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LABORATORY TEST FOR DETERMINING BRAZEABILITY OF COPPER-PLATED STEEL STRIP

Edward J. Roehl and Emory B. Michel, Warren, Ohio, assignors to Pittsburgh Steel Company, Pittsburgh, Pa., a corporation of Pennsylvania

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This invention relates generally to testing procedures, but has reference more particularly to the laboratory testing of copper-plated steel strip, which is to be used for manufacture into brazed tubing, in order to determine the brazeability of such strip.

In making copper-plated steel strip for use in the manufacture of brazed tubing, it has been the practice to electroplate cold rolled continuous steel strip on both sides with copper to an approximate thickness of .000125".

The coils of copper-plated strip are then annealed, slit into specified widths, and then utilized for the manufacture of the brazed tubing. This tubing is usually made by forming the plated strip around a mandrel with a slight draw to insure good contact of the plated surfaces, which are then brazed together by heating the formed tubing in a brazing furnace, in an inert gas atmosphere, at temperatures slightly above the melting point of copper. In this manner, copper-coated steel tubing of various diameters is produced.

Most of the copper-plated steel strip made for this use is satisfactory and produces a good tubular product. In some cases, however, the copper-plated steel strip does not braze properly, resulting in lack of proper adhesion between the adjacent copper surfaces.

Attempts through laboratory tests to insure the manufacture of copper-plated steel strip, which will braze properly and insure the production of a uniformly good tubular product, have heretofore been unsuccessful, mainly, because it is difficult to duplicate manufacturing conditions on a laboratory scale.

After considerable work along this line, and based on a large number of tests and experiments, we have devised a laboratory test for the brazeability of copper-plated steel, which has been found to be entirely satisfactory, and which gives results that can be properly correlated with the behaviour of such material in the tube manufacturing process. The test is relatively simple and easy to perform, but involves a number of variables which must be strictly controlled within rather narrow limits, in order to obtain consistent results, which can be repeated and checked.

The present invention is directed primarily to this laboratory test, and to the control of the aforesaid variables.

In accordance with the invention, a sample or specimen of the copper-plated steel to be tested may be of any reasonable size, but for convenience and consistency of results should not vary in size. For this purpose, a strip about 10 inches long and about 1½ inches wide is preferably or desirably used.

The strip can then be cleaned, if necessary, by any convenient effective method or means, as, for example, by dipping it in carbon tetrachloride and wiping it dry. It should be clean and dry before starting the test, which is the subject of the invention.

As a preferred embodiment or example of the invention, the following sequence of steps may be employed.

(1) The copper plating is stripped or dissolved off both sides of the strip by any convenient method, as for example by completely immersing the strip in an aqueous solution containing 450 grams per liter of chromic acid (CrO_3) and 50 grams per liter of concentrated sulphuric acid (H_2SO_4) at a temperature of 80° F. for about seven (7) minutes. Neither the method of stripping nor the composition of the stripping bath are critical so long as the copper plating is completely removed without etching the steel.

(2) The stripped steel is removed from the stripping bath and thoroughly rinsed in cold running water.

(3) The clean wet strip is immediately immersed in distilled water containing 30 parts per million of chloride ion as sodium chloride, at 80° F., for three (3) minutes. The purpose of this dip is to bring the temperature of the strip to 80° F. and to provide a surface film of water containing 30 p.p.m. of chloride, the reason for which will appear hereinafter, in connection with the description of the next step. The concentration of chloride is critical to ±5 p.p.m., and the temperature is critical to ±1° F., which is maintained by a thermostatically controlled water bath.

(4) The strip, wet with the 30 p.p.m. chloride ion solution, is then immersed, for two minutes, in an immersion copper plating bath of carefully controlled composition and having a temperature of 80° F., which temperature is maintained by a thermostatically controlled water bath. The bath composition is:

Copper sulphate ($CuSO_4.5H_2O$)	250 gms./l.	(±5 gms./l.).
Oxalic acid ($H_2C_2O_4.2H_2O$)	8 gms./l.	(±2 gms./l.).
Chloride ion as NaCl	30 parts per million	(±5 p.p.m.).

The copper sulphate should be of a high degree of purity, virtually free from chloride, and is first dissolved in distilled water. The oxalic acid, dissolved in distilled water, is then added. A precipitate of copper oxalate is formed, which does not interfere with the test. The solution is saturated with copper oxalate and an equilibrium is established at pH=1.5, ±0.1. This is critical. A variation in oxalic acid concentration of ±2 gms./l. corresponds to a pH variation of ±0.1. The purpose of the oxalic acid is to establish and maintain pH=1.5, ±0.1, hence the concentration is fixed.

The solution is then made up to volume and analyzed for chloride content, which is then adjusted to 30 ±5 p.p.m. by adding sodium chloride as required. The solution is then brought to 80° F. and maintained at that temperature ±1° F. in a thermostatically controlled water bath, and is now ready for use.

