DESIGN CONSIDERATIONS OF REACTOR CONTAINMENT SPRAY SYSTEMS - PART X.

THE STRESS CORROSION CRACKING OF TYPES 304 AND 316
STAINLESS STEEL IN BORIC ACID SOLUTIONS

J. C. Griess and G. E. Creek
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Contract No. W-7405-eng-26

Reactor Chemistry Division

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MAY 1971

OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee
operated by
UNION CARBIDE CORPORATION
for the
U.S. ATOMIC ENERGY COMMISSION
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The Spray and Absorption Technology Program is coordinated by Oak Ridge National Laboratory for the AEC. The program involves research on all aspects of containment spray systems proposed for use as an engineered safety feature in pressurized water reactor containment buildings and investigations of certain aspects of the pool-pressure-suppression containment concept as applied to boiling water reactors. A document (ORNL-4360, Spray and Pool Absorption Technology Program) has been issued.

This document reports work on the corrosion of materials by low pH (4.5-7.5) spray solutions. The emphasis in this work was placed on chloride induced stress-corrosion cracking of types 304 and 316 stainless steel. Another report in this same series will cover the aspect of general materials corrosion by similar solutions.


Thomas H. Row
Technical Coordinator
Spray and Absorption Technology Program
THE STRESS CORROSION CRACKING OF TYPES 304 AND 316 STAINLESS STEEL IN BORIC ACID SOLUTIONS

J. C. Griess and G. E. Creek

ABSTRACT

The stress corrosion cracking of types 304 and 316 stainless steel in simulated pressure-suppression and fission-product-absorption sprays was investigated. The test solutions contained 0.28M H₃BO₃ (3000 ppm B) with pH values and chloride concentrations ranging between 4.5 and 7.5, and 5 and 200 ppm, respectively. In addition one test solution contained 2 ppm iodide. All test specimens, mostly U-bends, were exposed for 1 day at 141°C and 7 days at 100°C in a recirculating spray loop and were then transferred to a tank for an additional exposure of 2 months at 82°C. The results showed that both alloys underwent cracking in all solutions. The tendency to crack was greater the lower the pH and the higher the chloride concentration. Type 316 stainless steel was somewhat more resistant to cracking than type 304 stainless steel, and both alloys were more susceptible when sensitized than when annealed. Specimens that were sensitized in air and covered with thin oxide films cracked more frequently than similarly treated specimens that were pickled after sensitization. In all cases except one, the cracks were branching and transgranular. In one case where the solution had a pH of 6.5 and contained 20 ppm chloride and 2 ppm iodide, wide short cracks were found only in the vicinity of welds; no other localized attack was found on any other specimen.

INTRODUCTION

The design of many nuclear power plants utilizing water-cooled reactors includes provisions for a containment vessel spray system to be used in the event of a serious accident. The function of the spray is to reduce the pressure caused by a rupture of the reactor primary system and to absorb certain fission products that may be released to the containment vessel. One important aspect of a spray solution is its corrosiveness to materials in the total system. In a previous report (1) the corrosion rates of a variety of metals and alloys in two typical spray solutions were given.
These solutions contained 0.28M H$_3$BO$_3$ (3000 ppm B) and 0.15M NaOH either with or without 0.064M Na$_2$S$_2$O$_3$ (1 wt. %); in both cases the pH was 9.3 to 9.5. The results of these tests showed that most of the aluminum alloys corroded at very high rates in both solutions and that stressed specimens of types 304 and 316 stainless steel did not develop cracks under the conditions of test when 100 ppm chloride (as NaCl) was added to the solution.

