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CORROSION RESEARCH - TUBALLOY AND ALLOYS

(First Progress Report on Problem No. 391-ML-54-4e; file Serial 22)

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SUMMARY

Problem under Investigation:

The purpose of the investigation is to gather fundamental information on the corrosion of tuballoy, with the aim of using the uncoated metal or an alloy of it in an atomic power plant. The present study is limited in scope to obtaining corrosion information about the metal and its promising alloys in water solutions.

Results:

Corrosion rates have been determined for ^uthe metal in static distilled water and in $10^{-5}N$ H_2O_2 at temperatures from room temperature to 90°C. Rates increase normally with temperature except that there is a 20-fold increase in a ten-degree rise between 70 and 80° in water, and between 80 and 90° in $1 \times 10^{-5}N$ H_2O_2 . Rates below this sharp rise are probably not too serious, none exceeding 0.5 mil/mo.

Corrosion rates of ^{Al, Be, Cu, Co, Pb, Si, Sn, Ni, and Sb} alloys of ^uthe were determined. For alloys of the most promising metal, ^{Ni}nickel, rates were measured in water and dilute peroxide-chloride mixtures at temperatures from 40 to 90°C.

A marked increase in corrosion rate was noticed when the tests were run in a closed vessel, with no solution refreshment. In this system, the effects of surface preparation, surface oxide, pH and concentration of other ions in the solution were measured. Some ideas of the galvanic effects of coupling to metals such as stainless steel and ~~aluminum~~ were also obtained.

Recommendations:

This is an interim report and no recommendations will be made that refer to the construction of a pile using uncoated metal. It is felt that a valuable bit of information could be obtained from corrosion experiments in the presence of different dissolved gases, hydrogen and oxygen being considered as the two most important. The Discussion of Results in this report considers the reasons for the proposed experiments.

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GENERAL DISCUSSION

Origin of Investigation:

When the possibility of a water-cooled Pile became certain, it was immediately of interest to know the corrosion behavior of the metal. If one could show corrosion not to be serious in water under a certain set of conditions, then it would only be necessary to find a source of suitable water, and a pile could be operated with no coating on the metal itself. It would be a further requirement that the water be present in quantity such that radiation from the fission products picked up would not too seriously pollute the water after being mixed with that returned from the pile. Materials other than water also have been considered.

If some alloy or alloys of tuballoy were more corrosion resistant than the metal itself, then it might be possible to operate a pile with less restrictions on the composition and/or temperature, of the cooling water. Either the alloy itself, if the alloying elements were present in small enough amounts, or pure metal suitably coated with an alloy might be used.

Consequently work was started here and on allied projects to determine the corrosion rate of tuballoy in air, water, and other media (1-8). To simulate the effect of radiation on the water, hydrogen peroxide was frequently added to the testing solutions.

The water corrosion program in this group was expanded to cover some alloys when they became available.

PRESENT CONDITIONS

Present Operational Plans:

For the first three water-cooled plants now under construction the specifications call for aluminum cans to be used over "slugs" of tuballoy and information on the corrosion rate of the metal is valuable only in helping predict the danger due to a puncture of the coating used.

Causes Prompting Investigation:

The interest in the problem is in possible future designs of water cooled piles. Since the application to the first plants is very slight, the tuballoy corrosion studies have been assigned a relatively low priority, B-1.

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INVESTIGATION

Course Pursued in Study:

Corrosion rates were determined for tuballoy in distilled water, in 10⁻⁵N Hydrogen peroxide at temperatures ranging from room temperature to 90°C, for various alloys at 90°C, and for tuballoy-nickel alloys containing up to 10 atom percent nickel in water and dilute hydrogen peroxide-chloride ion mixtures at 40, 70, 80, and 90°C.

All tests were run without rapid motion of the solution. In most cases, 400 ml beakers were used, heated from below by electrical coil heaters similar to hot plates. The solutions were continually replenished with the replenishing rate being sufficient to keep the concentration of hydrogen peroxide in the beaker the same as the replenishing solution. The temperatures were controlled by suitably regulating the voltage supplied the heaters by means of Varitrans, small auto-transformers. In these tests the only mixing given the solutions was the normal thermal agitation from heating at the bottom, and the mechanical action of the small streams of replenishing solutions. The replenishing flow-rates varied from about 10 cc/min. for distilled water at non-corrosive temperatures up to ca 40 cc/min. when decomposition of H₂O₂ was rapid, as at 90°C.

