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Application of Induction Coil Measurements to the Study of Superalloy Hot Corrosion and Oxidation

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      The hot corrosion of U700 was studied at 900 C in a Mach 0.3 flame doped
      with 0.85 wppm of sodium. The change of inductance was found to define the
      known corrosion behavior and to suggest its use as a tool with predictive
      capabilities. Sufficient sensitivity exists to detect oxidation of this
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APPLICATION OF INDUCTION COIL MEASUREMENTS TO THE STUDY OF SUPERALLOY HOT CORROSION AND OXIDATION

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E-1937

SUMMARY

The assessment of the degree of hot corrosion attack on nickel based alloys is a difficult task, especially when the definition specifies that it must be in terms of metal consumed and even more difficult if the measurement must be nondestructive. It is known that the inductance of a solenoid coil responds to changes in volume of fill and composition of metal cores, therefore, it was reasoned that it may be used for nondestructive measurement of hot corrosion. The hot corrosion of U700 was studied at 900° C in a Mach 0.3 flame doped with 0.85 wppm of sodium. The change of inductance was found to define the known corrosion behavior and to suggest its use as a tool with predictive capabilities. It also had sufficient sensitivity to detect oxidation of this alloy at 900° C.

INTRODUCTION

The ability to measure the extent of corrosive flame attack of alloys is necessary to alloy development and the formulation of control strategies. Measures of corrosive flame attack such as weight change, external diameter change and others have been used extensively (refs. 1 and 2) but the interpretation of the results, due to scale buildup, for example, can in many cases be misleading. Therefore, by definition, the extent of corrosive attack will here be defined as the consumption of metal. The only totally suitable method to determine metal consumption is by destructive means such as cutting the specimen and measuring the cross-sectional diameter or area of the unattacked metal and comparing it to the respective starting values. However, this is slow and costly in terms of both specimens and manpower.

Such measurements are also difficult to perform due to the physical alignment problems arising during cutting, grinding, mounting and measuring operations. Therefore, a measurement which gives direct information about the metal consumption and compositional changes taking place as the corrosion test progresses would be desirable. In addition, this measurement should be relatively easy to implement.

Based on the above considerations, it was decided to examine the inductive properties of solenoid coils with metal samples as coil cores for application to hot corrosion and oxidation attack studies of the samples. It has been established (ref. 3) that the inductance of a coil of wire carrying a high frequency signal is proportional to the conductivity and magnetic permeability of the metal which in turn is a function of the composition of the metal core inserted into the coil and the extent of fill of the coil's volume. Furthermore, when nonmagnetic cores are used the inductance of the coil decreases while magnetic cores increase the coils inductance (ref. 3). Also, at

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high frequencies, for cores of equal volume, most of the core effect on inductance is caused by a thin surface layer of the core material. It was reasoned that these properties may provide a dual sensitivity method for the study of hot corrosion attack. First, this method may provide a measure of the rate of skin depletion or surface zone formation in early stages of attack, long before any apparent macro-corrosion could be detected. Second, it may provide a means of following the volume change of the metal at later times during catastrophic corrosion (ref. 4) and thus yield information on the rate of attack. In fact, the use of the "coil technique" for study of hot corrosion has been previously reported (ref. 5). The main thrust of that work was the study of corrosive conditions while, the present report is a more detailed discussion of the technique itself and its application.

To demonstrate the properties of coils, the inductance change of an air core coil with respect to specimen core diameter and degree of fill was measured for 304 stainless steel and U700 alloy cores. The verification of the skin effect was studied by inserting successively thicker hollow brass cylinders into the coil. The inductance change of a coil was measured periodically for a U700 hot corrosion test specimen heated to 900° C by a Mach 0.3 flame containing 0.85 wppm sodium. It was found that during the initial or induction period (ref. 4) of hot corrosion the inductance of the coil-core combination increased rapidly reflecting the skin effect caused by compositional change of the core surface. This was then followed by a period in which essentially no inductance change occurred. Then, with the onset of catastrophic corrosion (ref. 4) the inductance again increased rapidly reflecting a volume change of the core. The same experiment was repeated using an equivalent U700 specimen but without the sodium added to the flame. The change of inductance with oxidative exposure had the same general shape as for hot corrosion.

