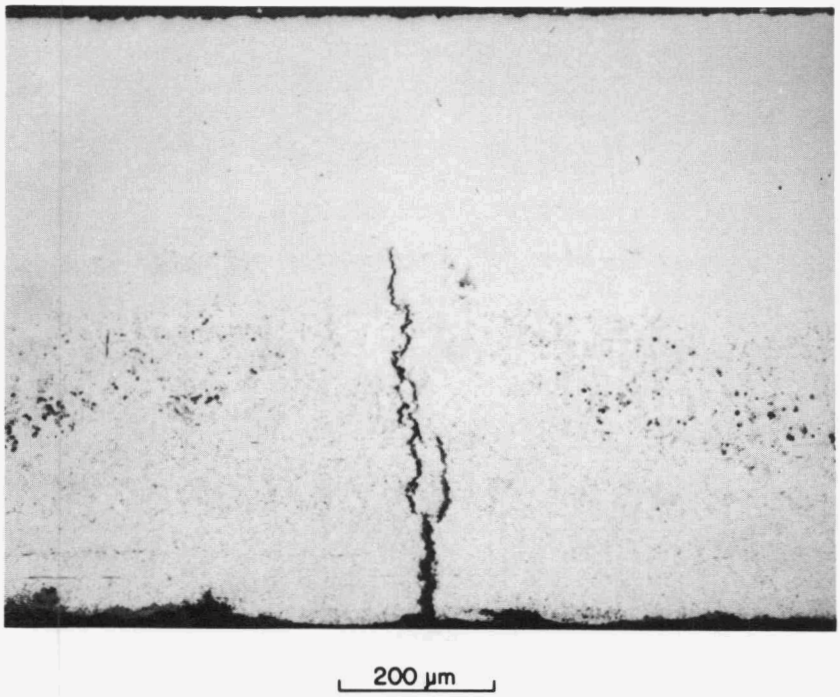

Fig. 1. Cross section of 25.4-mm (1-in.) A285 steel plate taken from lithium electrolytic cell.

molten salts[4-7] and liquid metals.[8-15] Results from tests in lithium indicated that very low carbon steels or steels with carbides more stable than lithium carbide should be used. Carbides reported to be less stable than lithium carbide (below 500°C) include iron carbide, manganese carbide, and molybdenum carbide (marginally); more stable carbides include chromium carbide, niobium carbide, titanium carbide and tantalum carbide.[16,17] Thermodynamic calculations based on recent data indicate that molybdenum carbide is more stable than lithium carbide.[18] On the basis of this information, five materials were selected for evaluation. The compositions of these five materials together with that of A285 Grade A steel are listed in Table 1.

Interstitial free steel (I-F steel) and TI-Namel both contain very low carbon concentrations and are titanium stabilized so that carbon should be present as a titanium carbide or carbonitride. The 2.25Cr-1Mo steel has a fairly high carbon level and should contain iron, chromium, and molybdenum carbides. The modified 9Cr-1Mo steel contains slightly

400 μm

Fig. 2. Higher-magnification view taken in the vicinity of a crack in the A285 steel body of a lithium electrolytic cell. Note the presence of carbides in the material beyond the crack and the absence of carbides in the cracked material (50x).

less carbon than 2.25Cr-1Mo steel, and carbides should be present as $M_{23}C_6$(Nb,Cr) or MC(V,Cr), which are very stable. The other material, type 304L stainless steel, has a fairly low carbon content and sufficient chromium to avoid formation of the less-stable carbides.

EXPERIMENTAL PROCEDURE

Three types of corrosion tests were used to evaluate the materials selected. In the most prototypic type of test, samples of the material were exposed in an operating electrolytic cell so that they contacted both

Table 1. Nominal or specified composition (wt %) of A285 Grade A steel and proposed alternate materials for electrolytic cell containment vessels[a]

Element	A285 Grade A	I-F steel	TI-Name1	2.25Cr-1Mo	Modified 9Cr-1Mo	304L SS[b]
C	0.17 M	0.010 M	0.06 M	0.15	0.08-0.12	0.03 M
N		0.006			0.03-0.07	0.10 M
Cr				2.0-2.5	8.0-9.0	18-20
Mo				0.90-1.10	0.85-1.05	
Ni					0.2 M	8-12
Mn	0.90 M	0.24	0.30	0.30-0.60	0.30-0.50	2.0 M
Ti		0.12	0.30			
Nb		0.06			0.06-0.20	
S	0.040 M	0.008		0.035 M	0.01	0.030 M
P	0.035 M	0.011		0.035	0.02	0.045 M
Al		0.05	0.05			
V					0.18-0.25	

[a]If a maximum composition is specified, it is denoted by an M.

[b]SS = stainless steel.

lithium and the eutectic salt mixture in the presence of air. The samples were mounted on a rack (Fig. 3) that was attached to the A285 steel shell. Two samples of each alloy were mounted on the rack during each exposure, and some were exposed during two periods to get longer exposure times.

Materials were also exposed in capsules containing highly purified lithium to compare corrosion by "clean" lithium with that by the cell lithium that is contaminated through contact with air and salt. Advanced cell designs have been proposed that do not use moist air to remove the chlorine, and the lithium produced in these cells may be similar to that used in the capsule tests.

In a third set of experiments, samples were exposed to a mixture of moist air (50% relative humidity) and 1% Cl_2 gas similar to that encountered in the vapor space of the cell (450°C) and in the exhaust system (90°C).

After testing, the samples from each type of test were evaluated by weight change measurements and metallographic examination.

RESULTS

Three test exposures were made in electrolytic cell number 12 at the Y-12 Plant at a temperature of about 440°C. Test A involved a 200-h exposure, test B lasted for 1017 h, and test C exposed six new samples and six samples from test B for an additional 1031 h. Test A was terminated prior to the originally scheduled period, and the samples were not promptly cleaned and stored in a dry environment but were left in the wash water for several days. Consequently, the measured weight changes on samples from test A are of questionable value. Nevertheless, the A285 and 304L samples were metallographically examined, and, as shown in Fig. 4, intergranular attack to a depth of nearly 0.50 mm occurred in the A285 sample while no attack is evident on the type 304L stainless steel.

Average corrosion rates calculated on the basis of weight-loss measurements on samples from test B are shown in Table 2 (two samples of each material were tested). Assuming that the weight losses were due to uniform removal of material, the corrosion rate of type 304L stainless steel was less than 0.05 mm/year (2 mil/year). All of the other materials had

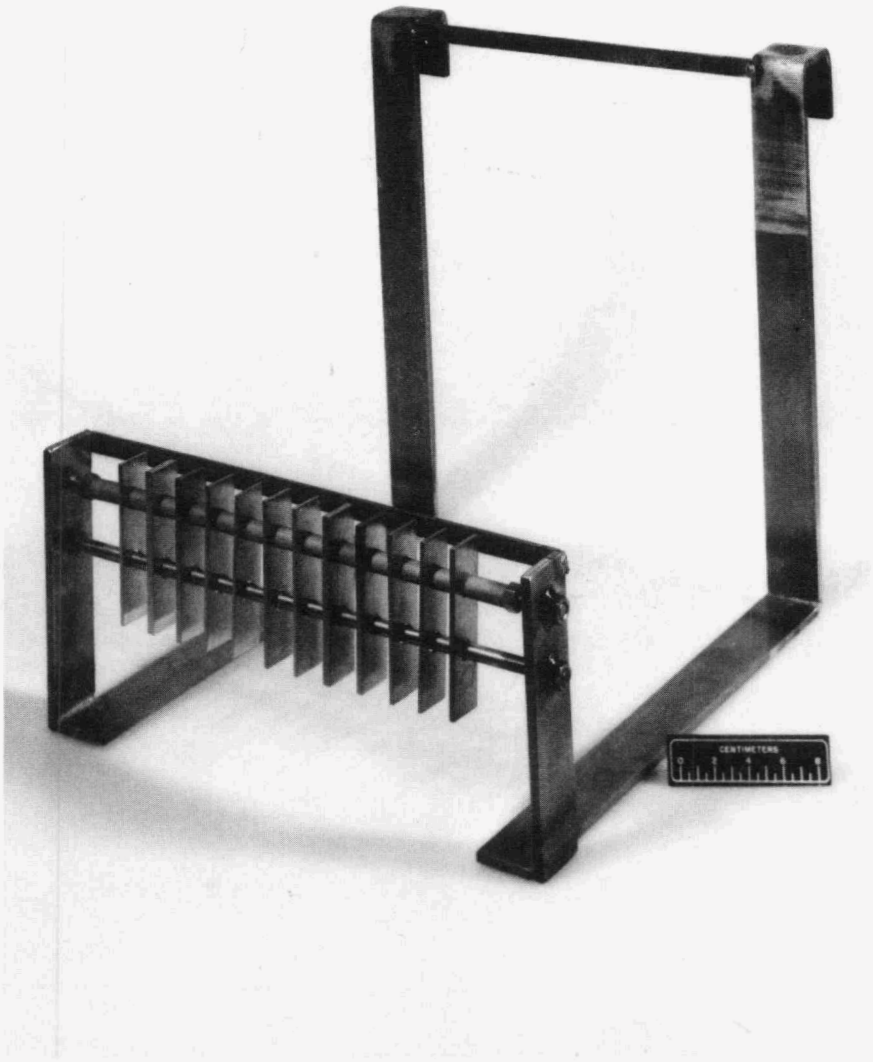

Fig. 3. Rack with 12 mounted samples as used for exposures of samples in operating lithium electrolytic cell.