Some nuclear plants have large quantities of aluminum in the reactor containment vessel, and in these cases a very high corrosion rate for the aluminum could result in the generation of enough hydrogen to contribute significantly to the development of an explosive mixture in the containment vessel. The corrosion rate of aluminum is less in slightly acid or neutral solutions than in alkaline solutions,\(^{(2)}\) and for that reason it may be desirable to use spray solutions which contain only boric acid or acid solutions adjusted to near neutral with sodium hydroxide as the initial spray solution. Unfortunately the tendency of the austenitic stainless steels to develop cracks increases as the pH is lowered.\(^{(3,4)}\)

Since data concerning the cracking behavior of austenitic stainless steels in chloride-contaminated boric acid solutions could not be found in the open literature, the tests described in this report were carried out. It should be pointed out that these tests were conducted under only a limited range of conditions which approach those that could exist as a result of a design basis accident; no attempt was made to determine mechanisms of the corrosion reactions.

**EXPERIMENTAL PROCEDURES**

All of the tests described in this report were conducted in a recirculating stainless steel loop containing a chamber through which the test solution was sprayed and in large stainless steel vessels. The loop used for these studies has been previously described in detail\(^{(1)}\) and its description will not be repeated here. During the tests the spray chamber, which contained a Teflon-insulated stainless steel rack on which the test specimens were suspended, was maintained half full of solution so that half the specimens were exposed to the spray and half were submerged in the liquid. The test solutions were made by dissolving enough boric acid in
deionized water to give a boron concentration of 3000 ppm (0.28%). The room temperature pH of this solution is 4.5, and higher pH values were obtained by adding reagent grade sodium hydroxide. Sodium chloride was added to bring the chloride concentration to the desired level. In all cases an atmosphere of air filled the free volume of the loop at the time of closure.

Two different types of stressed stainless steel specimens were used. One type consisted of a 1/2-in. length of stainless steel tubing from which a section was removed to form a C. Holes were drilled near the ends of the C, and using a stud and nuts the ends of the C were pulled together until the stress on the outside surfaces was 2/3 of the yield stress as determined by an attached strain gauge. In each run two specimens of this type were exposed in the spray and two were totally immersed in the solution.

The second type of stressed specimen was a U-bend formed by bending strips of thin sheet in a simple bending machine and then pulling the legs parallel with a stainless steel tie bolt. Thus in these specimens the steel was both plastically and elastically deformed. The strips from which the U's were formed were 3 in. by 5/8 in. with a 1/4 in. hole near each end. The radius of curvature of the U's was 5/16 in. Some of the U-bends were formed with the steel in the mill-annealed condition, and others were formed from strips which had been subjected to different treatments. Part of the specimens were sensitized at 677°C (1250°F) for 1 hr followed by furnace cooling and about half of these was subsequently pickled in a HNO₃-HF solution to remove the thin oxide film formed during the heat treating. Other strips were butted together and welded with an automatic seam welder in an inert atmosphere. All of the welds were ground and some were pickled. In all cases where U-bends were formed the welds were at the bottom of the U, that is in a region of maximum deformation. Part of the welded specimens were not formed into U's and these were exposed as straight strips with no applied stress. Double U-bends were also exposed to the test solutions. These were formed by bending two strips at the same time and using a single tie-bolt to draw the legs parallel. With this type of specimen a crevice existed on the compression side of one of the U-bends and on the tensile side of the other.
Only types 304 and 316 U-bend specimens were tested in this program. All of the U-bend specimens were prepared from the same sheets of the two alloys. The 304 sheet was 0.019 in. thick and the 316 sheet was 0.031 in. thick. The chemical analyses of the two steels are shown in Table 1. In each case 4 specimens of each alloy in each condition were exposed in the spray and 4 were submerged in the solution for the first part of the test.

Table 1. Composition of the Stainless Steel U-Bends

<table>
<thead>
<tr>
<th>Stainless Steel</th>
<th>Composition, weight percent</th>
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<tbody>
<tr>
<td></td>
<td>Cr</td>
</tr>
<tr>
<td>304</td>
<td>18.2</td>
</tr>
<tr>
<td>316</td>
<td>17.5</td>
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Also exposed with the stressed specimens were butt welded specimens of 304 and 316 pipe. The butt welds were made using type 308L welding rod with inert gas shielding. No external stress was applied to these specimens. For the first part of the tests two specimens of each alloy were exposed in the spray and two were beneath the liquid level.

