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THE EFFECT OF VARIATIONS OF COBALT CONTENT ON THE CYCLIC OXIDATION RESISTANCE OF SELECTED Ni-BASE SUPERALLOYS

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SUMMARY

Cobalt levels were systematically varied in the Ni-base turbine alloys U-700 (cast), U-700 (PM/HIP), Waspaloy, Mar-M-247, IN-738, Nimonic-115, U-720, and SX-R-150. The cobalt levels ranged from 0 wt % to the nominal commercial content in each alloy. The alloys were tested in cyclic oxidation in static air at 1000, 1100 and 1150 °C for 500, 200 and 100 hr respectively. An oxidation attack parameter, K_a derived from the specific weight change versus time data was used to evaluate the oxidation behavior of the alloys along with x-ray diffraction analysis of the surface oxides. The alloys tend to form either Cr_2O_3 /chromite spinel or Al_2O_3 /aluminate spinel depending on the Cr/Al ratio in the alloys. Alloys with a ratio of 3.5 or higher tend to favor the Cr oxides while those under 3.0 form mostly Al oxides. In general the Al_2O_3 /aluminate spinel forming alloys have the better oxidation resistance. Increased cobalt content lowers the scaling resistance of the high Cr alloys while a 5.0 wt % Co content is optimum for the Al controlling alloys. The refractory metals particularly Ta appear beneficial to both types of oxides perhaps due to the formation of the omni-present tri-rutile $Ni(Ta,Cb,Mo,W)_2O_6$. Both scales break down as increasing amounts of NiO is formed.

INTRODUCTION

NASA Lewis Research Center undertook a long range program in support of the aerospace industry aimed at reducing the need for critical metals used in gas turbine engines. The program was termed "COSAM" - Conservation of Strategic Materials. One of the major objectives was to understand and possibly minimize the use of a critical element such as cobalt in high strength nickel-base superalloys (refs. 1 and 2). This study focuses on the role of cobalt on high temperatures cyclic oxidation on some typical alloys of interest.

Seven high temperature nickel-base superalloys were studied in high temperature cyclic oxidation. The alloys include: U-700 cast and powder metallurgy (P/M) hiped samples; hot worked Waspaloy; cast MAR-M-247; hot worked IN-738; hot worked Nimonic-115; hot worked U-720; and cast single crystal (SX) R-150. The alloys were varied in their cobalt levels from 0 to their normal commercial levels. The chemistries for each alloy are shown in table I (ref. 1). These superalloys represent a range of high-temperature, high strength Ni-base turbine alloys with a range of Cr to Al ratios from 15:1 for Waspaloy and near 7:1 for U-720, which can be hot worked, to alloys with Cr/Al ratios near 1, which must be cast.

PROCEDURE

The supplied test alloys were machined into oxidation test coupons each 1 by 2 by 0.23 cm with a 0.3 cm diameter hanger hole. The samples after cleaning and weighing were automatically cycled in static air furnaces as described in reference 3. In this study the samples were tested for 1 hr cycles consisting of 1 hr at either 1000, 1100, or 1150 °C in the furnace and a minimum of 20 min above the furnace at a temperature of near 65 °C. The samples were removed periodically for weighing.

The test times were 100, 200, and 500 hr at 1150, 1100 and 1000 °C respectively. These data are used to generate specific weight change/time values to evaluate the severity of the oxidation attack.

In addition to the weight change data, each sample and its collected spall were removed and analyzed by x-ray diffraction after 1,100, and when applicable 200 and 500 hr.