EXPERIMENTAL

Coil Design and Inductance Measurement

Inductance was measured with a multifrequency LCR meter and associated test fixture. The cylindrical coil and its support were designed to fit the test fixture as shown in figures 1 and 2. The length of the coil was made to contain approximately the flame impingement area on the specimen and hence the length of the corroded area of the cylindrical sample (about 1 in.). It was found necessary to have the coil cover mainly the corroded length of the sample to provide the best sensitivity to attack detection. To attain the best sensitivity the inside diameter of the coil should be kept to a minimum but must be large enough to accommodate the sample core even with a small amount of scale formation. The cross-sectional shape should be similar to that of the specimen. The coil must also be designed to operate in a stable manner, that is, removed from its self-resonate condition.

The LCR meter was equipped with the IEEE-488 bus. This permitted control and measurement by a microcomputer. A program was developed which preset the measuring conditions at 10 megahertz signal frequency, 1.0 volt oscillator level, series mode and high resolution. The stray electrical properties of the fixture are manually zeroed out before the coil is mounted. Then the coil is mounted and adjusted in the fixture mounting clamp so that the inductance of the air filled coil is always the same initial value. The program was set to make four measurements of the inductance with the core in place and report the average. Between each measurement the core was removed and replaced randomly so as to average the core-coil axial alignment variation on the inductance. The inductance was measured to four decimal places and reported as microhenries.

Skin Effect, Volume Fill and Calibration

The effect of the skin thickness on the inductance of the coil was assessed by placing hollow cylinders, made up of 1 mil thick rolled up brass shim stock, into the coil. The inductance was measured after successive additions of 1 mil layers.

To demonstrate that the inductance of the coil was a function of the volume fill of the coil, a series of solid 304 stainless steel cylinders were machined to various diameters so as to effect a 40 to 70 percent fill of the coils volume. The inductance of the coil-core combination was measured for each core.

In addition the coil was calibrated for 304 stainless steel and U700 by measuring the inductance of the coil using solid cylinders of each material machined to diameters between 450 and 490 mils. The 490 mil diameter is that of the typical hot corrosion test specimens and this diameter change range corresponds to that of interest in hot corrosion studies.

Hot Corrosion and Oxidation Experiments

The hot corrosion attack of a U700 cyclindrical specimen initially 490 mils in diameter, was effected in a Mach 0.3 atmospheric pressure burner rig shown in figure 3. Eight specimens were contained in a carousel which rotated at 600 rpm in the flame path. In a carousel each specimen rotates off its own axis and thus sees some nonuniform conditions which can lead to nonuniform corrosion, as the pressure side is usually more corroded than the other. However, for the coil inductance measurements these variations are not critical because the measurement averages over the entire corrosion volume. The specimen temperature was maintained at a nominal 900° C for one hour then cooled for 3 min to about 100° C in a stream of air and this comprised one cycle of testing. Because the heating time is one hour, cycles and hours can be used interchangeably when reporting results in terms of test time. The flame was doped with 0.85 wppm (weight parts per million parts of air) of sodium added to the combustion chamber as a water solution of sodium chloride. Jet A-1 fuel was used and it contained 0.04 to 0.05 wt. sulfur.

At intervals during the cyclic test the specimen was removed and placed in the coil and the inductance measured as described above. The external diameter at the center of the flame impingement on the specimen was also measured with a thread micrometer caliper at 50 cycle intervals. The oxidation experiment was effected on an equivalent U700 specimen in the same fashion as described, but no sodium was added to the flame.

DISCUSSION OF RESULTS

Influence of Volume Fill of the Coil

Figure 4 is a plot of measured inductance as a function of the percentage of fill of the coil volume by 304 stainless steel cores. The inductance of the coil decreases with increase in fill and the experimental points were found by least squares to fit the line shown. The R squared value is the square of the correlation coefficient. Figure 4 indicates that the coil-core inductance measurement should respond to a change of metal volume occurring in a core which is a corrosion test specimen. That is, as metal is lost the fill of the coil decreases and the inductance changes in proportion. It should also be apparent that since volume is related to diameter and length, there is a relation between diameter change and inductance change for constant length. This is developed in the appendix.