All specimens were exposed continuously in the spray loop for 24 hr at 141°C (285°F) and 7 days at 100°C (212°F). At the end of this time the specimens were removed from the loop and placed on a Teflon-insulated stainless steel rack which was placed in a large stainless steel vessel. All specimens—those exposed in the spray and those totally submerged—were completely covered with a solution of the same composition as that used in the loop. The container was covered and the temperature was maintained at 82°C (180°F) for 2 months after which the specimens were examined.

All stressed specimens were examined visually using 30X magnification. At the higher chloride ion concentrations most of the specimens had deposits of reddish brown corrosion products on at least part of the surface. Frequently, but not always, cracks were found under these deposits. After the first visual examination, the corrosion products were removed and the specimens were re-examined. The corrosion products were removed by immersion in concentrated phosphoric acid (85 wt. %) at
130°C (266°F). This treatment dissolved the corrosion products and caused only minor corrosion damage of the stainless steel during the short immersion time. Those specimens that appeared to be visually free of cracks were subjected to a fluorescent dye penetrant treatment (ZL-22, Magniflux Corp.) and examined under ultraviolet light for evidence of cracks or other penetrations into the surface. In addition to the above examinations a number of specimens was sectioned, polished, and examined metallographically either to determine the nature of the crack or to verify the presence or absence of a crack in doubtful cases.

RESULTS

The results obtained with the stressed specimens exposed to the 0.28M H₃BO₃ (pH = 4.5) containing different chloride ion concentrations are shown in Table 2. In a few cases only a single crack was found in a specimen, but usually several cracks were found on those specimens that underwent cracking. In the case of the double U-bend specimens, the appearance of a crack in either one of the U’s is indicated as a cracked specimen in Table 2. Cracks usually began at the edges of the specimen in the region of greatest deformation, that is near the bottom of the U’s, and propagated in a direction perpendicular to the longitudinal axis of the strip; in a few cases, however, the cracks appeared to propagate in a direction nearly parallel to the longitudinal axis of the strip.

Overall Table 2 shows that type 316 stainless steel was more resistant to cracking than type 304 stainless steel. Of the 240 stressed specimens of each alloy (excluding the welded and ground straight strips and the butt welded pipe) exposed under all conditions 121 of the type 304 stainless steel specimens developed cracks whereas only 54 of the type 316 specimens cracked. The data also show that cracking was more prevalent in the spray than in the solution; 104 specimens cracked in the spray and only 65 cracked in the solution. The tendency of specimens to crack increased as the chloride ion concentration increased from 5 to 50 ppm, but at the 200 ppm level the cracking tendency was about the same as at the 50 ppm level. One interesting feature of the data was the observation that the sensitized specimens of both alloys (not pickled) were substantially more susceptible
Table 2. The Stress Corrosion Cracking of Types 304 and 316 Stainless Steel in Chloride-Containing 0.28M H₃BO₃ (pH = 4.5)

Conditions: 24 hr at 141°C (285°F) and 7 days at 100°C (212°F) in spray loop; 2 months at 82°C (150°F) in storage container

<table>
<thead>
<tr>
<th>Specimen Type and Condition</th>
<th>304 SS</th>
<th>316 SS</th>
<th>304 SS</th>
<th>316 SS</th>
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<tbody>
<tr>
<td>In Spray Region Cl⁻ Conc. (ppm)</td>
<td>During Loop Cycle Cl⁻ Conc. (ppm)</td>
<td>Submerged During Loop Cycle Cl⁻ Conc. (ppm)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>U-Bend Specimens</td>
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<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Annealed</td>
<td>0</td>
<td>2</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>Annealed, Double</td>
<td>0</td>
<td>1</td>
<td>4</td>
<td>3</td>
</tr>
<tr>
<td>Sensitized</td>
<td>4</td>
<td>4</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>Sensitized, Pickled</td>
<td>0</td>
<td>2</td>
<td>4</td>
<td>3</td>
</tr>
<tr>
<td>Welded, Ground</td>
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<td>4</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>Welded, Ground, Pickled</td>
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<td>0</td>
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<td>3</td>
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<tr>
<td>Welded, Ground, Double</td>
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<td>4</td>
<td>4</td>
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<td>3</td>
<td>0</td>
</tr>
<tr>
<td>&quot;C&quot; Specimen (2/3 yield)</td>
<td>0</td>
<td>0</td>
<td>1</td>
<td>2</td>
</tr>
<tr>
<td>Butt Welded Pipe</td>
<td>0</td>
<td>0</td>
<td>1</td>
<td>0</td>
</tr>
</tbody>
</table>