RESULTS AND DISCUSSION

ANALYSIS OF SPECIFIC WEIGHT CHANGE DATA

The oxidation behavior for each test sample can be determined from the specific weight change versus time data either by comparing the specific weight change at a given time or, more accurately by deriving an attack parameter, defined as K_a , from the fitted data which defines the overall oxidation resistance. The weight change/time data is fitted by multiple linear regression to:

$$\Delta W/A = k_1^{1/2} t^{1/2} - k_2 t \pm SEE \quad (1)$$

where $k_1^{1/2}$ represents an oxide growth constant and k_2 an oxide spalling constant. SEE is the standard error of estimate. A rejection level of 0.90 is chosen for significance. This leads to an attack parameter defined as:

$$K_a = (k_1^{1/2} + 10 \cdot k_2) \quad (2)$$

in certain cases a more appropriate estimating equation is a simple linear fit:

$$\Delta W/A = -k_2 t \pm SEE \quad (3)$$

which modifies the attack parameter to:

$$K_a = (20 \cdot k_2) \quad (4)$$

These equations, examples, and their rationale have been discussed previously in references 4 to 9.

In general the oxidation resistance can be rated as follows:

K_a < 0.20 excellent
0.20 to 0.50 excellent to good
0.50 to 1.00 good to fair
1.00 to 5.00 fair to poor
5.00 to 10.00 poor to catastrophic
>10.00 catastrophic

Where "excellent" means a low scale growth rate with minimal spall at one extreme while "catastrophic" means the sample is almost totally oxidized and usually converted to massive spall.

Selected specific weight change versus time curves for the various alloys are shown in figures 1 to 3. The individual data sets were fitted first to equation (1) by a multiple linear regression. If the significance level of either $k_1^{1/2}$ or k_2 did not exceed 0.90 it was dropped and the regression equation recalculated. Of the 127 runs for all alloys at the 3 test temperatures 99 give a good fit to (ref. 1) with the rejection level of 0.90. Fourteen runs had the first term drop out as not significant to the 0.90 level so (refs. 3 and 4) are used to estimate K_a . Twelve of the 127 runs were forced when $k_1^{1/2}$ is negative to a simple linear fit so again (refs. 3 and 4) are used rather than (refs. 1 and 2) to estimate K_a . Examples of these 3 types of fit are shown in table II. Finally 2 of the 127 runs were rejected outright as outliers. Table II summarizes this "fit" data for the 8 sets of alloys at each of the 3 test temperatures.

Tables III, IV, and V summarize the specific weight change data and the derived constants from equations (1) to (4). Additional data of a statistical nature are tabled in the appendix separately for each of the three test temperatures. In addition to the $k_1^{1/2}$, k_2 , and K_a derived parameters, specific weight changes at 100 hr and, where available, 200 and 500 hr are also listed to indicate sample behavior.

The K_a values derived for each set of alloys at each temperature are shown on log scale bar graphs in figures 4 to 6. The bars are grouped from low to high cobalt values from left to right on the plots. For each alloy series any replicates for a given alloy are also shown. The K_a rankings ranging from excellent to catastrophic are shown as horizontal lines.

The x-ray diffraction results from test sample surfaces are summarized in tables VI to VIII. There is a massive amount of data to be condensed because of the number of oxides found on these complex alloys. In general the alloys tend to be either Cr_2O_3 /chromite spinel formers or Al_2O_3 /aluminate spinel formers. Almost always present with either type is a tetragonal tri-rutile oxide with a d-lattice spacing on the (110) plane <3.30 Å. This is usually $Ni(Ta,Cb,Mo,W)_2O_6$ also termed tapiolite (ref. 10). Also found throughout the analyses was Cr titanate $Cr_2Ti_{13}O_{29}$, Ni titanate-(Ni,Co,Fe) TiO_3 , and Ni Tungstate- $Ni(W,Mo)O_4$ and finally the alloys fail as massive NiO is detected. Occasionally a tri-rutile structure with a d-value >3.30 is detected. An alloy like MAR-M-247 with 1.5 Hf will indicate $HfO_2 \cdot ZrO_2$ is also detected in certain cases.

At 1000 °C the Al₂O₃/aluminate spinel forming MAR-M-247, Nimonic-115, SX-R-150 have the best cyclic oxidation resistance. There is no obvious Co effect although there are K_a minimums at 5 Co for MAR-M-247, at 10 Co for Nimonic-115, and at 6 Co for SX-R-150—all less than their commercial cobalt levels. U-700 (cast and P/M hipped), Waspaloy, IN-738 and U-720 all are Cr₂O₃/chromite spinel controlled at 1000 °C. Their oxidation rates are considerably higher than the alumina formers but there is no obvious Co effect.