Calibration

It has been demonstrated above that the inductance of the coil changes linearly with volume of metal fill and since the volume of a cylinder is related to the diameter at constant length, as shown in the appendix, the relation of core diameter to inductance was further investigated. Figure 5 shows that the measured inductance for constant length (the coil length is constant) increases approximately linearly with diameter for 304 stainless and U700 alloys in the limited range of 450 to 490 mils, which is of interest in most hot corrosion studies. The slope or sensitivity factor for U700 is 0.0104 microhenries per mil diameter change and the factor for 304 stainless steel is 0.0093. The relationship of diameter instead of volume is preferred in practical measurements simply because it is easier to measure a diameter. In principle, inductance can be measured to 0.000X so the resolution is theoretically about 0.01 mils diameter. This cannot be approached in practice because of measurement problems such as core-coil axial misalignment. The core-coil axial misalignment problem is even worse when higher frequencies are used (above 20 megahertz).

Skin Effect

Figure 6 demonstrates the effect of the thickness of hollow cores of 1 mil thick nonmagnetic brass on the inductance of the coil. As the wall thickness of the hollow core increases in 1 mil increments the inductance decreases rapidly until approximately 2 mils total thickness is reached. At greater than this thickness no further inductance change is observed. The skin depth is a function of electrical resistivity, magnetic permeability and frequency of measurement (ref. 6). Skin depth is defined as the depth at which the coil's magnetic field intensity is reduced to 63 percent of the surface value and is given by D = 1.98 (R/F*P) 0.5 (in.) where R is the electrical resistivity microohm-cm, P is the magnetic permeability, which is one for nonmagnetic materials, and F is the frequency in Hertz. For brass, which is a nonmagnetic material, P = 1, R = 9, and F = 10 000 000 Hertz the calculated skin depth is 1.9 mils. This is in good agreement with the experimental result. Skin depth is a function of frequency and from the equation it is to be noted that an increase in measuring frequency will decrease the skin depth.

The important point to be made is that for equal volume of the core the surface or skin characteristics of the core determine the inductance and the remainder of the core composition is insignificant. In the brass example above the alloy composition remained constant and the thickness changes lead to the inductance changes. When the skin depth was exceeded the change in inductance ceased. In the case of hot corrosion or oxidation attack, the surface of the alloy can change chemical composition as testing proceeds, thus resulting in a change in inductance for a constant measuring frequency. The inductance changes because of the new electrical properties of the sample surface. If the composition of this surface layer, resulting from corrosion exposure, is constant and the surface layer is just thickening with time, then when the layer has a depth greater than the skin depth, further inductance change should stop. Any further changes in inductance would occur only if the volume changed.

Hot Corrosion of U700

Figures 7 and 8 show the measured inductance and the measured external diameter respectively as a function of the cyclic hot corrosion exposure for a U700 sample. In figure 7 there is an initial rapid rise in inductance up to about 30 cycles. This change could be due to a volume and/or compositional change. If the rise in inductance is converted to a change in diameter, using the 0.0104 microhenries per mil calibration factor, then the loss would be 15 mils. However, figure 8 shows no measurable change in the external diameter even up to 50 cycles. Also, measurements of the internal diameter after cutting, mounting and polishing showed only 2 mils of diameter change for an equivalent sample at 20 cycles. Furthermore, an equivalent sample was annealed in argon for 300 hr at 900° C and no change in the inductance was measured. One is thus lead to conclude that the initial rapid rise is due mainly to a chemical composition change in the surface of the specimen. The original U700 is nonmagnetic and the increase in inductance suggests the surface composition is changing toward a magentic alloy. In fact at 60 cycles the specimen showed some magnetic attraction when suspended next to a permanent magnet. The change in slope of the curve above 30 cycles suggests that a constant composition surface layer has been produced and it thickens until it equals the skin depth at about 35 cycles. Once the skin depth has been exceeded there should be no further change in the inductance until there is a change in mechanism. Indeed, this is what is observed as little change is measured between 30 and 45 cycles.