* In each test 4 of each type of specimen were in the spray and 4 were submerged, except only 2 "C" specimens and 2 butt welded specimens were exposed in each location.
to cracking than were the specimens that were sensitized and pickled before test.

As expected the welded and ground straight strips, while not completely immune, showed less tendency to crack than did similar specimens that were formed into U's. The double U-bends formed from the welded specimens underwent cracking to about the same extent as the welded single U-bends.

Some of the "C" specimens stressed to only 2/3 of their yield stresses also cracked. These specimens were made from mill-annealed tubing and should probably be compared with the annealed U-bends. It is difficult to make a meaningful comparison, however, because only two "C" specimens could be exposed in the spray and solution in each test. In the case of the 304 stainless steel, the "C" specimens seemed to be less susceptible to cracking than the U-bends except at the highest chloride concentration where almost all of the specimens cracked. A similar comparison with 316 stainless steel shows that the annealed U-bends were completely resistant to cracking under all conditions, but at the highest chloride level two of the four "C's" developed small cracks. Of the butt welded pipe sections only one 304 specimen cracked.

At the 200 ppm-chloride concentration all specimens underwent relatively heavy pitting in addition to the cracking reported above. The number of pits appeared to be greater on the 304 than on the 316 specimens as would be expected. As the chloride concentration decreased the frequency of pitting also decreased. Some cracks appeared to originate from these pits but other cracks developed in areas free from any other form of attack. In all cases a relatively heavy buildup of corrosion products marked the areas of localized attack.

The nature of the cracking was predominately transgranular in all cases where major cracking was observed. Figure 1 shows some of the cracks observed in a U-bend of sensitized 304 (not pickled) stainless steel exposed to the solution containing 5 ppm chloride and Fig. 2 is an enlargement of the branching part of the larger crack in Fig. 1 showing its transgranular nature. Figure 3 shows a sensitized and pickled type 304 stainless steel specimen that was also exposed to the solution.
Fig. 1. Photomicrograph of a section through a sensitized type 304 stainless steel U-bend exposed to a solution containing 5 ppm chloride with a pH of 4.5.
Fig. 2. An enlargement of the fine branching cracks associated with the larger crack in Fig. 1 showing the transgranular nature of the cracks.
Fig. 3. Photomicrograph of a section through a sensitized and pickled type 304 stainless steel U-bend exposed to a solution containing 5 ppm chloride with a pH of 4.5.
containing 5 ppm chloride. The intergranular penetrations on the surface were not recorded as cracks because the attack was due to the pickling as shown by Fig. 4 which is a similarly pickled specimen which was not exposed to the boric acid solution. Figure 5 is a major crack found in a sensitized 316 stainless steel specimen exposed to the solution containing 200 ppm chloride, and Fig. 6 is an enlargement of some of the fine cracks showing them to be largely transgranular.

Only two tests were conducted in 0.28M H$_3$BO$_3$ with pH's other than 4.5. In both of these cases the sample array and exposure conditions were the same as in the tests at a pH of 4.5. In the first of these tests the pH of the solution was 7.5 and the chloride concentration was 200 ppm. Under these conditions all four type 304 stainless steel specimens that had been sensitized (not pickled) cracked in the spray and 3 of the 4 that were totally submerged developed cracks. The only other specimen that showed any evidence of cracking was one of the annealed double U-bends of type 304 stainless steel that was immersed in the solution. The others, including all of the 316 specimens, showed no evidence of cracking. Also at the higher pH there was no evidence of pitting. A comparison of these results with those in Table 2 shows that increasing the pH of the solution from 4.5 to 7.5 greatly reduced the ability of the solution to produce cracks.

