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## Hydrogen Embrittlement of Tantalum

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# Hydrogen Embrittlement of Tantalum

Examination of the corrosion literature on tantalum would suggest that one of the prime defects in its resistance to corrosion in aqueous media arises from its susceptibility to hydrogen embrittlement under some conditions. However, it should not be inferred from the data that tantalum has a greater inherent tendency for hydrogen embrittlement than most other metals.

On the contrary, this is not generally true. Instead, the remarkable corrosion resistance of tantalum in most corrosive environments causes it to be used under conditions that are far too aggressive for nearly any other metal.

Under sufficiently aggressive conditions, tantalum can be susceptible to hydrogen embrittlement, such as in corrosion tests on specimens in sealed capsules, even when the corrosion rate is extremely small. In this section of the report, some conditions are cited where such embrittlement has been observed or encountered. Methods that have been proposed for minimizing or eliminating hydrogen embrittlement of tantalum are described.

Hampei (Reference 3) has stated that hydrogen when present in the tantalum is dissolved in interstitial solid solution or is present as an interstitial compound, and that expands the metal lattice, inducing stresses that cause the metal to become brittle. Hydrogen embrittled tantalum may be restored to a ductile condition by proper vacuum annealing; although this can readily be applied to samples, this is not always practical for commercial applications, especially with large equipment.

It is well known that hydrogen gas can be absorbed readily by tantalum at high temperatures; this technique is used to prepare some grades of tantalum powders for capacitor applications. Under some special conditions, tantalum can even become embrittled when exposed at room temperature to gaseous molecular hydrogen.

Apparently this requires special circumstances and occurs only when the tantalum is being mechanically deformed during the exposure, and also the presence of dissolved oxygen in the tantalum is necessary for the phenomenon to be observed.

According to Taylor (Reference 7), tantalum can absorb hydrogen to the extent of about 700 volumes to form the glass hard hydride, but begins to lose its ductility when only a few volumes have been taken up.

It begins to absorb *molecular* hydrogen at about 250°C, but readily takes up *atomic* hydrogen at room temperature. However, since the penetration rate is low at the lower temperatures, the exposed surfaces may become very brittle, while the interior of a section remains ductile. For this reason, an analysis of heavy sections of hydrogen may be somewhat misleading.

If tantalum containing hydrogen is heated to about 1200°C in a good vacuum, all of the hydrogen is removed and the metal becomes ductile.

Since tantalum is vulnerable to hydrogen embrittlement, it must be protected from possible hydrogen absorption.

It should not be exposed to molecular hydrogen above 250°C, nor to atomic hydrogen at any temperature.

Atomic hydrogen is liberated on the surface of tantalum when:

1. The metal is dissolved in hydrofluoric acid, thus liberating hydrogen.
- 2 . Some other metal which is connected to the tantalum is being dissolved chemically. (In this case, tantalum the cathode in an electrolytic cell.)
- 3 . Due to the process of a stray voltage, the tantalum becomes a cathode in the system. Note that the presence of stray currents can result from induction from adjacent lines, from leakages, from variable ground voltages, etc. It should be kept in mind that stray voltages may be intermittent or transient, but the effect on the tantalum is cumulative.

The protection of tantalum equipment from exposure to atomic hydrogen is most important:

1. The metal should not be exposed in service to hydrofluoric acid in any strength at any temperature.
2. Whenever there is a possibility of cell action due to chemical attack on another metal, the tantalum should be insulated from all other metals.
3. When stray voltages are known to exist or are even a possibility, and as a precaution in all cases whenever possible, the tantalum should be completely insulated from all other metals.
4. As a means of additional protection the insulated tantalum is sometimes connected to the positive pole of a DC source (about 15 volts) while the negative pole is connected to some other metallic part which is exposed to the solution in the vessel or to ground. Obviously, this is not always possible in electrolytes that attack tantalum, or for other reasons, but has been used successfully in some installations.
5. Essentially all of the hydrogen embrittlement in aqueous environments that has been reported on tantalum has been observed in laboratory tests in which specimens were exposed in sealed containers.

As reported in Reference 1, tantalum became embrittled in sealed container corrosion tests in concentrated hydrochloric acid at 190°C.