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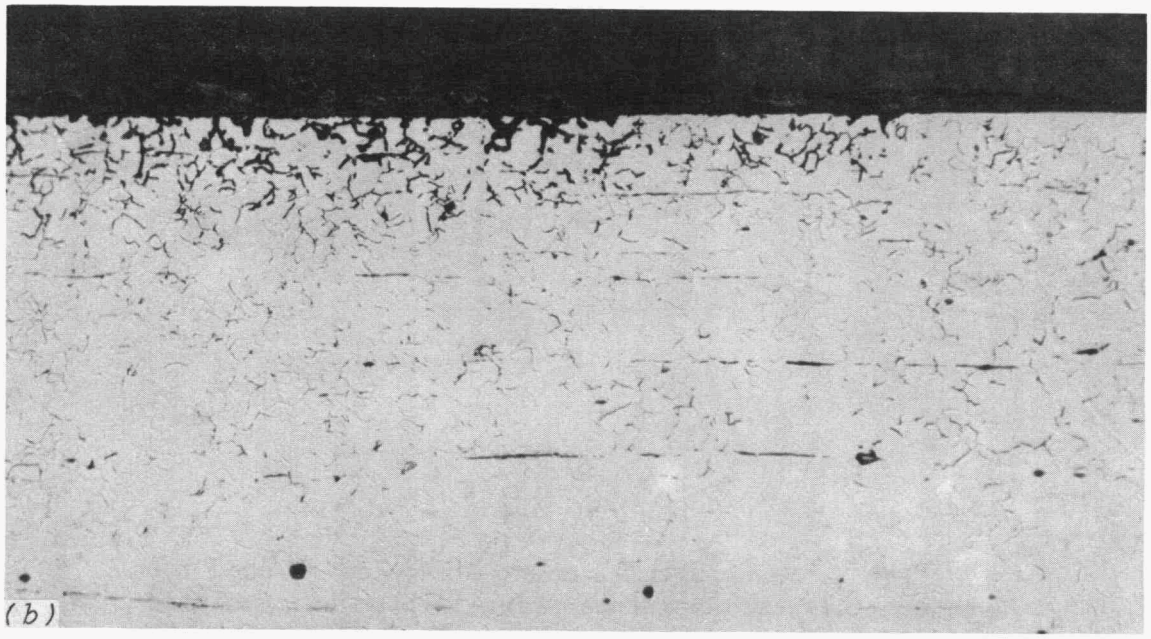

200 μm

Fig. 4. Cross-sectional view of samples exposed for about 200 h to lithium and molten salt in a lithium electrolytic cell operating at about 440°C; (*a*) 304L stainless steel, (*b*) A285 Grade C (carbon in Grade A range) steel.

Table 2. Corrosion rates calculated on the basis of weight losses in samples exposed 1017 h to lithium and molten salt in an electrolytic cell[a]

Material	Corrosion rate [mm/year (mils/year)]
TI-Namel	0.245 (9.6)
I-F steel	0.232 (9.1)
A285 steel	0.197 (7.7)
2.25Cr-1Mo	0.150 (5.9)
Modified 9Cr-1Mo	0.117 (4.6)
304L stainless steel	0.028 (1.1)

[a]Values shown are an average for sample sets 9 and 10, and the rates were calculated assuming uniform removal of material from the surface only.

corrosion rates of less than 0.25 mm/year (10 mil/year). Although this is a convenient way to assess material performance, it does not give a realistic indication of corrosion for materials like A285 where attack is nonuniform.

One set of samples from test B was examined metallographically. Pieces were cut from the top and bottom regions of each sample so that corrosion by both the lithium and the salt could be characterized. As is apparent in Fig. 5(*a*), the top section of A285 was attacked intergranularly to a depth of at least 1.2 mm. Shallow intergranular attack occurred in TI-Namel [Fig. 5(*c*)], while the I-F steel, 2.25Cr-1Mo, and modified 9Cr-1Mo had rough, irregular surfaces but showed no intergranular attack [Figs. 5(*b*), (*d*), and (*e*), respectively]. The surface of the type 304L stainless steel sample was relatively smooth [Fig. 5(*f*)].

Metallographic examination of the sections cut from the bottom region of each sample (exposed to the molten-salt mixture that may contain some dissolved lithium) showed that intergranular attack occurred on the A285 sample while the other five samples had smooth surfaces (Fig. 6). Thickness measurements on these samples (see Table 3) showed relatively little change in those portions of the samples immersed in the salt and, except

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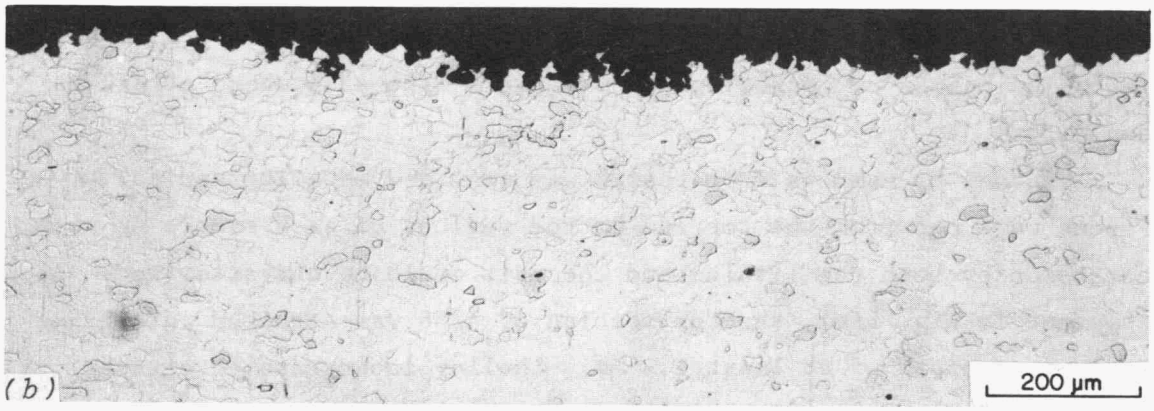

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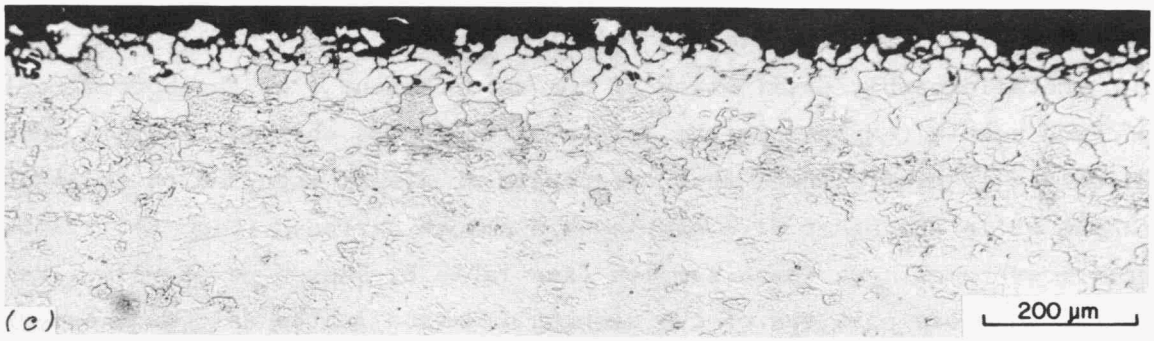

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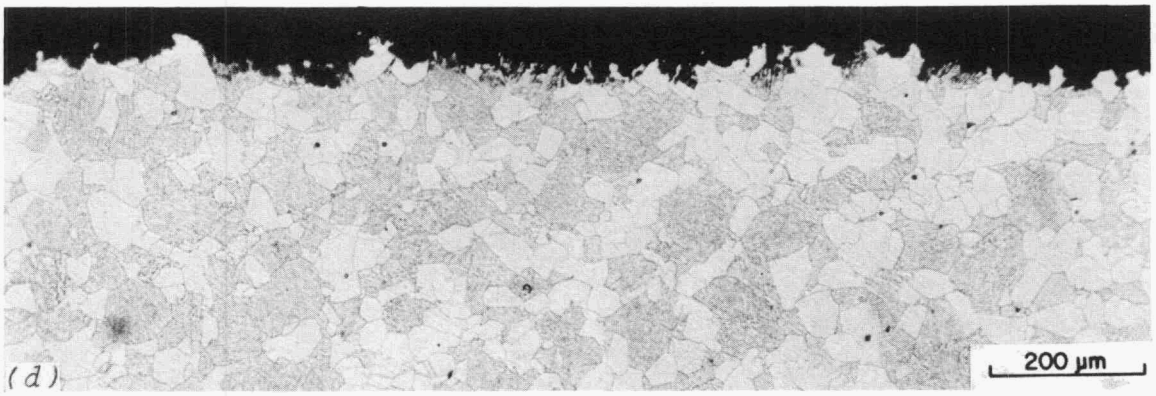