The standard treatment given the samples consisted of grinding to a smooth white metallic surface, using a medium fine grit belt. The grinding was done fairly slowly, with frequent cooling of the samples. They were then cleaned in alcohol or ether, dried and weighed. For the standard tests on pure metal, on various alloys, and some of the nickel alloys, this initial treatment as supplemented by a 2-5 minute dip in 25% nitric acid at room temperature. There was no noticeable difference in result from omitting the acid dip at this point. After removal from the test, usually of two to four days duration, the loose oxide was washed off the samples and they were then immersed in 25% nitric acid for 2-5 minutes to remove the remaining oxide. After washing first in water, then in alcohol or ether, the samples were dried in vacuum, or simply allowed to dry in air, and weighed.

The surface preparation aimed at giving a clean and smooth surface to facilitate area measurement and to remove the old oxide film. No significant difference in the corrosion rate was noticed due to the grade of grinding belt, although small effects would not be picked up in these tests. Likewise the use of special cleaning procedures such as anodization did not give greater reproducibility.

After 5 minutes in 25% nitric acid at room temperature a sample of the size used (about 15 cm² in area) shows no detectable change in weight. After 30 minutes the weight loss usually is 0.2-.3 milligrams. The accuracy of the weight determination is 0.1 mg.

Some special tests were run in closed systems with provision for measuring the amount of hydrogen liberated in the corrosion reaction, for a check on the weight-change measurements and a possible alternative measure of corrosion rate. Effects of certain variable influences on corrosion rate also were determined. The apparatus is sketched in Figure 1. The hydrogen was trapped in the gas collecting tube, periodically removed by opening the stopcock and its volume measured in a gas burette.

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Figure 1.

APPARATUS FOR SPECIAL CORROSION TESTS ON TUBALLOY

Data and Calculated Results:

As corrosion proceeded, in nearly all tests the surface of the metal became covered with a loose coat of black to brownish oxide. In the initial stage of forming this loose coat, it appeared to be quite adherent, but since the oxide occupied approximately three times the volume of the metal replaced, the coating soon became loose in those cases where the corrosion rate was appreciable. In the initial state of forming the film, the surface sometimes had a bluish or brown appearance in water, while the film formed in air was brown. The brownish black oxide which formed the loose coat was probably substantially UO_2 , and when peroxide was added to the solution a yellow higher oxide, probably UO_3 , was formed to some extent at the outer surface of the film. As is the case with iron rust, probably all stages of oxidation can be found, from the lowest state near the metal surface, to the highest state on the outside of the coat. It would not be expected that any treatment which would form a protective film on the metal would give any protective effect after the loose type of coating begins to form.

Tests in Open Beakers: No attempt was made to control the pH of the solutions. Measured values were all in the range 5.7-6.4. Results for Tu are given in monthly reports CT 610, p³¹-2; CT 687, p²⁴-5; CT 892, p¹². The tests reported in CT 610 and 687 were preliminary in nature and the results are not accepted as the best values of corrosion rate. The number of samples used was small, and the control of conditions was not too good. In these cases the value taken for the corrosion rate was the slope of the weight change against time curve. The samples were regularly weighed about every other day, without any attempt to remove the oxide adhering to the surface. When the weight-time curves became essentially linear for an appreciable length of time, the slopes were taken as the corrosion rates for the metal. The periodic drying of the surface oxide can markedly influence the corrosion rate under certain conditions as will be shown later, but in the cases here mentioned rates thus obtained compared reasonably well with those obtained under more carefully regulated conditions, given in Table I.

In a preliminary experiment on the action of fairly strong (about 0.007 N.) hydrogen peroxide on tuballoy at about 96°C, the rate for the first thirty hours was between 0.3 and 0.6 and in the next eight hours it

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rose to between 1.3 and 1.5 which is much closer to the value that would be expected at 96° from runs at lower temperatures. For the first ten hours only the yellow oxide was noticed on the samples; after 30 and 38 hours some black oxide was also visible, indicating that toward the end of the experiment the oxide was forming so rapidly that all of it was not oxidized to the higher yellow oxide. The slow rate at the start of the experiment may have been due to: (1) the formation of some type of more or less adherent protecting layer by immersion in peroxide of this strength, or (2) the weight loss in the first period being smaller because the samples were not cleaned in acid to remove all the oxide before weighing. This results in the weight loss for the first period being too small by the amount of oxide remaining on the sample at the end of the period.