In the second test the solution was adjusted to a pH of 6.5 and contained 20 ppm chloride and 2 ppm iodide. In this case cracks were found only in some of the welded U-bend specimens. A single welded, ground, and pickled U-bend of type 304 stainless steel cracked in the spray and two of those totally submerged cracked. One similar type 316 stainless steel U-bend cracked in the solution. Also a single welded and ground double type 316 stainless steel U-bend cracked in the spray. All other specimens were completely free of cracks or any other forms of localized attack. The cracks noted in the welded specimens were not readily visible under microscopic examination but became apparent when examined with the dye penetrant. In both alloys all of these cracks were located close to the welds and were of similar depth, about 5 mils. The cracks in no way resembled the typical branching cracks characteristic of
Fig. 4. Photomicrograph of a section through a sensitized and pickled type 304 stainless steel U-bend that was not exposed to the test solution.
Fig. 5. Photomicrograph of a section through sensitized type 316 stainless steel U-bend exposed to a solution containing 200 ppm chloride with a pH of 4.5.
Fig. 6. An enlargement of some of the fine branching cracks seen in Fig. 5 showing the transgranular nature of the cracks.
chloride-induced stress corrosion cracking. Not only were the tips of the cracks blunted, but also the cracks appeared to extend only a short distance across the surface of the specimen. Furthermore the cracks did not originate at the edges of the specimens as was typical of most other cracks observed in this study. Figure 7 shows a cross section through one of the cracks. In no other test was this type of cracking observed.

**DISCUSSION**

Some of the results obtained in this series of tests were more or less predictable and others were somewhat unexpected. The observations that cracking occurred more frequently the higher the chloride concentration, that cracking occurred mostly in the region of highest stress or greatest plastic deformation, and that type 316 stainless steel was generally more resistant to cracking than type 304 stainless steel were expected. Numerous investigators have made similar observations in different systems and these are conveniently summarized in Ref. 5. The effect of temperature was not specifically investigated in this study, but qualitatively it was found that most of the cracking occurred during the first 8 days of test when the temperatures were highest. Additional cracks may have originated and existing cracks may have propagated during the 2-month period at 82°C (180°F), but certainly most of the damage occurred at higher temperatures. The general observation that the likelihood of cracking increases with temperature is in agreement with the results of many investigators. It should be noted however that at least in some cases cracks in austenitic stainless steel have been reported to initiate and propagate at appreciable rates at temperatures as low as 74°C (165°F) with as little as 5 ppm chloride.

The present results, combined with those given previously, show rather clearly the effect of pH on the susceptibility of austenitic stainless steels to cracking in chloride-containing 0.28M H₃BO₃. Our tests have now been conducted under similar conditions with solutions having pH's of 4.5, 6.5, 7.5, and 9.3. No cracking was observed at a pH of 9.3, only occasional cracking was observed when the pH was 6.5 and 7.5, and prolific cracking was observed at the lowest pH. Although
Fig. 7. Photomicrograph of a typical crack found in welded U-bend specimens when solution contained 20 ppm iodide and had a pH of 6.5.
the chloride concentrations were not the same at all pH's, the conclusion
to be drawn is obvious: in the boric acid system the susceptibility to
cracking decreases markedly as the pH of the solution increases from 4.5
to 9.3. Based on data obtained in other systems\(^{(5)}\) the foregoing con-
cclusion was expected. That cracking was not observed when the pH was
9.3 should not be interpreted as complete freedom from cracking at the
high pH. Cracking of the austenitic stainless steels is always possible
when the temperature of the solution is ambient or above and chloride ions
and oxygen are present.