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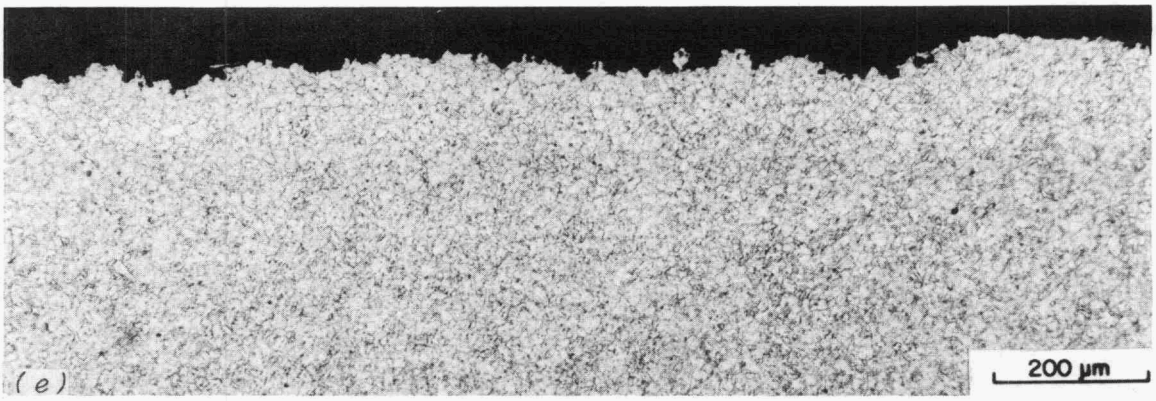

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Fig. 5. Photomicrographs of etched samples after 1017 h of exposure to lithium in an electrolytic cell at 440°C; (*a*) A285 steel, (*b*) I-F steel, (*c*) TI-Namel, (*d*) 2.25Cr-1Mo steel, (*e*) modified 9Cr-1Mo steel, and (*f*) 304L stainless steel.

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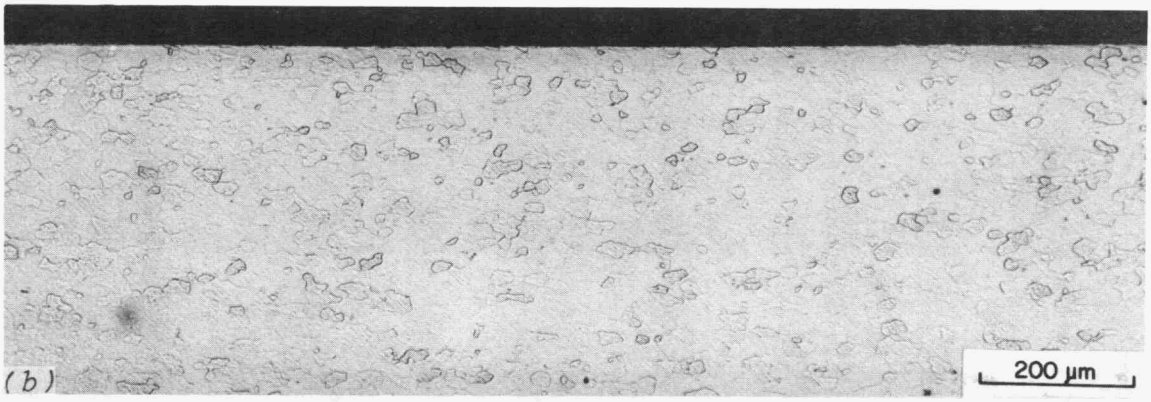

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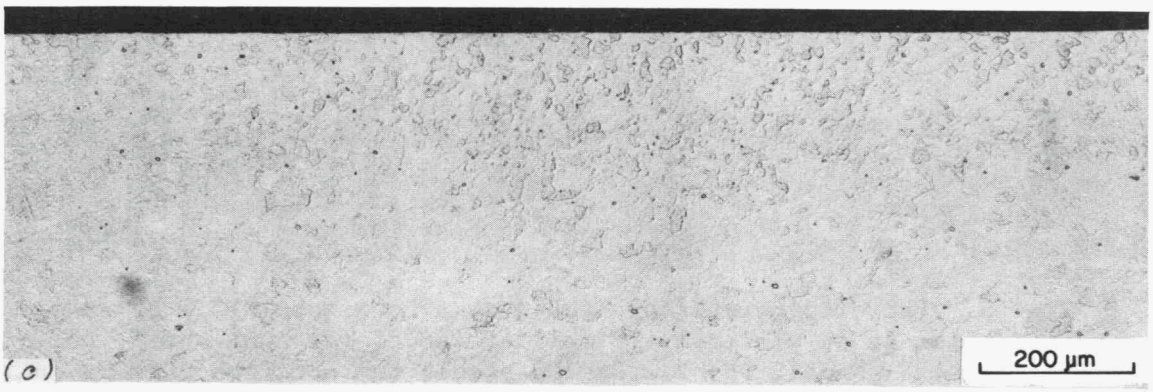

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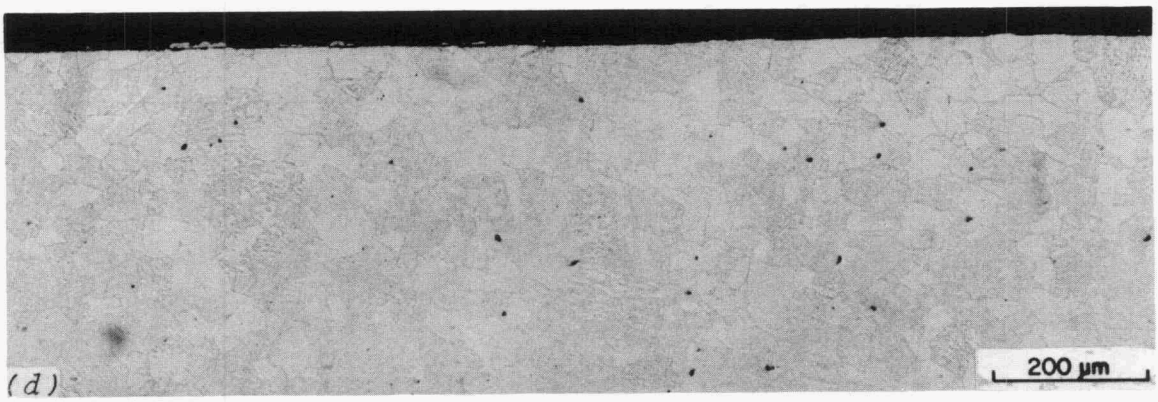

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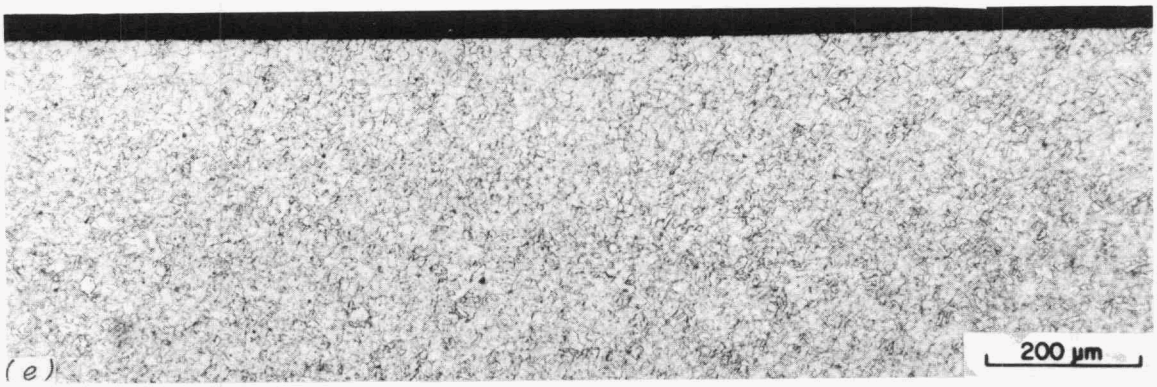

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Fig. 6. Photomicrographs of etched samples after 1017 h exposure to molten salt in an electrolytic cell at 440°C; (*a*) A285 steel, (*b*) I-F steel, (*c*) TI-Namel, (*d*) 2.25Cr-1Mo steel, (*e*) modified 9Cr-1Mo steel, and (*f*) 304L stainless steel.