Table I presents the best data in water and $10^{-5}N$ H_2O_2 . They are slightly changed from those in CT 892 by having some new determination of corrosion rate averaged into the older figures. The results were erratic in some cases and some idea of this effect may be gained from the range of values obtained listed in the parenthesis following the average value for each temperature. It may be seen that from room temperature up through 70°C, the corrosion rate goes up slowly in both solutions. However, there is a sharp break, an increase by a factor of twenty in a ten degree rise, occurring between 70 and 80° in distilled water and between 80 and 90° in $10^{-5}N$ hydrogen peroxide solution. Between 80 and 90° in water, the continued increase in rate is a more normal one again.

Alloys of various metals with tuballoy were prepared by Mr. Foote's group from commercial Tu, and their corrosion rates were determined in the standard manner in this group. Preliminary tests were reported in MUC-JED No.s 1, 9 (N-365), and 13. The complete values on all but the nickel alloys are given in table II. It was found that only the nickel alloys showed promising corrosion resistance to hot distilled water and dilute H_2O_2 solution.

Table I.

CORROSION RATE OF TUBALLOY IN WATER AND
IN DILUTE HYDROGEN PEROXIDE - pH 6.0-6.5

Temp. °C	Weight Loss, mg/cm ² /hr**	
	In distilled water	In $1 \times 10^{-5}N$ H_2O_2 (Superoxide added to distilled water.)
90	0.7 (.4 - .8)*	0.6 (.06-1.0)
80	.2 (.05- .4)	.03 (.02- .05).
70	.012 (.006 - .029)	.015 (.009-.022)
60	.011 (.002-.023)	.007 (.005-.010)
50	.005 (.001-.012)	.005 (.004-.010)
Room Temp (35-38)	.0011 (.0005-.0022)	.0020 (.0015-.0025)

** 1 mg/cm²/hr is equivalent to a uniform surface loss of .015"/yr.

* The values in parentheses give the range of results of experimental determinations.

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Table 11

CORROSION OF LOW PERCENTAGE ALLOYS OF TUBALLOY AT 90°C.

Sample No.	Alloying Element and its atomic percent in alloy	Corrosion Rate, mg/cm ² /hr Loss	
		in 4 x 10 ⁻⁵ N H ₂ O ₂ pH = ca. 6.0	in 3 x 10 ⁻⁵ N H ₂ O ₂ 2 ppm Cl ⁻
274	Cu - 1/4	0.3 (.08-.57)	0.58 (.48-.62)
273	1/2	.23 (.14-.30)	.3 (.08-.44)
215	1	.4 (.11-.65)	.9 (.5-1.5)
218	5	.2 (.10-.40)	.4 (.13-.62)
220	10	.2 (.06-.29)	.42 (.26-.56)
227	95	Sample swelled with wt. gain, then broke up	Sample swelled & broke up
219	99	.007(.004-.012)	.007(.002-.017)
265	Sn - 1/4	0.2 (.07-.29)	0.46 (.32 - .67)
262	1/2	.9 (.3-1.3)	1.4 (.8 - 2.2)
260	1	Sample broke up	Sample broke up
264	5	Sample broke up	Sample broke up
260	10	Disintegrated rapidly	Disintegrated rapidly
270	Cr - 1/4	0.6 (.16-.85)	0.8 (.2-1.4)
269	1/2	.3 (.11-.43)	.4 (.18-.52)
211	1	.7 (.55-.78)	.6 1 det.
214	5	.31 2 det.	.36 2 det.
210	10	.08 (.03-.13)	.5 2 det.
209	15	.06 (.04-.08)	.22 2 det.
228	20	.02 (.01-.03)	.22 2 det.
268	50	.03 (.02-.05)	.04(.03-.06)
293	Si - 1/4	0.24 (.18-.36)	0.51 (.46-.56)
292	1/2	.8 (.59-.87)	.9 (.6 -1.3)
277	1	.7 (.45-.93)	.8 (.5 -1.4)
276	5	.57 (.52-.60)	.3 2 det.
273	10	.34 2 det.	-----
242	Al - 1/8	.42 (.38-.51)	0.4 (.18-.73)
243	1/4	.2 (.07-.48)	-----
241	1	.48 (.40-.54)	-----
239	5	Broke up-intergranular attack	Broke up-intergranular attack
232	Be 1/4	0.91 (.82-1.0)	0.5 2 det.
231	1/2	.3 (.09-.55)	.8 (.4-1.2)
229	5	.50 (.41-.55)	.5 2 det.
230	10	.80 (.66-.89)	.56 2 det.
297	Sb - 10	1.5 (1.4-1.6)	2.0 (0.4-3.6)
244	Pb - 1	Lose wt. rapidly, went to pieces	
246	5	Mitto	