At 1100 °C MAR-M-247 and Nimonic-115 alloys behave similarly to the 1000 °C test alloy except the K_a values are somewhat higher and critical NiO formation takes place at shorter times. The oxidation resistance is still generally good and again 5 Co is an apparent oxidation minimum for MAR-M-247 while the minimum for Nimonic-115 is also at 5 Co. SX-R-150 while still an apparent Al₂O₃/aluminate spinel former oxidizes massively to NiO and NiWO₄. This apparent anomalous behavior is difficult to explain based strictly on composition. The remaining alloys are basically chromia/chromite spinel formers at 1100 °C although alumina and/or aluminates start to show up with longer times in both the U-700 series alloys. The oxidation resistance of the U-700, Waspaloy, IN-738, and U-720 generally fall into the poor category. The resistance is usually worse at the highest cobalt levels. NiO shows up at 100 hr on the U-700, IN-738, and U-720 series samples while Cr₂O₃ still controls at 100 hr for the Waspaloy. Even at 200 hr Cr₂O₃ still is quite strong for the Waspaloy while the other 3 chromia formers show mostly NiO present. This appears to be due to the high (19.5) Cr level in Waspaloy although U-720 with an 18 Cr level might be expected to behave in a similar manner.

At 1150 °C MAR-M-247 and Nimonic-115 series still show fair cyclic oxidation resistance and are mostly Al₂O₃/aluminate spinel formers. MAR-M-247 starts out as a complex NiO/<3.30 tri-rutile/NiWO₄/Cr₂O₃/HfO₂ former. At 100 hr aluminate spinel/Al₂O₃ shows up as well. The Nimonic-115 series indicates mostly Cr₂O₃/chromite spinel/NiTiO₃ after 1 hr for the 5, 10, and 15 cobalt levels. The 0 Co indicates only Al₂O₃ after 1 hr but chromite spinel along with NiO, Al₂O₃ and aluminate spinel. At the 5, 10, and 15 Co levels only Al₂O₃, aluminate spinel and <3.30 tri-rutile are observed. For both alloy systems the minimum in K_a values are at the 5 Co level.

U-700 (cast and P/M), Waspaloy, IN-738, and U-720 all are strong Cr₂O₃ formers after 1 hr along with <3.30 tri-rutile and in some cases Cr₂Ti₁₃O₂₉ and chromite spinels. At 100 hr U-700 becomes a strong NiO/chromite spinel former but with some Al₂O₃/aluminate spinel showing up as well along with NiWO₄ at the higher cobalt levels. Waspaloy is somewhat similar but without any Al₂O₃/aluminate spinel being detected. For IN-738 NiO becomes dominant along with Cr₂O₃/chromite spinel, and to a lesser extent NiTiO₃ and <3.30 tri-rutile. U-720 is similar to IN-738 except NiWO₄ shows up as well but as a minor phase. In general even for the 100 hr test the K_a value indicates poor to catastrophic oxidation resistance with the poorest resistance at the highest cobalt value. The SX-R-150 alloy series is in the high catastrophic range even though the alloys are initially Al₂O₃/aluminate spinel formers but rapidly forms massive NiO as they did at 1100 °C.

In general the cyclic oxidation behavior tends to fall into two categories. Three of the alloy sets: SX-R-150, Nimonic-115, and MAR-M-247 with Cr to Al ratios of 0.91, 2.98, and 1.49 tend to form Al_2O_3 /aluminate spinels as the controlling oxide. Waspaloy, U-720, and IN-738 with Cr:Al of 15.0, 7.20, and 4.71 tend to form Cr_2O_3 /chromite spinels as the controlling scale. While U-700, with a Cr to Al ratio of 3.49, seems to be close to the borderline as far as oxide control is concerned but usually forms Cr_2O_3 /chromite spinel. These trends tend to be verified by both the derived K_a values and the x-ray diffraction results.