Table 3. Thickness change and subsurface penetration of materials exposed in electrolytic cell 12 for 1017 h at about 440°C[a]

Material	Decrease in thickness (mm)		Intergranular penetration (mm)	
	Top	Bottom	Top	Bottom
A285 steel	0.38	0.00	1.25	1.20
I-F steel	0.38	0.03	<0.03	<0.03
TI-Namel	0.41	0.05	0.05	<0.03
2.25Cr-1Mo	0.33	0.03	<0.03	<0.03
Mod 9Cr-1Mo	0.41	0.10	<0.03	<0.03
304L stainless steel	0.15	0.00	<0.03	<0.03

[a]For all samples the majority of the material lost was from the top of the sample that was exposed to lithium. Only the A285 sample suffered extensive subsurface penetration. (Multiply by 8.6 to express in material lost or penetration depth per year.)

for the type 304L stainless steel, fairly uniform changes in thickness in portions of the samples exposed to the lithium.

Table 3 also shows the extent of subsurface penetration that occurred; note that the A285 sample was the only material exhibiting appreciable subsurface attack. Measurement of the depth of attack in the top portion of each sample is complicated by the fact that salt and lithium levels fluctuated, thereby causing irregular attack. The values shown in the table indicate the maximum penetration observed.

Results on the samples that were exposed only in test C were qualitatively consistent with the previous studies in that the materials generally lost weight (except for A285), they showed a reduction in thickness for the portion exposed to lithium, and A285 and TI-Namel were attacked intergranularly. However, conditions in the cell must have been significantly different from test B because weight losses were much lower during this test. Data on these samples are given in Table 4.

Results on samples exposed in both tests B and C (sample set 10) (see Table 5) also indicate that conditions were different for the two

Table 4. Corrosion rates and subsurface penetration of materials from sample set 7 that were exposed in electrolytic cell 12 for 1031 h at about 440°C[a]

Material	Corrosion rate		Intergranular penetration (mm)	
	mm/year	mils/year	Top	Bottom
A285 steel	Weight gain	Weight gain	1.09	1.30
TI-Namel	0.005	0.2	0.18	0.18
I-F steel	0.009	0.3	<0.03	<0.03
2.25Cr-1Mo	0.006	0.2	<0.03	<0.03
Mod 9Cr-1Mo	<0.003	<0.1	<0.03	<0.03
304L stainless steel	0.011	0.4	<0.03	<0.03

[a]Samples exposed during this operating period generally had relatively small weight losses; however, subsurface attack was appreciable on some samples.

Table 5. Weight change and subsurface penetration of materials from sample set 10 that were exposed during runs B (1017 h) and C (1031 h)

Material	Weight change (mg/cm^2)		Intergranular penetration (mm)	
	Run B	Run C	Top	Bottom
A285 steel	-16.3	+1.81	>3.18	>3.18
TI-Namel	-25.0	-7.74	0.10	0.23
I-F steel	-17.6	+0.25	<0.03	<0.03
2.25Cr-1Mo	-9.7	+0.33	<0.03	<0.03
Mod 9Cr-1Mo	-8.5	+0.42	<0.03	<0.03
304L stainless steel	-1.5	+0.91	<0.03	<0.03

test periods. Samples that lost weight in test B gained weight in test C (except TI-Namel). However, the net overall change (tests B and C) was a weight loss. The A285 sample was intergranularly attacked through its entire thickness, and TI-Namel incurred shallow intergranular attack as shown in Fig. 7.

The second type of test involved exposing samples of these same materials to high purity lithium in capsules (or liners) that were constructed of the same materials as the samples. The lithium used was purified by cold trapping and subsequent gettering at 815°C for 100 h in a titanium-lined pot containing zirconium foil. Typical impurity concentrations of the purified lithium were nominally 30 to 100 wt ppm of nitrogen and 30 to 130 wt ppm of oxygen as measured by micro-Kjeldahl and neutron activation analysis techniques, respectively. Two tests (800 and 2000 h) were conducted on each material at 500°C. As before, the samples were cleaned and weighed after testing before being examined metallographically.

For materials exposed about 800 h, corrosion rates calculated from the weight changes indicated that material loss was negligible. As evidenced by the photomicrographs in Fig. 8, there was no evidence of corrosion except for A285 steel, which again showed intergranular attack to a depth of about 1.25 mm.

A second set of materials (except for type 304L stainless steel) was exposed for about 2000 h in purified lithium. The A285 steel showed a small weight gain while the other materials showed slight weight losses. Calculated corrosion rates were again very low. Metallographic examination showed no evidence of corrosion except for A285, which exhibited intergranular penetration (see Fig. 9).

The increased corrosion resistance exhibited by the samples exposed to high purity lithium compared with those exposed in the electrolytic cell is almost certainly due to impurities in the electrolytic cell lithium. Many investigators have described an increase in corrosivity of steels caused by nitrogen in lithium.[19-22] The reaction

$$5Li_3N + Cr(s) = Li_9CrN_5 + 6Li \ , \qquad (1)$$

has been observed and is thought to indicate how nitrogen can increase the corrosivity of lithium.[23] It would not be surprising if the lithium

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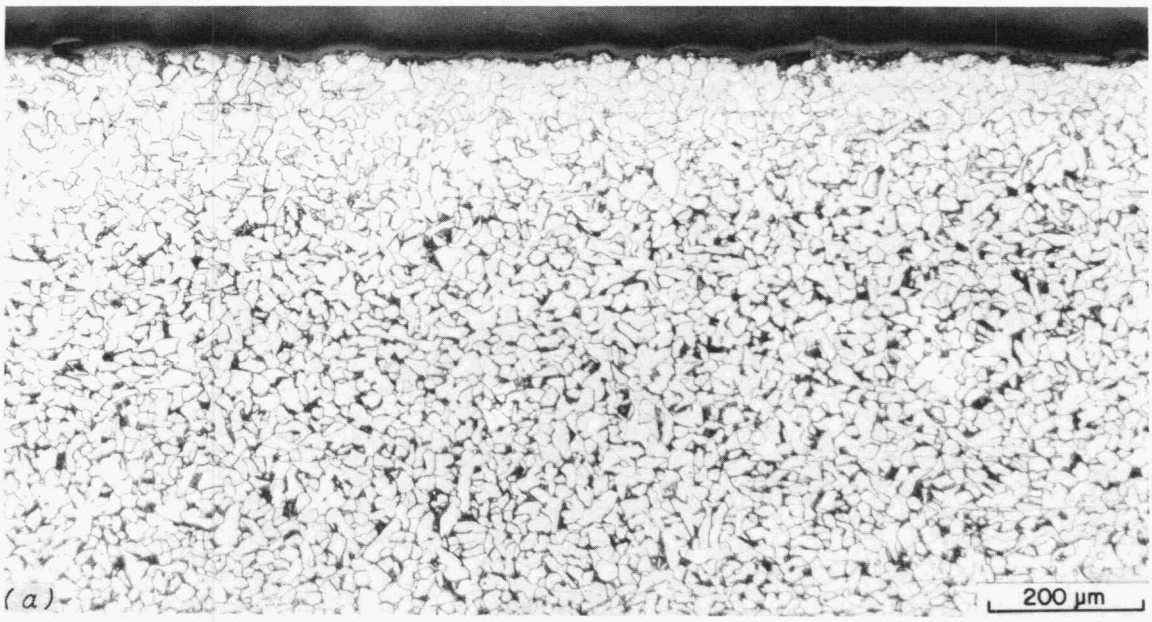

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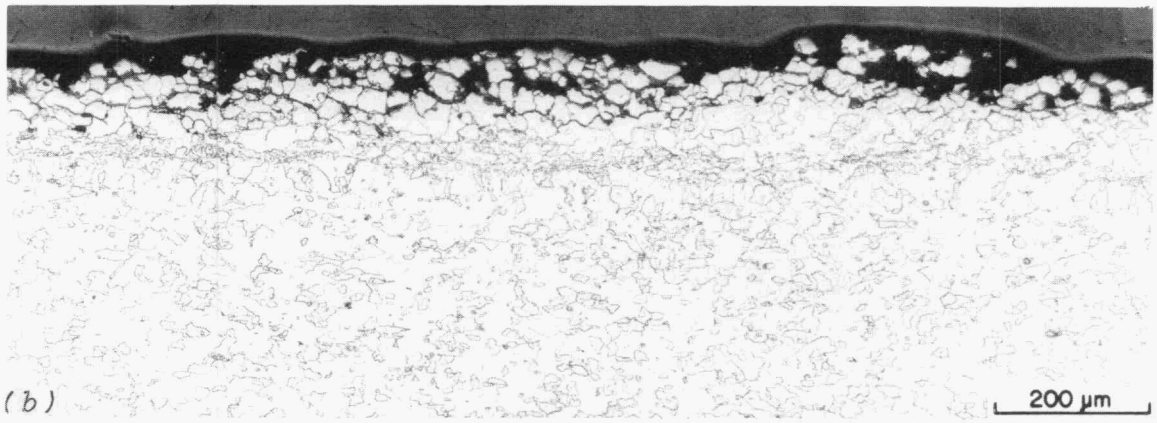