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Some other alloys, e.g. aluminum, tin, copper, showed lower corrosion rates than pure metal when present in only a small amount, (between 0 and 1 atom %) but the effect did not continue with the addition of more of the alloying element so that the corrosion rate increased again.

With the favorable effect of adding nickel to the alloy known, corrosion rates were determined for alloys up to 10 atom % Ni in water and in various peroxide-chloride solutions, not exceeding 10^{-3} N H_2O_2 or 2 ppm Cl^- , at temperatures from 40 to 90°C. The results are given in tables and figures, showing the variation in corrosion rate with the amount of nickel added to the alloy, in MUC-JED #28 (N 846), which please see. In general, good corrosion inhibition is afforded by the addition of small amounts of nickel to the alloy at 90, 80 and 70°C. The corrosion rate is reduced by a factor of 10 at 90°C, and by a progressively lower factor with lowering temperature. At 40° there seems to be increased corrosion caused by the addition of nickel to the alloy, but the effect may not be real because the unalloyed Tu used was different from that used in making the alloys. Table I of this report, however, shows that in water and 10^{-5} N H_2O_2 the expected rate for ordinary metal is not over ca. 005 mg/cm²/hr, even up to 50°C. The values for 2½ to 10% Ni alloy at 40°C were .007 to .010 mg/cm²/hr.

Special Tests: This work was done by Hoxeng and Rebol in this group and is reported in CT 687, p 27-31; CT 752, p. 22-6; CT 817, p. 20-21; and CT 892, p. 16-18. Please see these reports for full descriptions and data from the tests.

Amount of Gas Evolved: If, in the corrosion of tuballoy, the conditions are such that only the lower oxide TuO_2 is formed, 0.188 ml. of hydrogen will be liberated for each milligram of metal corroded. For all determined values (29 in number) of the ratio of ml of H_2 evolved to mg of metal corroded at 70° which were considered as "good values", i.e. not having some obvious complicating factor, there was an average deviation of about 7% from that value. CT 687 and 752.

Condition of Metal: The need for knowledge as to the effect of purity of the metal on the corrosion rate was recognized early, and some work was undertaken to corrode metal other than the standard commercial material. Metal which had been kept carbide free by casting in a magnesia crucible had a corrosion rate in water only about one-half as high as metal cast in graphite, but no further work was done. The indication is not too certain due to irregular results. CT 752.

The following experiment was interpreted as showing the effect of grinding.

Type of belt used in grinder	Rate of wt. loss in dist. H_2O at 70°C, mg/cm ² /hr	Initial potential in water, V. neg. to 0.1N calomel electro
fine	.321	.351
medium	.463	.498
coarse	.403	.402

The difference appears clear but should be corroborated to make sure that it is not caused by inherent differences in metal samples. CT 752.

The best method of preparing the surface of the sample so as to get uniform results was found to be surface grinding on a coarse alundum belt, followed by anodization in concentrated sulfuric acid and de-passivation in 25% nitric acid to remove the anodic film of oxide. CT 817. When samples were dried on a hot plate at 100°C and then returned to water at 70°C there was no gas evolution for three days. Samples dried at room temperature in a stream of dry air gave no gas for a period of several hours up to as much as one day after being placed back in water. The actual weight loss was retarded somewhat in these cases, but in some instances the hydrogen evolution was retarded to a greater degree, possibly indicating some depolarization effect. CT 687 and 752.