In figure 7 the inductance is seen to rise rapidly from 45 to 200 cycles. It will be noted in figure 8 that the external diameter starts decreasing at about 50 cycles. This final inductance change, starting at 45 to 50 cycles, is caused by a metal volume change apparently resulting from catastrophic corrosion attack. Thus the rate of diameter change per cycle, measured by the micrometer caliper and, that calculated from the final inductance data would be expected to be equal. A least squares line was fitted to the measured inductance versus test cycle plot for the range from 45 to 200 cycles. The slope was found to be 0.0018 microhenries per cycle. When this slope is divided by the calibration factor previously determined for U700 (0.0104 microhenries per mil) the catastrophic corrosion rate is equal to 0.17 mils metal diameter change per cycle. This value compares well with the corrosion rate of 0.21 mils diameter change per cycle obtained from the external diameter measurements made at the center of the attack zone. Two points must be recognized when comparing these methods of attack measurement. First, the inductance coil method measures over the whole specimen length and thus it yields an equivalent or average diameter change for the entire attack zone within the coil. However, the attack profile is not uniform over the length of the attack zone: usually attack is greatest in the center of the attack zone corresponding to the highest temperature produced by the central part of the flame. The second point to consider is the fact that the attack product scale plays a vital role in caliper diameter measurements. For U700 during hot corrosion there is very little scale adherence and so caliper diameters are expected to be proportional to metal consumption. For alloys where scale buildup occurs it is evident that the caliper results would be of little use, and in fact during oxidation of U700 the caliper diameters increase with exposure. The inductance coil method thus gives the average of equivalent attack rate independent of scale buildup, while the caliper method, where it can be used, gives the maximum rate because it is measured over a very narrow region at the maximum attack zone. The coil used here was 1-1/16 in. long and covered the total attack length for the Mach 0.3 flame. This accounts for the lower corrosion rate yielded by the induction coil method. In fact, it has been observed in unreported ancilary work that at higher Mach numbers where the flame becomes narrower measurements made with the 1-1/16 in. long coil yield attack rates about 1/2 of those determined by the caliper method. If the coil had been shortened or the specimen had been moved closer to the burner nozzle, the measured rates would have been in better agreement. Thus, it is important for an absolute measurement to make the coil length such that it covers mainly the maximum attack zone and to center the maximum attack zone in the center of the coil reproducibly before each measurement of inductance. Under the best of conditions and when care is taken, the coil method can approximate the true rate. However, on a relative basis, for comparison of alloys or test conditions for example, the coil length with respect to attack length makes little difference as long as the coil and test specimen are reproducibly positioned before measurements are made. Of course, the sensitivity is better if the coil covers the zone most corroded.

The results of auxiliary work indicate that except for the initial rise in the inductance at short corrosion times, there is much deviation in the shape of the inductance versus test time curve for alloys other than U700. This is due to the fact that superalloys of differing composition corrode by different physico-chemical mechanisms. Some alloys spall extensively in each cycle (as U700) some buildup an extensive scale and some corrode more in cooler portions of the specimen. It has been observed that some alloys show a rapid decrease in inductance very soon after the initial rise and no plateau is ever established. This is believed to be due to "metal lifting", that is metal particles are dispersed in the scale and this brings metal close to the coil and in effect acts like an increase in metal volume and so inductance decreases. Other alloys do not show a flat plateau, but their inductance slowly "drifts" up with corrosion exposure. This behavior has been observed only for more resistant alloys and may be due to a slow change in the composition and thus electrical properties of the skin layer so that the skin depth is changed.