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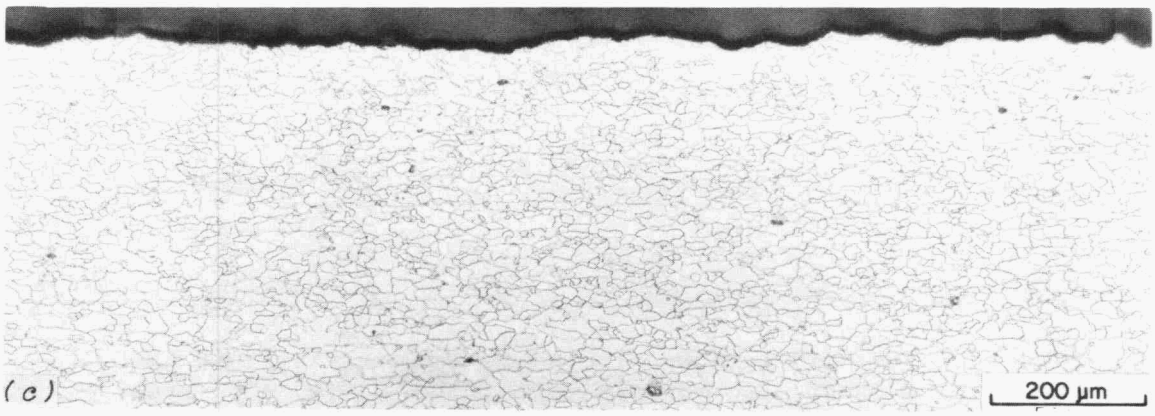

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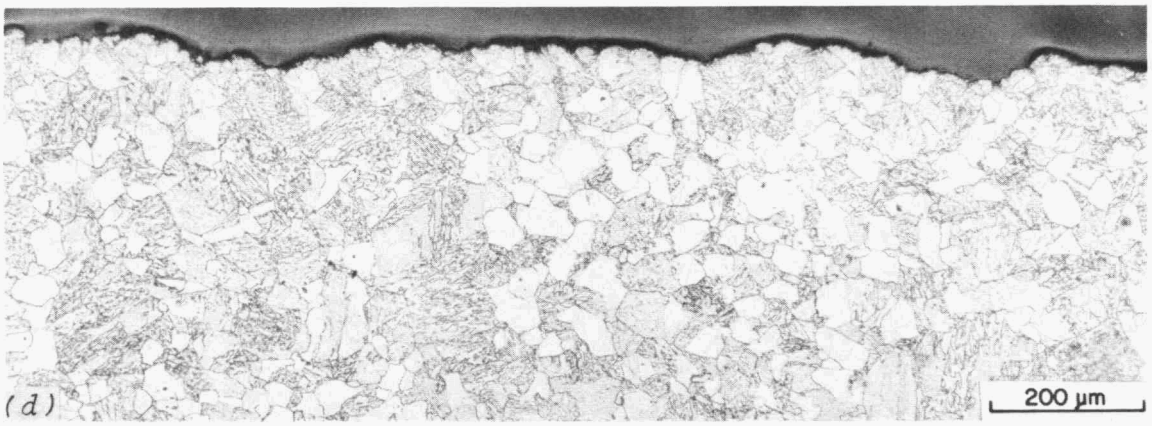

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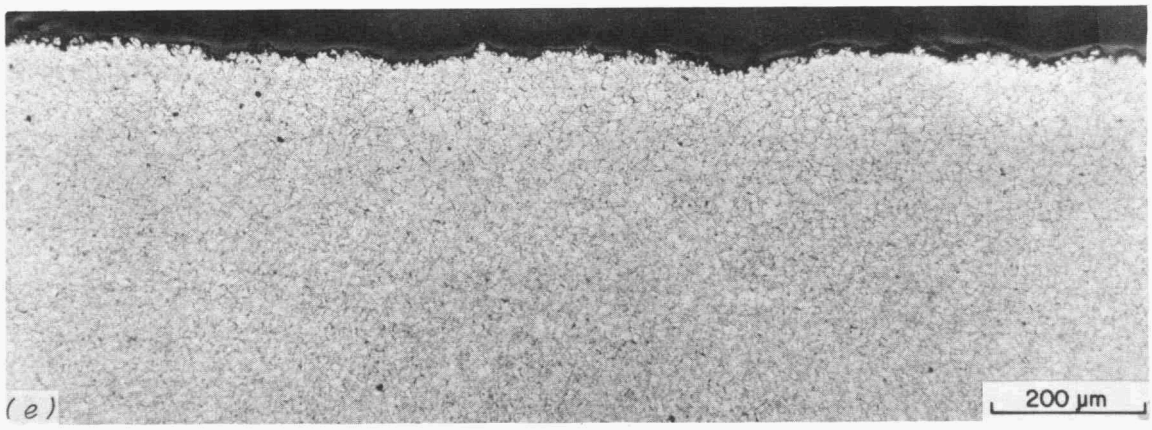

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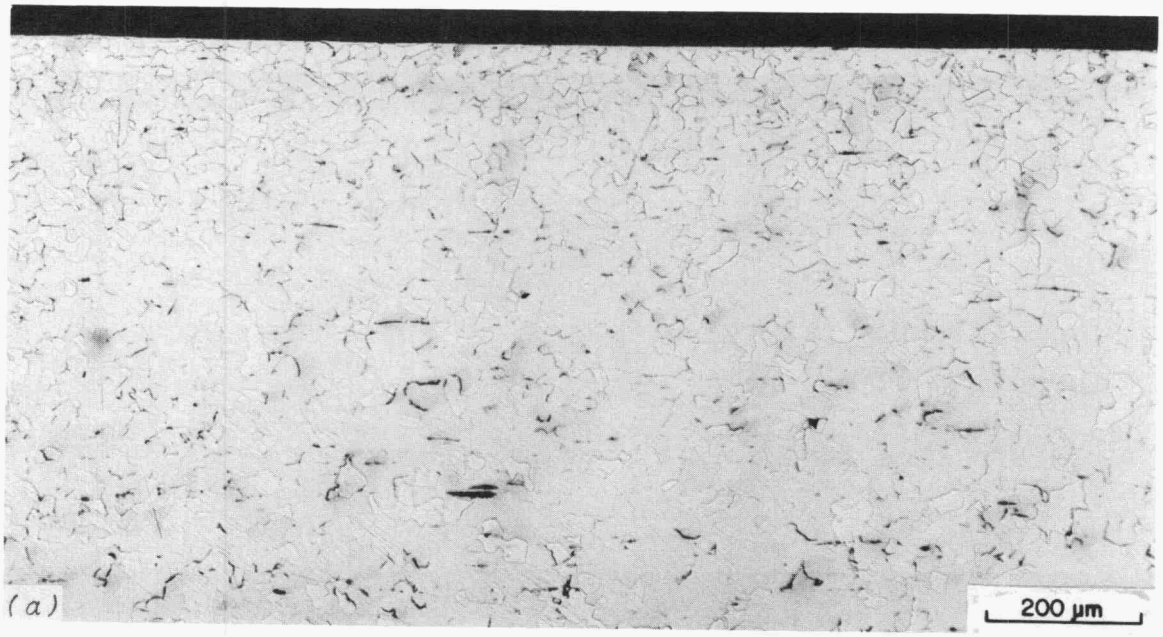
Fig. 7. Photomicrographs of etched samples after 2048 h of exposure to lithium in an electrolytic cell at 440°C; (*a*) A285 steel, (*b*) I-F steel, (*c*) TI-Namel, (*d*) 2.25Cr-1Mo steel, (*e*) modified 9Cr-1Mo steel, and (*f*) 304L stainless steel.