Galvanic Effects: Tuballoy is generally anodic to stainless steel when coupled but it does not corrode so badly as when it is coupled to aluminum. In the latter case the aluminum generally stays anodic for the first 50-75 hours, and the tuballoy loses weight at up to one and one third times the usual rate. The weight loss of the couple is intensified several fold if the aluminum is brazed to the tuballoy with Al-Si or Zn-Sn, probably because of better electrical contact. The triple couple thus formed might also play some part in the effect. CT 687, 817, 892.

The Corroding Solution: At 70°C, the corrosion rate found was 0.18 (.15-.40) mg/cm²/hr in distilled water and 0.28 mg/cm²/hr in genuine W water. The difference could have been caused by different pH or the presence of other ions and materials in the W water, probably the latter. At 50°, the rate in W water was found to be 0.06 mg/cm²/hr. CT 817.

Adding sulfuric acid reduced the corrosion rate of the metal. To determine whether this was due to pH or anion concentration, corrosion was measured in sulfuric and phosphoric acid solutions, with constant sulfate in one case and constant phosphate in the other, both at varying pH. It was found that at relatively high sulfate (695 ppm) and phosphate (167 ppm) concentrations the corrosion rates at 70° were essentially independent of the pH from 2 to ca. 5.5. The values found were 0.082 and .18 mg/cm²/hr, respectively. This would indicate a considerable reduction in corrosion rate by the addition of these anions. CT 892.

RESULTS OF INVESTIGATION

Discussion:

Good results on the corrosion of tuballoy are very difficult to obtain because of the metal used. Although it is quite pure by ordinary standards, the chief impurity causes a considerable amount of inhomogeneity. The presence of carbon in the melted metal causes the formation of crystalline tuballoy carbide in the final product. This invariably results in corrosion samples, the surfaces of which vary widely from piece to piece, giving rise to serious differences in corrosion behavior. It is not even possible to get really reproducible results from one piece of metal because in resurfacing the sample, new inhomogeneities are exposed and old ones erased.

Because of this difficulty the data from beaker tests are frequently rounded off to only one significant figure, while the measurements usually are good to three. The figures in table I are nearly all averages of a considerable number of determinations. From 70 through 90°, for example, the number of determinations for each condition ranges from 9 to 18.

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As an interesting check to the conclusion that non-uniformity in the metal or other unreproducible corrosion behavior causes the poor check and not experimental error, one can calculate the percent relative probable error according to the method of Mears and Daniels (9). If the relative probable error is $N\%$, it means that there is a 50% chance that the true corrosion rate is within $N\%$ of the reported average value. The probable error is made up of two components: (1) an experimental error, which should be constant regardless of the value of corrosion rate, and (2) an error due to the metal corroded which should vary with the corrosion rate. Now if the relative probable error is constant for a series of widely different corrosion rate values, it means that the experimental error is unimportant, since it would cause a larger per cent error in small values than in large ones. A rather constant relative probable error, from 5 to 8% was found for the corrosion rates in H_2O_2 , table I. It is therefore concluded that in these cases experimental error is negligible compared to differences inherent in the metal itself. The treatment does not lead to a similar conclusion for the values in water, (no H_2O_2) perhaps because of the smaller number of determinations at the lower temperatures, where the relative probable error is larger than at the higher temperatures.

The very large difference in corrosion behavior between the open beaker tests and the closed, hydrogen collecting tests is startling. The only factors known to be different are (a) the solutions in beakers are saturated with air, while those in special tests are saturated with hydrogen; (b) the pH goes up during a special test and (c) there must be built up at least a very small concentration of uranium ions in the solution during the special tests. Factor (b) has been shown to be unimportant to the order of magnitude involved, if not non-existent. It is hard to see how factor (c) could cause an increase in corrosion rate. It would be expected to decrease the rate to some extent and it would not have been surprising if such an effect were noticed; instead the rate increased by a factor of 32 over that in beaker tests.