These tendencies are shown schematically in figure 7. With increasing time the tendency of the scales is to break down to NiO. This appears to take longer for the Al_2O_3 /aluminate formers. With increased temperature the time for breakdown to NiO decreases. This tendency for NiO formation is negated slightly by the increased inclination for Al_2O_3 formation vis a vis Cr_2O_3 formation with temperature (ref. 11). With increasing temperature Co seems to accelerate NiO formation when Cr_2O_3 /chromite scales control. For Al_2O_3 /aluminate control Co seems to give consistent minimums in K_a values near 5.0 percent for all three test temperatures. This study also seems to reinforce previous studies conducted at this laboratory that the refractory metals (Cb, Mo, W and particularly Ta) are beneficial at certain optimum concentrations. This may be due to the formation of the tri-rutile structure which appears to stabilize particularly the Al_2O_3 /aluminate spinel oxides (refs. 6, 9, and 10).

CONCLUSIONS

(1) The cyclic oxidation behavior of selected Ni-base γ/γ' superalloys depends primarily on their Cr and Al contents particularly on the Cr/Al ratios.

(2) If the ratios are greater than 3.5, as typified by U-720, Waspaloy and IN-738, the alloys tend to form the protective Cr_2O_3 /chromite spinel while alloys with ratios of <3.0, like Mar-M-247 and Nimonic-115, tend to form protective Al_2O_3 /aluminate spinel. U-700 with a ratio between the two can form either type.

(3) The oxidation attack parameter K_a derived from the specific weight change/time data indicates the Al_2O_3 /aluminate spinel is more protective and longer lasting than the Cr_2O_3 /chromite spinel.

(4) These scales tend to breakdown as increased amounts of NiO are formed.

(5) In these alloys tri-rutile $Ni(Ta, Cb, W, Mo)_2O_6$ was associated with formation of both protective oxides. When present, $Ni(W, Mo)O_4$ seemed to trigger the deleterious NiO. Ta appears particularly beneficial.

(6) Increasing Co levels are associated with higher K_a values and increased NiO formation in the high Cr/Al alloys. In the low Cr/Al alloys a minimum in K_a values occurs near 5 Co at the three test temperatures.

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TABLE I. - NOMINAL COMPOSITIONS AND VARIATIONS IN COBALT LEVELS IN NICKEL-BASE SUPERALLOYS SELECTED FOR THE COSAM PROGRAM

| | Weight percent | | | | | | | | | | | |
|-----------------------|----------------|------|-----|-----|-----|------|-----|-----|------|------|------|-------|
| | Co | Cr | Al | Ti | Mo | W | Ta | Hf | B | Zr | C | Cr/Al |
| U-700 | 18.5 | 15.0 | 4.3 | 3.5 | 5.2 | ---- | --- | --- | 0.03 | ---- | 0.08 | 3.49 |
| Waspaloy | 13.5 | 19.5 | 1.3 | 3.0 | 4.3 | ---- | --- | --- | .006 | .06 | .08 | 15.0 |
| MAR-M-247 | 10.0 | 8.2 | 5.5 | 1.0 | .6 | 10.0 | 3.0 | 1.5 | .020 | .09 | .16 | 1.49 |
| IN-738 | 8.5 | 16.0 | 3.4 | 3.4 | 1.8 | 2.6 | 1.8 | --- | .010 | .10 | .17 | 4.71 |
| Nimonic-115 | 14.0 | 14.6 | 4.9 | 4.0 | 3.5 | ---- | --- | --- | .017 | .001 | .16 | 2.98 |
| U-720 | 14.7 | 18.0 | 2.5 | 5.0 | 3.1 | 1.24 | --- | --- | .031 | .031 | .04 | 7.20 |
| SX-R-150 ^a | 12.0 | 5.0 | 5.5 | --- | 1.0 | 5.0 | 6.0 | --- | ---- | ---- | ---- | .91 |