Further detailed tests described in Reference 1 show that tantalum is not embrittled by hydrogen when exposed to boiling hydrochloric acid in concentrations of 20% or less. At 190°C, tantalum is resistant to hydrogen embrittlement in concentration up to about 30% hydrochloric acid. Above this acid concentration, it became embrittled rapidly at high temperatures in the enclosed capsule tests. For these tests, the samples were sealed in heavy-wall Pyrex glass tubes containing the acid, and were inserted into an autoclave heated to the appropriate temperature. Tantalum from a variety of commercial sources was all found to be similarly embrittled by the exposure in concentrated hydrochloric acid at 190°C. The evidence of embrittlement was shown by a bend test on the samples. Also shown was that a variety of alloy additions to tantalum did not significantly influence its resistance or sensitivity toward hydrogen embrittlement in this environment under these conditions. All samples of the various materials became brittle after 66 to 90 hours exposure.

Various heat treatments have been suggested as a method of controlling embrittlement. However, materials from a variety of commercial sources given various heat treatments in vacuum showed no significant differences in their susceptibility to hydrogen embrittlement.

Also at 190°C, but in boiling nitric acid, tantalum showed excellent resistance to corrosion and did not exhibit embrittlement by hydrogen.

Also at a temperature of 190°C, tantalum corrodes at a rate of less than 1 mil per year in all concentrations of sulphuric acid, whereas in various boiling sulphuric acid solutions, where the boiling point of the acid increases rapidly with concentration, tantalum became embrittled when exposed in sealed containers to concentrations of sulphuric acid greater than 90%, and corroded at relatively high rates.

There are three general approaches that might be applied to the prevention of hydrogen embrittlement of tantalum:

1. Modify the metallurgical structure so that the effect of hydrogen in the lattice would be less detrimental. The above cited work on heat treatments and alloy modifications indicate that this has not proven to be a simple solution to the problem, and major alloy modifications might alter the other desirable corrosion resistance properties of tantalum.

2. Prevent hydrogen from entering the metal by some surface treatment. One classical technique is to use minor amounts of noble metals on the surface; another is by electrolytic anodizing, which is applied in tantalum capacitor technology. These two techniques are discussed in more detail below.

The first of the surface treatment techniques involves contacting the tantalum with another metallic element that has a low hydrogen over voltage and that is electrochemically cathodic to tantalum in the same environment. In electrolytes that do not significantly attack the tantalum oxide surface layer, tantalum appears to occupy a position in the electromotive series close to platinum and gold. It assumes a negative polarity when in contact with all metals above platinum and gold in the series. Only the noble metals generally meet the above overvoltage and electrochemically cathodic requirements, but under some circumstances some other elements might perform similarly. Reference 1 points out that remarkably small areas of platinum, palladium, gold, iridium, rhodium, osmium, rhenium, and ruthenium were effective laboratory tests.

Contact of the noble metal with the tantalum can be made by riveting or welding a small spot of noble metal onto the surface, or by electrode position of the noble metal on the surface, or even by rubbing noble metal powder onto the tantalum surface. In the case of exposure to hydrochloric acid, small concentrations of the chloride salts of the noble metals can be added to the solution and provide an effective way to prevent embrittlement in laboratory tests. The last named method, the addition of the chloride salt, may or may not be permissible or allowable in the commercial system.

Very small areas of low-overvoltage elements are effective in preventing hydrogen embrittlement of tantalum (Reference 1). Data on the ratio of the surface area of the noble metal to that of the tantalum show that a specific minimum ratio is necessary for protection, where platinum or palladium was used as that contact material. Platinum appears to be the preferred noble metal for protection of tantalum against hydrogen embrittlement because of its particularly low hydrogen over-voltage and the general excellent corrosion resistance of platinum itself. In all tests at 190 and 250°C, platinum was effective in preventing the hydrogen embrittlement of tantalum.

Exposure at 190°C in concentrated hydrochloric acid for over 1,000 hours when the surface area ratio of platinum to tantalum was only 1 to 9,500 did not cause damage to the tantalum. Under similar tests conducted at 250°C, no embrittlement of the tantalum, protected by this surface area ratio of platinum, was observed after 200 hours. At this temperature, a longer test did not appear to be reasonable since the corrosion rate of tantalum has become rather high.