We are therefore led to the opinion that the gases dissolved in the solutions are the cause of the difference found. The two influences which are proposed are that (1) oxygen is responsible for keeping a protecting type of oxide film on the surface of the metal in the beaker tests and clearly cannot when hydrogen has removed it from solution, and (2) nascent or molecular hydrogen plays an unknown but important role in the corrosion of the metal. In the special tests hydrogen liberated at the corrosive surface would be able to influence the corrosion much more than in the beaker tests, where it probably would react very quickly with oxygen in the solution. This idea may be supported by findings in slug corrosion tests. There it was found that when a pinhole is put in an unbonded can and the slug corroded, the cause of the first small lump or pimple in the can after about a day is the presence of solid tuballoy hydride. If the slug is allowed to corrode further, and the lump to grow, the corrosion product causing the swelling of the can is oxide, and not hydride. It is not difficult to believe, then, that hydrogen may play a very important role in the corrosion of the metal. Experiments to test this opinion have not yet been done due to shortage of manpower and relatively low priority. Beaker corrosion tests saturated with H_2, O_2, N_2 are indicated.

The difference in behavior of the metal under the two conditions was remarked in a letter from J. E. Draley to M. C. Leverett at Clinton in MUC-JED#150, dated October 26, 1943.

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Briefly, the advantages of using the hydrogen evolution test over the open test are that measuring the gas evolved does not interfere with the progress of the corrosion, and it is relatively quick and easy to run. On the other hand it cannot be applied to flowing systems and the results are not applicable to any system other than one much the same as the testing set-up.

Conclusions:

Of particular interest in thinking about using uncoated metal in a pile is the rather sudden jump in corrosion rate as the temperature increases. If it were possible to stay at a temperature of 70°C or below, for example the corrosion rate might not be too serious in refreshed distilled water. For any specific application tests would have to be run in the water proposed under the conditions actually expected to be used. It seems important at present to remember the difference between stagnant and circulating water.

This is an interim report and work will continue, probably with only minor emphasis placed upon it. In storing metal powder and scrap it is desirable that corrosion rate be as low as possible, particularly so that hydrogen evolution will be a minimum. Some corrosion inhibition at room temperature has been tried without success. Further experimenting along this line is planned.

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APPENDIX

References

- (1) T. Wathen, BR-223 (CC) -- Corrosion of Tu in Air and Steam up to 500°C. Rate incr in both. In steam at 150°C is 8.8 cm/yr and at 200°C is 57 cm/yr.
- (2) Greenwood, ME-24(CT) -- Corrosion of Tu in Air at 100, 150, 200°C. Adding 1 or 1.7% Si to the alloy decreases rate considerably.
- (3) J. P. Howe, CT 423, -- Corrosion of Tu in Air at 200-300°C
- (4) R. B. Hoxeng at Ames, CT 483 -- Pre-Surfacing metal offered only temporary Resistance to Air Corrosion.
- (5) H. S. Brown, Memo 41, dated Jan. 18, 1942 (really written in '43) --Tu not appreciably corroded by UF₆ and F₂ at 80°C.
- (6) C. A. Hutchinson et al, A 1249 (CC) -- In rotating tests, corrosion of Tu is levelling off to a lower rate during first 400 minutes.
- (7) Greenwood, BR 78 (CT) or CTB 120 - corrosion rate in boiling water 9.0 mm/yr. This can be reduced by a factor of 7 with the best inhibitor tried.
- (8) R. B. Hoxeng at Ames, CT 423 - Corrosion rate in boiling water 2.2-2.4 mg/cm²/hr. Oxide and sulfide films effective as protection for a time but wear off.
- (9) R. B. Mears and H. E. Daniels, Trans. Electrochem. Soc. 68, 375----- Errors in Corrosion Research.

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NOTEBOOK REFERENCES

The recorded data may be found in the following notebooks:

Preliminary tests on Tuballoy

Draley N.B. 217 27 and 51-93
Hirt N.B. 393 pages 29-135

Tuballoy in water and dilute hydrogen peroxide

English N.B. 435 pages 25-194
Levon N.B. 461 pages 96-124

Tuballoy alloys, including nickel alloys

English N.B. 435 pages 118-194
Levon N.B. 461 pages 96-124

Special Tests

Rebol N.B. 743 pages 2-39 (hydrogen evolution and
non-flowing system)

Hoxeng N.B. 1035 pages 12-13 (potential of Tuballoy Couples
with aluminum and stainless
steel).

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