Y-208214



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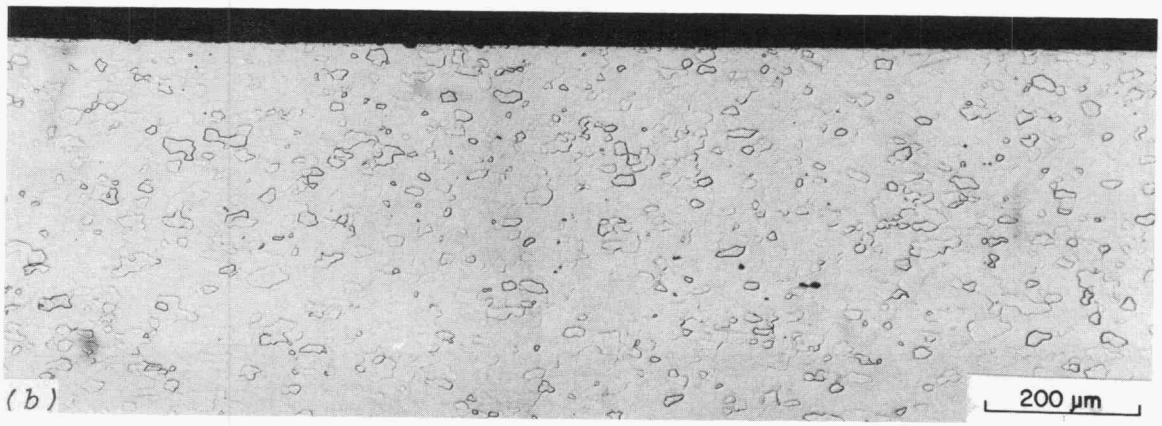

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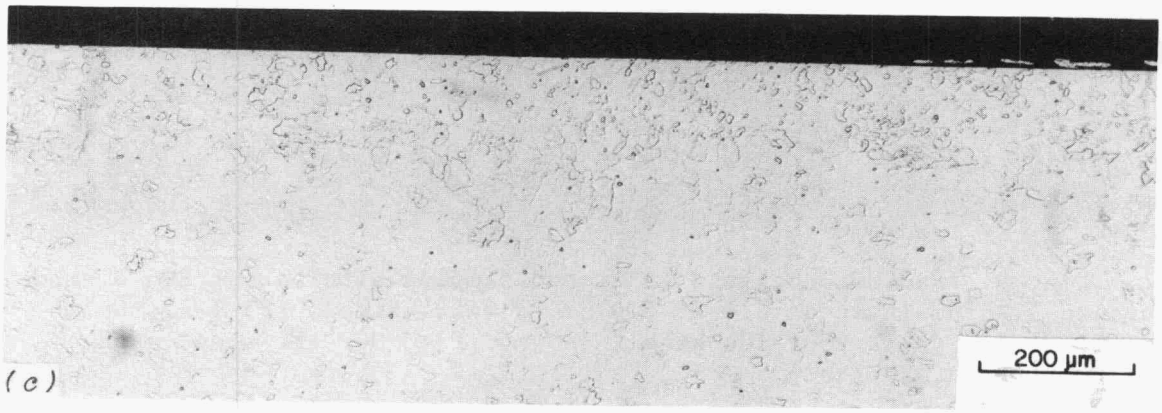

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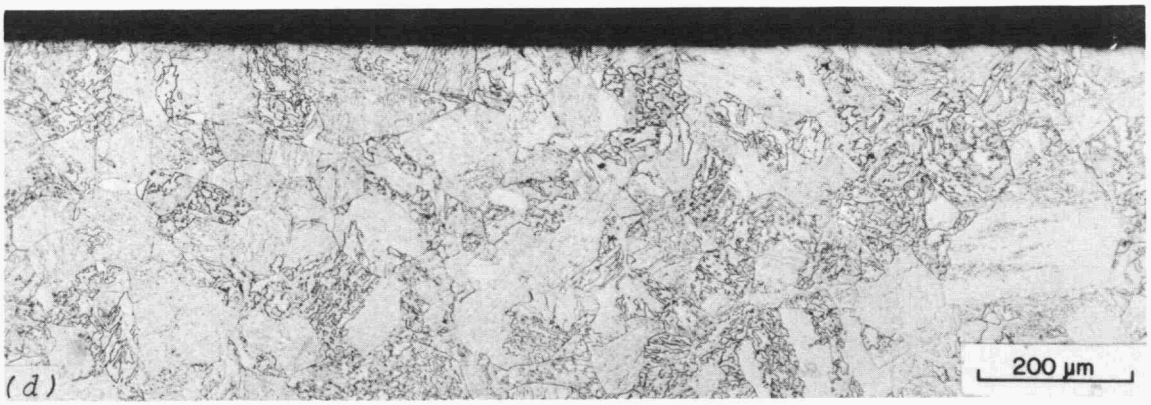

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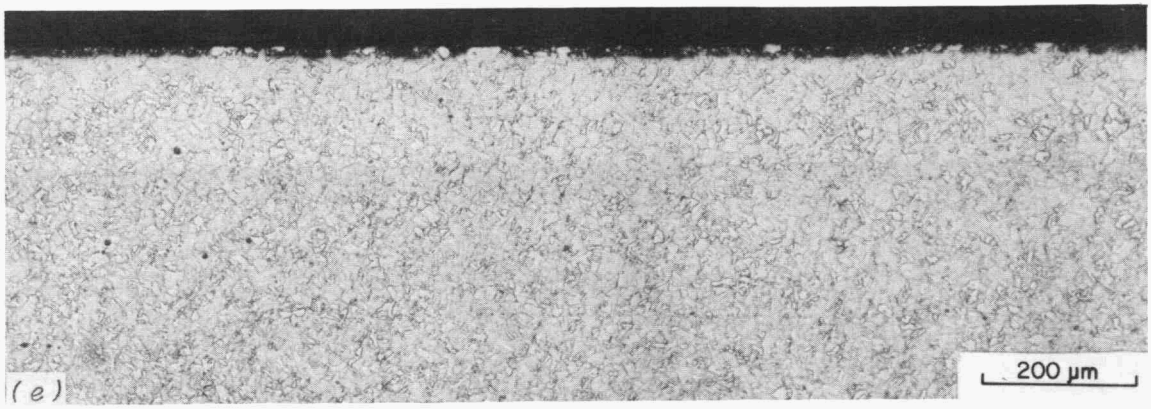
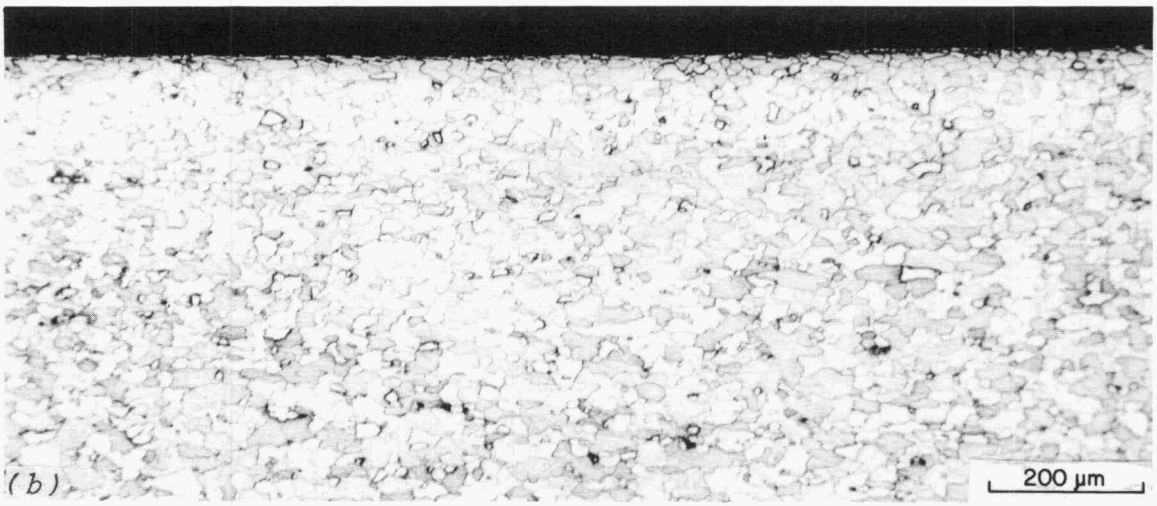


Y-208217

Fig. 8. Photomicrographs of etched samples after about 800 h exposure to lithium in a capsule at 500°C: (*a*) A285 steel, (*b*) I-F steel, (*c*) TI-Namel, (*d*) 2.25Cr-1Mo steel, (*e*) modified 9Cr-1Mo steel, and (*f*) 304L stainless steel.