^a3Re-2.2V

VARIATIONS IN COBALT LEVELS

| | |
|-------------------------|---------------------------|
| U-700-cast | 0.1, 4.3, 8.6, 12.8, 17.0 |
| U-700-powder met/hipped | 0, 4.3, 8.6, 12.8, 17.0 |
| Waspaloy-hot worked | 0, 4.5, 9.0, 13.5 |
| MAR-M-247-cast | 0.1, 5.0, 9.8 |
| IN-738-hot worked | 0, 4.0, 8.0 |
| Nimonic-115-hot worked | 0, 5.0, 10.0, 15.0 |
| U-720-hot worked | 0, 7.5, 14.7 |
| SX-R-150-cast | 0, 6.0, 12.0 |

TABLE II. - SUMMARY OF REGRESSION CURVE FITS FOR SELECTED NICKEL-BASE SUPERALLOYS TESTED IN CYCLIC OXIDATION AT 1000°, 1100° AND 1150 °C

| | 1000 °C | | | 1100 °C | | | 1150 °C | | |
|-------------|-------------|--------|------------|----------------|--------|------------|-------------|--------|------------|
| | Paralin-ear | Linear | Force-lin. | Paralin-ear | Linear | Force-lin. | Paralin-ear | Linear | Force-lin. |
| U-700 cast | 6 | 0 | 0 | 7 | 1 | 1 | 6 | 2 | 0 |
| U-700 P/M | 6 | | | 2 | 3 | 1 | 3 | 2 | 0 |
| Waspaloy | 9 | | | 7 ^a | 0 | 0 | 8 | 1 | 0 |
| MAR-M-247 | 6 | | | 4 | 0 | 3 | 4 | 1 | 2 |
| IN-738 | 3 | | | 3 | 0 | 0 | 3 | 0 | 0 |
| Nimonic-115 | 5 | | | 1 | 2 | 1 | 1 | 0 | 3 |
| U-720 | 4 | | | 3 | 0 | 0 | 3 | 0 | 0 |
| SX-R-150 | 3 | | | 2 | 0 | 1 | 1 | 2 | 0 |
| Totals | 41 | 0 | 0 | 29 | 6 | 7 | 29 | 8 | 5 |

^a2 of 9 samples poor fits-rejected as outliers.

TABLE III. - SUMMARY OF CYCLIC OXIDATION SPECIFIC WEIGHT CHANGE DATA
INCLUDING DERIVED RATE AND ATTACK PARAMETERS AT 1000 °C