Probably the most interesting and potentially useful feature of the inductance coil method is the initial or short time information obtained before any visual appearance of gross corrosion. From auxiliary work it has been observed for all alloy compositions tested that the slope of the initial portion of the inductance-time curve up to about 50 cycles varies with the initial alloy composition. In general, if slope comparisons are made for alloys of similar aluminum and titanium contents, the greater the slope for the first thirty cycles the less resistant the alloy proves to be in long time corrosion testing. This indicates that the inductance coil method may have some predictive value, that is, forecasting long term corrosion resistance from short time test data. It was reported in reference 5 that for U700 a strong relationship exists between the initial slope and final slope for changes in sodium content of the flame. This again indicates that long time behavior may be predicted from short time measurements.

Oxidation

The measured inductance versus the number of corrosion cycles for the oxidation experiment is given in figure 7. The general shape of this curve is similar to that for hot corrosion except the changes in inductance are much smaller. In hot corrosion the final catastrophic stage was entered at 50 cycles while the final oxidation stage was not reached until 100 cycles. The slope of the inductance curve in the catastrophic oxidation stage was 0.00027 microhenry per cycle as determined by a least squares fit. This is about ten times smaller than the hot corrosion value of 0.0018 microhenry per cycle. When the slope of the final portion of the oxidation-inductance curve is divided by the calibration factor, for U700 of 0.0104 microhenry per mil diameter change, then the rate of metal diameter loss is 0.026 mil per cycle. The caliper measurements of the external diameter yielded an increase with increasing exposure due to a lack of spalling of the scale. Therefore, the internal metal diameter was measured on equivalent specimens after 100 and 200 cycles after sectioning, mounting, polishing, and etching. It was found that the diameter loss between 100 and 200 cycles was 2 mils. This yields a final rate of 0.02 mil of metal diameter loss per cycle assuming a linear relation between the two measured points. This compares favorably with that determined from the coil measurements when considering the internal diameter measurement uncertainty of 0.8 mil (ref. 7) at best.

CONCLUSIONS

(1) The inductance change of a coil-sample core combination shows two responses important to its use as a tool for measurement of attack in hot corrosion or oxidation studies: First, it detects the skin effect caused by compositional changes in the alloy surface before the onset of macro attack. Second, there is a bulk or volume effect response due to gross loss of metal associated with catastrophic attack.

(2) There is a good correlation between the catastrophic hot corrosion rate determined by the coil method and the micrometer measurement of the external diameter for U700.

(3) The induction coil method of measurement of hot corrosion attack has potential for use as a tool with predictive capabilities of long time behavior from short time tests.

(4) The coil method has sufficient sensitivity to detect oxidation of U700 at 900° C in a Mach 0.3 flame.

APPENDIX

In the following discussion the relationship of the inductance of the coil with respect to diameter change of a solid cylindrical specimen core is given. The inductance is related to volume of fill of the coil by

$$L = aX + b \qquad (1)$$

Where $X = Vs/Vc$ and Vs = volume of sample and Vc = volume of the coil, L = inductance of the coil and a and b are constants. Also it is known that

$$Vs = PI/4*Ds\hat{\ }2*LENs \qquad (2)$$

$$Vc = PI/4*Dc\hat{\ }2*LENc \qquad (3)$$

Where Ds = diameter of the sample, Dc = diameter of the coil, $LENs$ = length of the sample and $LENc$ = length of the coil. Now it is given that the length of the sample is approximately equal to the length of the coil neglecting end effects, So, L, is a function of diameter as:

$$L = a(Ds\hat{\ }2/Dc\hat{\ }2) + b \qquad (4)$$

For the case of an air core coil where $Ds = 0$ then $L = b$, designating the air core inductance as Lo then:

$$L = a\ (Ds\hat{\ }2/Dc\hat{\ }2) + Lo \qquad (5)$$

Now let $A = a/Dc\hat{\ }2$ where A is a constant. This then gives:

$$L = A*Ds\hat{\ }2 + Lo \qquad (6)$$

So the inductance thus depends on the square of the sample diameter and A, which is the slope of the L versus $Ds\hat{\ }2$ curve, but the first derivative of equation (6) is given by:

$$dL/dDs = 2*A*Ds \qquad (7)$$

Which shows that the change in inductance is a function of the diameter change of the sample.