Y-208743

Y-208747



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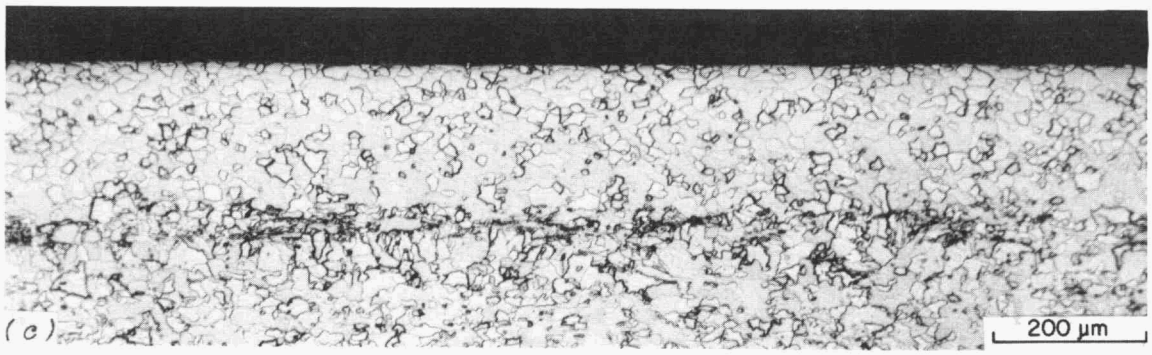
Y-208753



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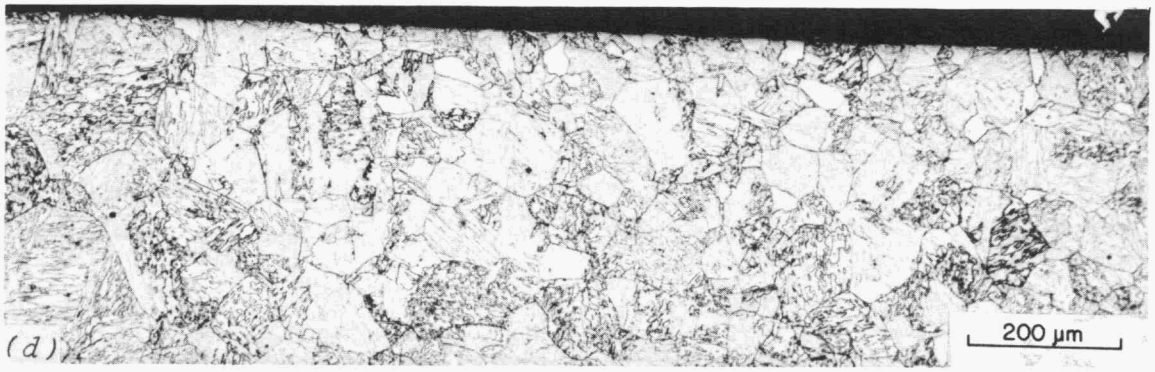

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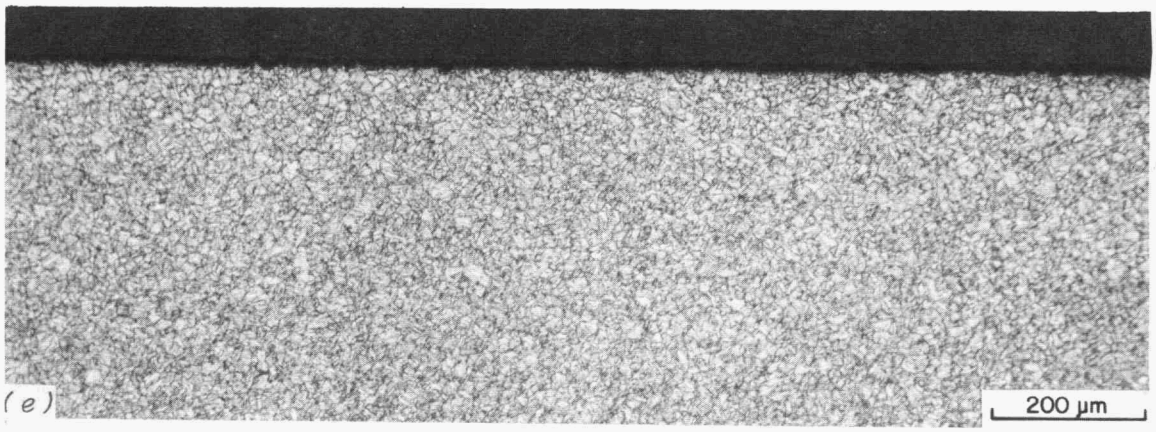

Fig. 9. Photomicrographs of etched samples after about 2000 h of exposure to lithium in a capsule at 500°C; (*a*) A285 steel, (*b*) I-F steel, (*c*) TI-Namel, (*d*) 2.25Cr-1Mo steel, and (*e*) modified 9Cr-1Mo steel.

picked up nitrogen during exposure to the moist air used to flush chlorine from the electrolytic cell. In addition, impurities could also be acquired from the salt that is periodically added to the cell.

The current means of disposal of the electrolytically produced chlorine is to mix it with a large quantity of room air and react it with an NaOH solution to form NaCl. Some corrosion of the venting system has also been noted, so an evaluation of the materials in chlorine was included as part of the overall work scope of this study. A series of three tests was conducted to determine the resistance of the materials to a chlorine environment; all tests were conducted in a gas atmosphere that was a 50:50 mix of fully humidified air with air containing 2% chlorine. In the first test, nine samples were exposed for 240 h to this mixed-gas atmosphere at about 400°C. In the second test, nine samples were exposed for 526 h at about 450°C, and the third test involved twelve samples at 90°C for about 265 h.

Corrosion rates calculated from weight losses, assuming uniform removal of material, were fairly low for all of the materials in each of the three tests. As shown in Tables 6-8, severe corrosion was not encountered in any of the exposures. However, the experimental system did not allow testing under fully prototypic conditions. If the relative humidity had been maintained at a higher level and the samples had been cooler than the gas, as is the case for the ventilation ducts of the cell system, more serious corrosion might have occurred. Consequently, if a more definitive evaluation of these materials to the chlorine gas environment in the cell system is required, additional testing under different conditions, perhaps as corrosion samples in the ventilation ducts, should be carried out.

CONCLUSIONS AND RECOMMENDATIONS

All five alternative materials evaluated in these studies showed considerably better corrosion resistance to the electrolytic cell environment than did the A285 steel. Although corrosion rates calculated on the basis of weight-change measurements were not severe for any of the materials tested, metallographic determination of subsurface attack permitted a clear differentiation among the materials.

Table 6. Corrosion rates calculated from weight-loss measurements on samples exposed 240 h to air with 1% chlorine and 50% relative humidity[a]

Material	Corrosion rate	
	mm/year	mils/year
TI-Namel	0.168	6.6
I-F steel	0.161	6.3
A285 steel	0.126	5.0
Hastelloy C-276	<0.002	<0.1
Nickel	<0.002	<0.1
304L stainless steel	<0.002	<0.1
2-mil electroless nickel[b,c]	<0.002	<0.1
2-mil electrolytic nickel[c]	<0.002	<0.1
5-mil electrolytic nickel[c]	<0.002	<0.1

[a]Samples were exposed at 348, 418 and 447°C for approximately equal periods.

[b]Proprietary nickel coating with 9-10% phosphorus.

[c]Coatings were placed on I-F steel samples.

Of the five alternative materials, TI-Namel was the only one that showed significant intergranular attack in any of the studies. Even though intergranular attack was appreciably less than that in A285, this alloy probably should not be considered further as a primary candidate for cell construction. In addition to intergranular attack, the relatively high carbon content of TI-Namel steel (compared with that of Armco iron) causes us to have some concerns about the properties of welds in this alloy. Furthermore, information about the elevated temperature properties of this material has been very difficult to acquire, although it is probably safe to assume that its properties are similar to those of Armco iron or I-F steel.

Table 7. Corrosion rates calculated from weight-loss measurements on samples exposed 526 h at 450°C to air with 1% chlorine and 50% relative humidity

Material	Corrosion rate	
	mm/year	mil/year
2.25Cr-1Mo	0.200	8.0
TI-Namel (reused)	0.170	6.8
TI-Namel (new)	0.098	3.9
Modified 9Cr-1Mo	0.077	3.1
Nickel	0.069	2.8
5-mil electrolytic nickel[a] (reused)	0.023	0.9
2-mil electroless nickel[a,b] (reused)	<0.002	<0.1
Type 308 stainless weld overlay	<0.002	<0.1
Hastelloy C-276	<0.002	<0.1

[a]Coatings were placed on I-F steel samples.

[b]Proprietary nickel coating with 9-10% phosphorus.

Type 304L stainless steel performed extremely well in the lithium and chlorine compatibility tests; however, the alloy should probably be dropped from further consideration because of two potential problems. First, austenitic stainless steels are susceptible to stress corrosion cracking in environments containing chloride. Secondly, nickel has a relatively high solubility in lithium,[9,11,24,25] and type 304L stainless steel contains about 10% nickel. For the eventual product application, it is especially undesirable to have nickel in the lithium product; therefore, application of a 300-series stainless steel as the cell containment material is probably not justifiable.

Selecting the most suitable of the remaining three materials is not straightforward, because advantages and disadvantages can be described for each.