In the laboratory tests in concentrated hydrochloric acid, the specific geometry or location of the platinum did not appear to be particularly critical. For example, Reference 1 showed that a spot of

platinum at one end of a tantalum strip protected the other end which was six inches away. However, the maximum distance that platinum can be located from the tantalum in chemical process equipment and still provide protection from hydrogen embrittlement still needs to be established.

The corrosion rate of tantalum was generally reduced by contact with platinum. An unexpected observation was even small traces of platinum in contact with tantalum were effective over a long time, even though the corrosion resistance of platinum is not considered good in concentrated hydrochloric acid at high temperatures. Corrosion tests in which the various low-overvoltage noble were either coupled or not coupled to tantalum show the reason for this unexpected behavior. Both platinum and palladium by themselves corroded at relatively high rates in concentrated hydrochloric acid at 190°C, but when they were in contact with tantalum, the corrosion rate on the noble metal was negligible; thus both tantalum and platinum are mutually benefitted by galvanic contact.

Palladium, although it exhibits a somewhat higher hydrogen overvoltage than platinum while still being a low-overvoltage element, it is of interest because its lower cost could be important in commercial applications.

However, palladium is not as effective as platinum for protecting tantalum and requires a larger contact area to provide adequate protection.

Reference 1 suggests the effectiveness of platinum and palladium in preventing hydrogen embrittlement of tantalum exposed to 85% sulphuric acid at 238°C in sealed capsules. Contact of the tantalum with platinum reduced the corrosion rate and prevented hydrogen embrittlement during an exposure of over 550 hours when the area ratio of platinum to tantalum was only 1 to 1590. Although palladium also showed some beneficial effect in preventing hydrogen embrittlement of tantalum, the corrosion resistance of palladium in this environment is too poor for practical considerations.

Under some commercial operating conditions, tantalum also can become embrittled by hydrogen by being subjected to stray cathodic currents. Reference 1 cites, for example, that tantalum plugs are often used to seal holes in glass-lined vessels. If during use, an additional hole is produced in the glass coating, a galvanic couple is produced between the tantalum plug and the steel shell of the structure. The current that flows in this couple is a function of the specific geometry of the system and of the conductivity of the environment. Reference 1 presents data showing the influence of contact with platinum and palladium on tantalum that has been made cathodic in sulphuric acid at current densities of 1 and 10 ma/cm<sup>2</sup>. The latter current density is considerably higher than one would expect to find in actual operating conditions. These data show again that platinum is more effective than palladium in preventing hydrogen embrittlement of tantalum.

Even at this high current density, an area ratio of platinum to tantalum of 1 to 15 prevented embrittlement in a two-hour test.

Some practical commercial applications might be expected to be developed from the laboratory experimental data cited above in which strips of platinum might be used to reduce hydrogen embrittlement of tantalum chemical process equipment in some severe chemical applications.

A technique that is used in electrical testing of tantalum capacitor powders to prevent hydrogen embrittlement of the tantalum sheet cathode, when used, is to anodize the tantalum sheet to 20 volts.

Thickness of the tantalum oxide film formed on tantalum at various voltages are listed in Table XXII.

This possibility should be investigated further since, providing the oxide film could be maintained on the tantalum at all times during service, it could provide a low cost method to provide improved resistance of the tantalum to severe environments.

3 . A third way to prevent hydrogen embrittlement of tantalum is to completely electrically insulate tantalum from all metals, as cited under Paragraph 6: "Galvanic Effects," in Section VII of this report.

Reference 97 describes electrical insulation methods for various types of tantalum equipment, including bayonet heaters, coil and "U" type heaters, and shell and tube-type heat exchangers.

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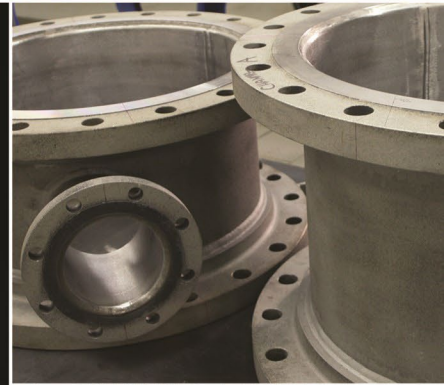
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