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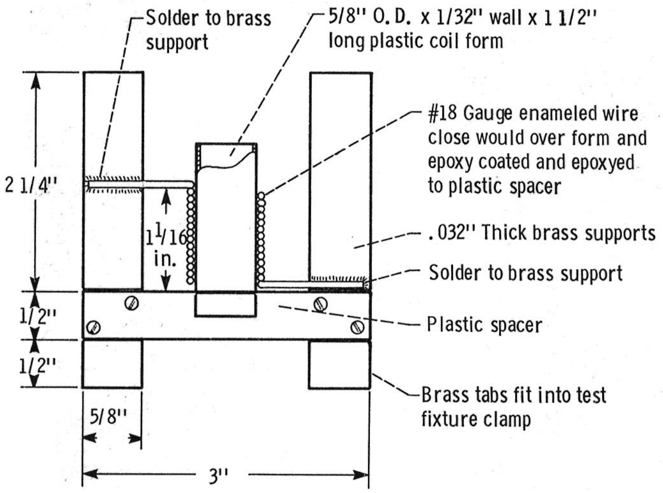


Figure 1. - Coil and coil support design.

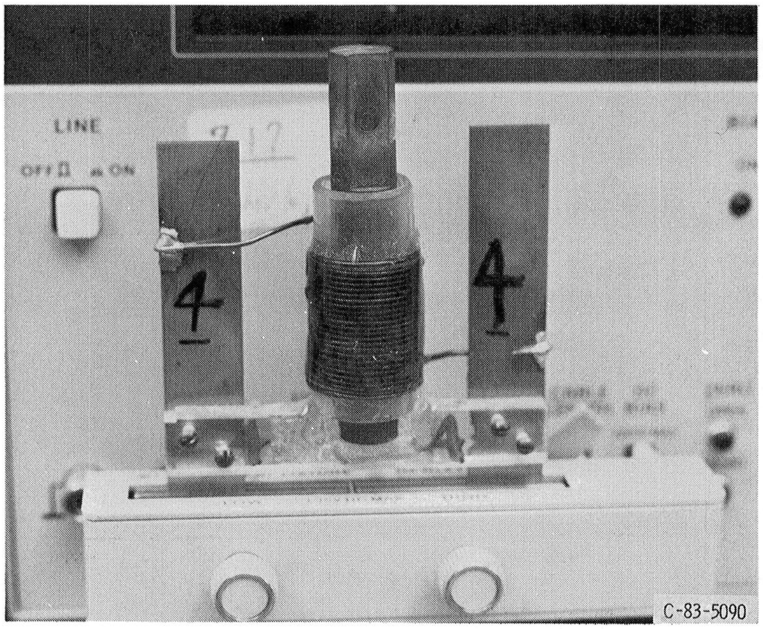
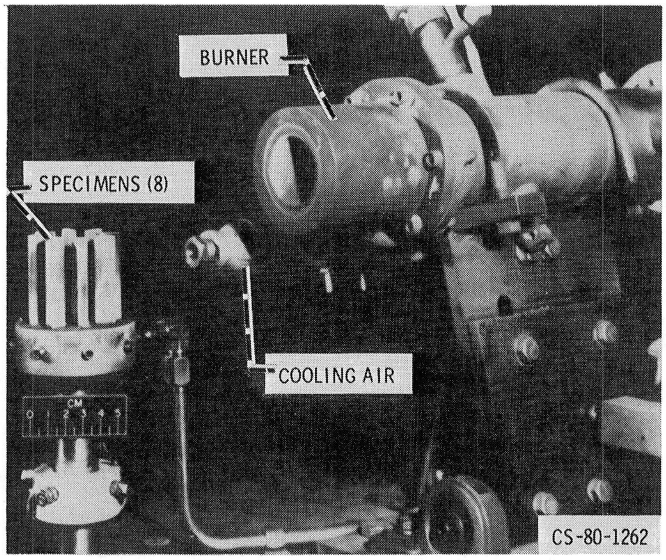


Figure 2. -Photograph of coil mounted in test fixture.



BURNER RIG

Figure 3. - Mach 0.3 burner rig.