| Alloy | Percent Co | Run no. | Specific weight change, mg/cm ² | | | k ₁ ^{1/2} | k ₂ | K _a |
|-------------|------------|---------|--|--------|--------|-------------------------------|----------------------|----------------|
| | | | 100 hr | 200 hr | 500 hr | | | |
| U-700-cast | -0.1 | 424-1 | 3.04 | 2.83 | 0.41 | 0.4851 | 0.02011 | 0.6862 |
| | .1 | 452-2 | 2.85 | 1.57 | -6.73 | .7208 | .04675 | 1.188 |
| | 4.3 | 424-2 | 3.23 | 1.87 | -.67 | .4840 | .02329 | .7169 |
| | 8.6 | 424-3 | 2.53 | 1.25 | -2.24 | .4440 | .02312 | .6751 |
| | 12.8 | 424-4 | 2.27 | .64 | -7.20 | .5657 | .03619 | .9276 |
| | 17.0 | 424-5 | 2.13 | .73 | -4.38 | .4720 | .02892 | .7612 |
| U-700-(P/M) | 0 | 447-2 | 2.87 | 2.88 | 0.30 | 0.5231 | 0.02470 | 0.7701 |
| | 4.3 | 447-3 | 2.29 | 1.35 | -1.04 | .4811 | .02299 | .7110 |
| | 8.55 | 447-4 | 1.60 | .93 | -4.25 | .4170 | .02343 | .6514 |
| | 12.77 | 447-5 | 1.78 | -1.35 | -3.94 | .2912 | .02086 | .4998 |
| | 17.0 | 447-6 | 1.61 | .61 | -7.15 | .4038 | .02434 | .6472 |
| Waspaloy | 0 | 436-3 | ---- | 0.51 | -76.46 | 3.029 | 0.2300 | 5.328 |
| | 0 | 615-6 | 2.75 | 2.65 | -74.13 | 3.514 | .2733 | 6.247 |
| | 4.5 | 436-4 | ---- | 2.42 | -77.84 | 3.472 | .2591 | 6.062 |
| | 4.5 | 480-4 | 3.08 | 2.97 | -70.03 | 3.663 | .2900 | 6.563 |
| | 9.0 | 436-5 | ---- | 1.28 | -92.16 | 4.356 | .3398 | 7.754 |
| | 9.0 | 480-5 | 2.84 | 3.64 | -74.49 | 3.662 | .2852 | 6.514 |
| | 13.5 | 436-6 | ---- | 1.62 | -75.73 | 3.668 | .2862 | 6.530 |
| | 13.5 | 480-6 | 2.42 | 2.77 | -57.36 | 3.061 | .2450 | 5.511 |
| | 13.5 | 615-5 | 1.34 | 2.15 | -44.80 | 1.702 | .1253 | 2.955 |
| MAR-M-247 | 0.1 | 452-3 | 0.33 | 0.42 | 0.50 | 0.04138 | 0.0008771 | 0.05015 |
| | .1 | 480-1 | .31 | .40 | 0.18 | .05390 | .002017 | .07408 |
| | 5 | 452-4 | .27 | .31 | .30 | .03696 | .001094 | .04789 |
| | 5 | 480-2 | .28 | .27 | .42 | .03170 | .0008846 | .04055 |
| | 9.76 | 452-5 | .33 | .43 | .46 | .04708 | .001215 | .05923 |
| | 9.76 | 480-3 | .30 | .27 | .24 | .03427 | .001221 | .04648 |
| IN-738 | 0 | 674-1 | 2.86 | 2.52 | -12.38 | 1.005 | 0.06670 | 1.672 |
| | 4 | 674-2 | 3.19 | 1.89 | -11.28 | .9427 | .06107 | 1.553 |
| | 8 | 674-3 | 3.68 | 1.52 | -12.65 | 1.028 | .06705 | 1.698 |
| Nimonic-115 | 0 | 675-1 | 0.05 | -0.17 | -1.44 | 0.06846 | 0.005765 | 0.1261 |
| | 5 | 675-2 | 1.21 | 1.03 | -3.35 | .3182 | .01786 | .4967 |
| | 10 | 675-3 | .50 | .55 | .65 | .06514 | .001709 | .08222 |
| | 15 | 675-4 | 1.04 | -.19 | -1.47 | .1930 | .01242 | .3171 |
| | 15 | 675-5 | 1.14 | 1.18 | -4.60 | .3220 | .01779 | .4999 |
| U-720 | 0 | 674.4 | 2.25 | -8.77 | -70.44 | 2.915 | 0.2690 | 5.604 |
| | 7.5 | 674.5 | 2.77 | -.56 | -56.84 | 2.847 | .2335 | 5.182 |
| | 14.7 | 674.6 | 2.80 | -14.98 | -77.59 | 2.959 | .2821 | 5.776 |
| | 14.7 | 675-6 | -.85 | -7.34 | -93.57 | 3.569 | .3431 | 7.000 |
| SX-R-150 | 0 | 615-1 | 0.16 | 0.27 | -4.94 | 0.2002 | 0.1463 | 0.3465 |
| | 6 | 615-2 | .16 | .14 | .25 | .01349 | Not sig ^a | .01349 |
| | 12 | 615-3 | .31 | .13 | -16.78 | .6394 | .05139 | 1.153 |

^aSpecial parabolic case (i.e., parabolic).