The concentration of copper sulphate is arbitrary and can be anywhere between 200 and 325 grams per liter, but after the concentration is selected, it must be maintained within 5 grams per liter, in order to obtain consistent and reproducible results, as hereinafter disclosed. Similarly, the chloride ion concentration selected is arbitrary and can be anywhere between 10 and 50 parts per million, but after the concentration is selected, it must be maintained within 5 p.p.m. in order to obtain consistent and reproducible results, because for reasons, which have not yet been ascertained, this has a marked effect on the results obtained.

The temperature of the copper plating bath is also arbitrary, and can be any conveniently maintained temperature, but once selected, must be held within ±1° F. in order to obtain consistent and reproducible results, because, like the chloride ion concentration, it has a marked effect on the results obtained. The temperature of 80°

F. was selected because it is easily maintained constant in the laboratory and does not involve appreciable evaporation losses with attendant changes in solution concentration.

The reason for step 3 will be evident from the significance which has been attached to the maintenance of constant temperature and chloride ion concentration in the immerion test solution, as described above.

(5) At the end of two minutes immersion, the strip is removed from the plating solution and examined visually. It will be found to be covered on both sides with an immersion deposit of copper. The physical characteristics of this immersion copper plate can be directly correlated with the brazeability of the original copper electroplated steel from which the sample or test strip came. Good plate indicates good brazeability and bad plate indicates the opposite. By good plate is meant one that is bright, smooth, lustrous, adherent, of good color, and appears like a shiny film of pure copper. By bad plate is meant one that is dull, dark, spongy, non-adherent, porous, non-uniform, or any other variation from good plate. There are very few specimens which are either all good or all bad—most show variations of greater or less degree. Hence each side of the test specimens is rated numerically from one to ten on the basis of estimated area of good plate as follows.

Rating:	Percent of area of good plate
1	0–10
2	10–20
3	20–30
4	30–40
5	40–50
6	50–60
7	60–70
8	70–80
9	80–90
10	90–100

The ratings for each specimen are then recorded. For example, 10–10 is the highest rating and means that the immersion test plate was over 90% good on both sides and that the original copper plated steel has good brazeability, while 1–1 is the lowest rating and means that the immersion test plate was less than 10% good on both sides, and that the original copper plated steel has poor brazeability. In-between ratings, such as 7/5, 3/8, 6/4, etc., must be carefully correlated with actual brazing experience with the plated steel from which the test specimens were taken, in order to establish a standard of acceptability, and this will vary with the concentration of test solution and temperature at which the tests were made, emphasizing again the importance of holding these variables constant, once they have been selected. By changing the critical variables—solution concentration, chloride ion concentration and temperature—the numerical values of the ratings can be moved up or down at will, so that the degree of sensitivity of the test can be varied as desired to best correlate with brazeability data obtained from actual performance experience, in order to establish a fixed test procedure and a corresponding rating standard that will predict the brazeability of copper plated steel as it is produced and before it is actually used.

It is to be understood that various changes can be made in the concentrations of the solutions, temperatures, and other factors, without departing from the spirit of the invention, or the scope of the appended claims.

Having thus described our invention, we claim:

1. In a method of laboratory testing of the brazeability of copper-plated steel strip, the steps which comprise taking a specimen of the strip, cleaning the specimen, removing copper plating from the specimen, rinsing the stripped specimen in water, dipping the rinsed specimen in an aqueous solution containing about 30 parts per million of chloride ion as sodium chloride, at a selected but relatively constant temperature, for about three minutes, then without rinsing, immersing the specimen for about two minutes in an aqueous immersion copper plating test bath containing from about 200 to about 325 grams per liter of $CuSO_4.5H_2O$, about 6 to about 10 grams per liter of oxalic acid, and from about 10 to about 50 parts per million of chloride ion as sodium chloride, and having a selected but relatively constant temperature, whereby to provide an immersion deposit of copper thereon, and visually examining the immersion copper deposit in comparison with a pre-established rating schedule to determine the relative brazeability of the original copper-plated strip from which the specimen has been taken.

2. In a method of laboratory testing of the brazeability of copper-plated steel strip, the steps which comprise taking a specimen of the strip, cleaning the specimen, removing the copper plating from the specimen by immersing it in an aqueous solution containing about 450 grams per liter of chromic acid and about 50 grams per liter of sulphuric acid, rinsing the stripped specimen in water, dipping the rinsed specimen in an aqueous solution containing about 30 parts per million of chloride ion as sodium chloride, at a temperature of about 80° F., for about three minutes, then without rinsing, immersing the specimen for about two minutes in an immersion copper plating test bath containing about 250 grams per liter of $CuSO_4.5H_2O$, about 8 grams per liter of oxalic acid, and about 30 parts per million of chloride ion as sodium chloride, and having a temperature of about 80° F., whereby to provide an immersion deposit of copper thereon, and visually examining the immersion copper deposit in comparison with a pre-established rating schedule whereby to determine the relative brazeability of the original copper-plated steel strip from which the specimen has been taken.

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