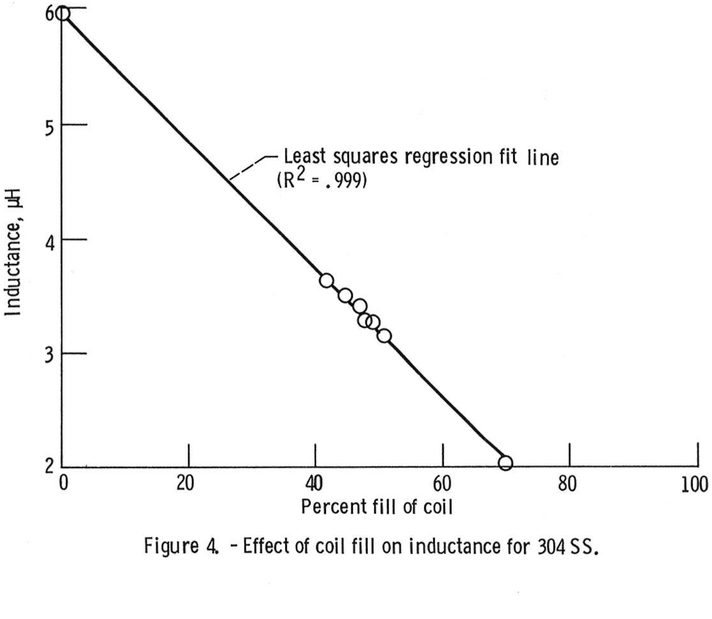


Figure 4. - Effect of coil fill on inductance for 304 SS.

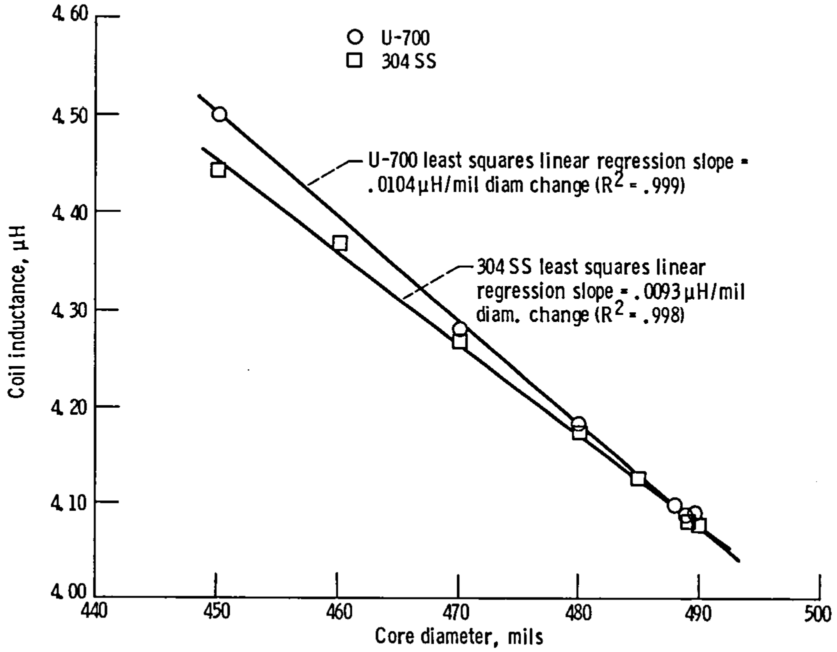


Figure 5. - Effect of core diameter on coil inductance for 304 SS and U-700 Alloys.

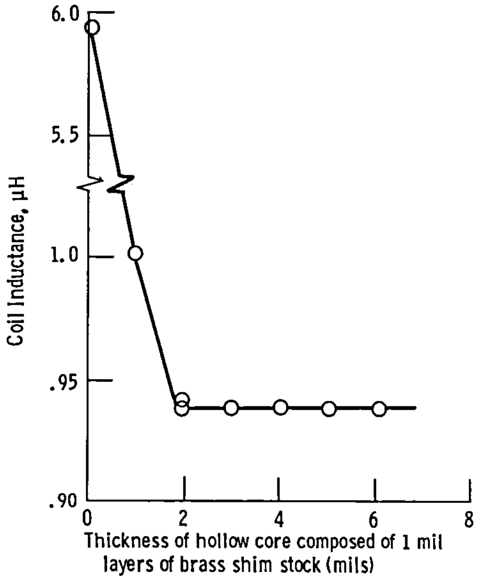


Figure 6. - Effect of a brass shim stock core thickness on coil inductance.