Table 8. Corrosion rates calculated from weight-loss measurements on samples exposed 265 h at 90°C to air with 1% chlorine and 50% relative humidity

Material	Corrosion rate	
	mm/year	mil/year
304L stainless steel	0.023	0.9
2.25Cr-1Mo	0.006	0.3
I-F steel (avg. of 2)	0.005	0.2
2-mil electrolytic nickel[a]	<0.002	<0.1
5-mil electrolytic nickel[a]	<0.002	<0.1
A285 (avg. of 2)	<0.002	<0.1
2-mil electroless nickel[a,b]	<0.002	<0.1
Hastelloy C-22	<0.002	<0.1
Modified 9Cr-1Mo	<0.002	<0.1

[a]Coatings were placed on I-F steel samples.

[b]Proprietary nickel coating with 9-10% phosphorus.

We consider I-F steel to be an excellent candidate for replacement of the A285 steel. It showed good compatibility with lithium and the moist air–chlorine gas mixture, and its very low carbon content together with the presence of titanium and niobium as carbon stabilizers should avoid the long-term problem of carbide attack by lithium. Potential problems with I-F steel that must be addressed are weldability, corrosion resistance of welds, elevated-temperature strength, and availability in the required forms.

Relative to weldability, there should be few or no carbides if matching filler wire is used, and, if the concentration of the stabilizing elements can be maintained in the weld, any carbides that do form should be stable. To resolve any concerns about welds, we recommend exposing welded samples of this material in one of the operating cells for a period of 1000 h or, preferably, longer.

Another possible concern with I-F steel is a lack of high-temperature strength data. Available information on the elevated-temperature mechanical properties of A285 steel, I-F steel, 2.25Cr-1Mo steel and modified 9Cr-1Mo steel are given in Appendix A. Armco, Inc., developer of I-F steel, has provided data at 538 to 760°C (1000 to 1400°F), which show that I-F steel in sheet form has a yield strength greater than a low-carbon steel. Information on creep strength has been difficult to find, and it may be necessary to perform creep tests on the product form that would be used for the cells. A more highly alloyed variation of I-F steel is currently made in sheet form for a hot dipped aluminum product. This product has improved strength and might be available in plate form without the aluminum coating.

Availability of I-F steel could also present a problem if a relatively small quantity is ordered. Discussions should be held with the steel producers to assess cost and availability. In addition, unlike 2.25Cr-1Mo and modified 9Cr-1Mo steel, American Society for Testing and Materials specifications do not exist for I-F steel in the forms needed for an electrolytic cell. Consequently, the specifications summarized in Appendix B were written.

Modified 9Cr-1Mo and 2.25Cr-1Mo steel generally performed slightly better than the I-F steel in the corrosion tests. Although both chromium-molybdenum steels require a normalizing and tempering treatment (see Appendix C), they are very well characterized as to their fabricability and elevated-temperature strength, and both should be more than adequate for this application. Both steels would require postweld heat treating (see Appendix C), but they should be more readily available than the stabilized low-carbon steels. The carbides formed in the modified 9Cr-1Mo alloy are a chromium-vanadium-niobium type and should be highly resistant to attack by lithium. After certain heat treatments, the carbides in 2.25Cr-1Mo steel are known to be susceptible to attack by lithium.[16]

One concern with use of these two steels relates to their heat-treating requirements. It should be established that the heat-treating specifications developed for sodium service are acceptable for the electrolytic cell environment.

Modified 9Cr-1Mo steel is more resistant to decarburization in sodium than 2.25Cr-1Mo steel and, in fact, behaves similarly to type 316 stainless steel.[26] Therefore, we would expect this alloy to perform well in the cell environment, but we hesitate to select modified 9Cr-1Mo as the primary candidate until additional compatibility information can be acquired.

Hastelloy C-276 has been proposed as the material to be used for the cover of the electrolytic cell, and no experiments were performed on cover materials because that application involves different considerations than those for the cell-body material. The cover material needs sufficient strength to support the anodes and should be resistant to corrosion by chlorine and hydrochloric acid. We note, however, that an electroless nickel coating on one of the alloy steels, modified 9Cr-1Mo or 2.25Cr-1Mo, would be a less expensive alternative to Hastelloy C-276.

ACKNOWLEDGMENTS

We acknowledge P. S. Bishop, M. Howell, J. W. Hendricks, and L. T. Ratcliff for conducting the experimental studies; B. G. Davis for his assistance in conducting the electrolytic cell tests; and J. R. Mayotte and the Y-12 metallography group for performing the metallographic examinations.

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Appendix A

MECHANICAL PROPERTIES OF THE PRIMARY CANDIDATE STEELS

Property	A285	I-F steel	2.25Cr-1Mo	Mod. 9Cr-1Mo
Room-temperature properties				
0.2% yield strength, MPa (ksi)	165 (24)	172 (25)	207 (30)	414 (60)
Ultimate tensile strength, MPa (ksi)	345 (50)	331 (48)	414 (60)	572 (83)
S_0, design strength, MPa (ksi)	90 (13)	83 (12)	103 (15)	145 (21)
Elongation, %	30	43	37	25
Elevated-temperature (900°F, 482°C) properties				
0.2% yield strength, MPa (ksi)		124 (18)	186 (27)	310 (45)
Ultimate tensile strength, MPa (ksi)	138 (20)		345 (50)	421 (61)
S_0, design strength, MPa (ksi)	34 (5)		90 (13)	110 (16)
Elongation, %			25	20

Appendix B

SPECIFICATION FOR EXTRA-LOW-CARBON KILLED-STEEL PLATES, SHAPES, AND BAR

1.0 Scope

1.1 This specification covers extra-low-carbon-steel plates, shapes, and bars made by vacuum degassing and killing steel practiced by the producer.

1.2 The maximum thickness for plates under this specification is limited to 5.08 cm (2 in.).

2.0 Applicable Documents

2.1 American Society for Testing and Materials Standards

A20/A20M Specification for General Requirements for Steel Plates for Pressure Vessels including Supplement 1 and Supplement S2.

A6/A6M General Requirements for Rolled-Steel Plates, Shapes, Sheet Piling, and Bars for Structural Use.

3.0 General Requirements

3.1 All plates supplied to this specification shall conform to Specification A20/A20M, including Supplements 1 and 2.

3.2 All shapes and bars supplied to this specification shall conform to Specification A6/A6M as amended by this specification.

3.3 Each plate, shape, or bar shall be identified by stamp or stencil with the manufacturer's name, brand, or trademark, the heat number, and the material type.

3.4 All material supplied shall be subjected to vacuum degassing in the molten state during manufacture.

3.5 All material supplied shall have a fine austenitic grain size.

4.0 Heat Treatment

None required.

5.0 Chemical Requirements

5.1 The steel shall conform to the following chemical composition:

Elements	Composition (%)
Carbon, max.	0.010
Manganese, max.	0.35
Titanium, min.	0.05
Phosphorus, max.	0.015
Sulfur, max.	0.030
Nitrogen, max.	0.010
Niobium, min.	0.05
Aluminum, min.	0.02

6.0 Mechanical Requirements

6.1 Tension tests

Yield strength (0.2% strain)	137.9 MPa (20,000 psi) min.
Ultimate strength	310.3 MPa (45,000 psi) min.
Elongation (2-in. gauge length)	30% min.

7.0 Certification and Test Reports

A manufacturer's certification that the material was manufactured and tested in accordance with this specification together with a report of the analysis and tension test results shall be furnished at the time of shipment. The report shall include the name of the manufacturer, material type and grade, heat number, size, and authorized signature.

Appendix C

HEAT TREATING REQUIREMENTS FOR THE HEAT-TREATABLE PRIMARY CANDIDATE STEELS

The 2.25Cr-1Mo and modified 9Cr-1Mo steels can be heat treated to optimize the mechanical properties of the material. The recommended heat treatments for these materials are as follows.

2.25Cr-1Mo Steel

Full anneal requires holding at 927°C (1700°F) for 1 h for each 25.4 mm of thickness, 4-h maximum, 1-h minimum, cooling at 55°C (100°F) per hour to 316°C (600°F), air cooling to room temperature.

Normalizing requires holding at 913-954°C (1675-1750°F) for 1 h for each 25.4 mm of thickness, 4 h maximum, 0.5 h minimum, then air cooling.

Tempering requires holding at 675-732°C (1250-1350°F) for several hours (e.g., 1 h for each 25.4 mm of thickness), then air cooling to room temperature.

Postweld heat treating requires holding at 727°C (1340°F) for at least 0.3 h and up to 4 h, with cumulative time at 727°C (1340°F) not to exceed 40 h for repair of all welds, cooling to room temperature.

Modified 9Cr-1Mo Steel

Austenitizing requires heating to 1000-1040°C.

Normalizing consists of heating the alloy to 1040°C, holding for 1 h (for thicknesses up to 25 mm), and then air cooling to room temperature.

Tempering treatment consists of heating the normalized steel to 760°C, holding for 1 h (for thicknesses up to 25 mm), followed by air cooling to room temperature.

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