One of the unusual features of the results is that the sensitized
stainless steel specimens that were not pickled were considerably more
susceptible to cracking than those that were given a relatively heavy
pickle after sensitization. As indicated by Fig. 4 the pickling produced
substantial intergranular attack, and it was expected that cracking would
start readily from these penetrations. Table 2 indicates that this was
definitely not the case. To minimize the possibility of cracking in
water-cooled research and production reactors pickling of areas of
unstabilized stainless steel that may have been sensitized as a result
of welding or other fabricational procedures is avoided because of the
experience in the Savannah River Reactors.\(^{(6)}\) Several of the major
failures experienced in these reactors occurred in type 304 stainless
steel components that were heavily sensitized during fabrication and
subsequently heavily pickled to remove the last traces of oxidation
products from the surface. Why pickling of sensitized stainless steel
makes it less susceptible to cracking in boric acid solutions and more
susceptible to cracking in reactor waters cannot be answered at this time.

Another unexpected result was the complete absence of cracks originat-
ing from the identification numbers stamped on each specimen. This process
upsets the metal in the immediate vicinity of the numbers, and in tests in
other environments\(^{(7,8)}\) cracks frequently originated at the edges of the
numbers. No rational explanation can be given for the lack of cracking
in these locations in the present tests. In conformity with previous
experience,\(^{(7,8)}\) most cracks originated from the sheared edges of the
specimens.
In all cases except for the unusual cracks found in the vicinity of the welds when the pH was 6.5, cracking was predominately transgranular. The failures in the Savannah River Reactors that occurred in the sensitized type 304 stainless steel were intergranular in nature.\(^{(6)}\) Again no reasonable explanation can be given for the difference in the mode of cracking between the two systems. The cracks that formed near the welds in both types 304 and 316 stainless steel when the solution contained 20 ppm chloride and 2 ppm iodide were totally different from all the other cracks observed. In fact it is not certain that these should be classified as stress corrosion cracks; however, the absence of such cracks in the straight welded strips implies that stress was necessary for their development. Since this was the only test in which iodide was present, it is possible that the iodide ion played some part in the development of these cracks. The general shape of the cracks and the fact that the extent of penetration was only about 5 mils during more than 2 months of exposure indicate that failures in the heat removal components of a reactor system would be unlikely from this type of attack.

Absolute freedom from failure of any complex system such as a spray system can never be guaranteed, but by proper design, fabrication, and control of the corrosive environment the probability of failure can be significantly reduced. As indicated by Table 2 the likelihood of cracking is less the lower the stress level, and sensitization significantly increases the probability of cracking. The stress level is controllable, at least to a certain extent, by proper design, and the problem of sensitization around welds could be eliminated by using a stabilized stainless steel such as 304ELC or 316 ELC. Even with types 304 and 316 stainless steel the degree of sensitization around welds can be minimized by proper welding procedures.

The data obtained in this study indicate that the best way to minimize cracking besides the complete elimination of chloride ions (which may be impossible) is to use a spray solution which has a high pH. If the overall containment system contains large amounts of aluminum, it may not be practical to operate the spray with a solution pH of 9 or higher where the probability of cracking would be very low. However, in such a case the
adjustment of the solution pH to 6.5 to 7.5 after spraying of the containment building has ceased may represent a practical approach. In this pH range the probability of cracking is low and the corrosion rates of aluminum alloys also are acceptably low. Since chloride ions are such universal contaminants, it seems likely that any spray solution will always contain some chloride ions. However, by using reagents and water with low chloride concentrations, and by proper standards of housekeeping, it should be possible to maintain a very low concentration of chloride ions, and consequently have a low probability of stress corrosion cracking failure.

The other type of cracking that could possibly cause failure of stainless steel components in a spray system is that caused by the presence of fluoride ions. It has been reported that in alkaline or neutral solutions as little as 1 ppm of fluoride is capable of producing cracks in heavily sensitized stainless steels. Cracking occurred even at room temperature but was faster at 82°C (180°F). However, cracking was not noted in heat-affected zones adjacent to welds on type 304 stainless steel, apparently because the welding process did not produce the necessary degree of sensitization. Hence, the reported data indicate that fluoride-induced cracking in a spray system should not be a problem unless stainless steel components are sensitized as could happen during a stress-relief treatment at high enough temperature (800 to 1500°F).
References


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