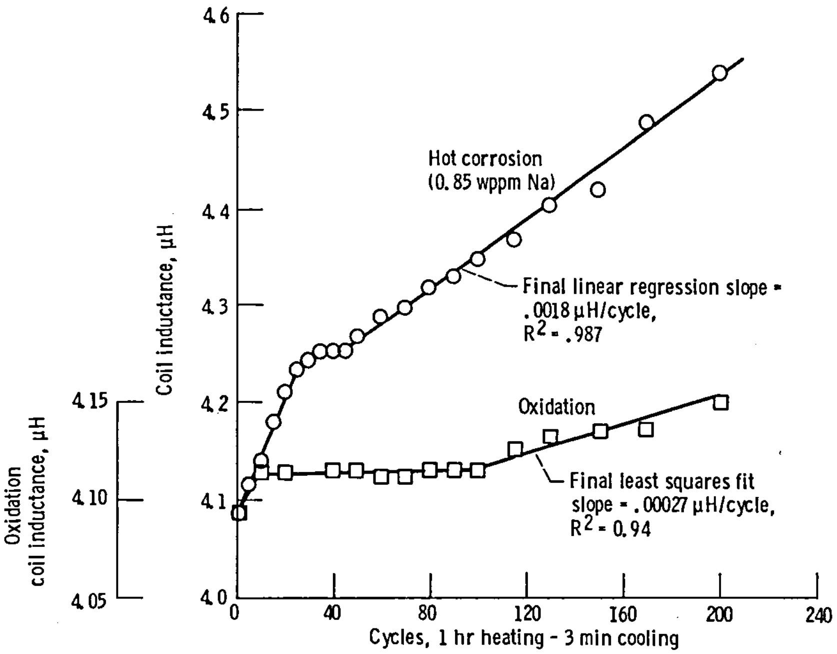


Figure 7. - Change of coil inductance with hot corrosion and oxidation of U700 Alloy at 900° C - MO.3.

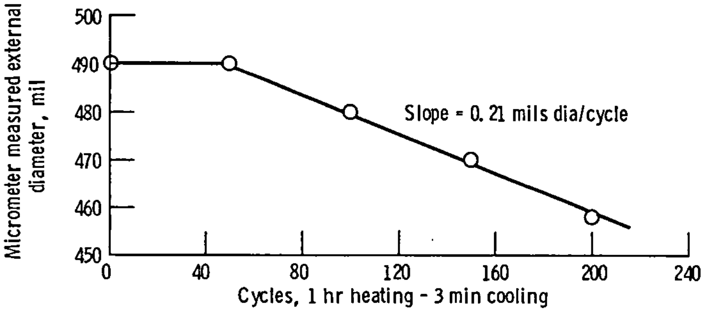


Figure 8. - Change in external diameter with hot corrosion of U-700 Alloy at 900° C - MO.3 - 0.85 ppm Na.

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15. Supplementary Notes

16. Abstract

The assessment of the degree of hot corrosion attack on nickel based alloys is a difficult task, especially when the definition specifies that it must be in terms of metal consumed and even more difficult if the measurement must be nondestructive. It is known that the inductance of a solenoid coil responds to changes in volume of fill and composition of metal cores, therefore, it was reasoned that it may be used for nondestructive measurement of hot corrosion. The hot corrosion of U700 was studied at 900° C in a Mach 0.3 flame doped with 0.85 wppm of sodium. The change of inductance was found to define the known corrosion behavior and to suggest its use as a tool with predictive capabilities. It also had sufficient sensitivity to detect oxidation of this alloy at 900° C.

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