TABLE IV. - SUMMARY OF CYCLIC OXIDATION SPECIFIC WEIGHT CHANGE DATA
INCLUDING DERIVED RATE AND ATTACK PARAMETERS AT 1100 °C

| Alloy | Percent Co | Run no. | Specific weight change, mg/cm ² | | | k ₁ ^{1/2} | k ₂ | K _a |
|-------------|------------|---------|--|----------------------|---------|-------------------------------|----------------|----------------|
| | | | 100 hr | 200 hr | 500 hr | | | |
| U-700-cast | ~0.1 | 422-1 | -11.69 | -23.59 | | 0.8799 | 0.1879 | 2.759 |
| | .1 | 453-2 | -20.54 | -60.17 | | 1.455 | .3826 | 5.281 |
| | 4.3 | 422-2 | -11.43 | -23.97 | | ----- | .1143 | 2.286 |
| | 8.6 | 422-3 | -20.39 | -48.90 | | .4837 | .2639 | 3.123 |
| | 12.8 | 422-4 | -31.65 | -87.71 | | ----- | .3530 | 7.060 |
| | 17.0 | 422-5 | -41.59 | -174.23 | | 8.551 | 1.405 | 22.60 |
| | 17.0 | 437-2 | -38.13 | -186.38 | | 10.93 | 1.662 | 27.54 |
| | 17.0 | 610-5 | -25.52 | -53.16 | | ----- | .2383 | 4.765 |
| | 655-5 | -10.82 | -111.17 | | 7.377 | .9441 | 16.82 | |
| U-700-P/M | 0 | 448-2 | -13.02 | -33.62 | | ----- | 0.1465 | 2.929 |
| | 4.3 | 448-3 | -14.17 | -37.19 | | ----- | .1544 | 3.089 |
| | 8.6 | 448-4 | -12.15 | -39.47 | | ----- | .1565 | 3.131 |
| | 12.8 | 448-5 | -14.93 | -109.76 | | 5.641 | .8016 | 13.66 |
| | 17.0 | 448-6 | -12.10 | -108.30 | | 5.677 | .7721 | 13.40 |
| | 17.0 | 610-6 | -13.34 | -17.31 | | ----- | .1048 | 2.096 |
| Waspaloy | 0 | 437-3 | -4.04 | -24.26 | | 1.512 | 0.2038 | 3.550 |
| | 0 | 614-6 | -.92 | -4.57 | | .3660 | .04550 | .8210 |
| | 4.5 | 437-4 | -4.19 | -28.77 | | 1.843 | .2431 | 4.274 |
| | 4.5 | 481-4 | -2.28 | -27.15 | | 2.195 | .2632 | 4.827 |
| | 9.0 | 437-5 | -6.85 | -49.97 | | 3.564 | .4604 | 8.168 |
| | 9.0 | 481-5 | -.22 | +1.11 | | - poor fit - | ----- | ----- |
| | 13.5 | 437-6 | -2.86 | -23.91 | | 1.663 | .2097 | 3.760 |
| | 13.5 | 481-6 | .10 | +3.39 | | - poor fit - | ----- | ----- |
| | 13.5 | 614-5 | -.72 | -14.48 | | 1.099 | .1271 | 2.371 |
| | MAR-M-247 | 0.1 | 453-3 | -3.36 | -7.38 | | 0.1437 | 0.04744 |
| .1 | | 481-1 | .27 | .12 | | .08980 | .005592 | .1457 |
| 5 | | 453-4 | -2.49 | -5.05 | | .04398 | .02908 | .2543 |
| 5 | | 481-2 | .07 | -.31 | | .09107 | .007895 | .1700 |
| 9.76 | | 453-5 | -2.98 | -5.30 | | ----- | .02800 | .5600 |
| 9.76 | | 481-3 | -2.61 | -4.92 | | .07888 | .03204 | .3997 |
| 9.76 | | 657-1 | -1.04 | -3.50 | | .2228 | .03335 | .5564 |
| IN-738 | | 0 | 662-6 | -30.99 | -139.07 | | 7.243 | 1.096 |
| | 4 | 664-1 | -4.44 | -52.50 | | 4.160 | .5094 | 9.254 |
| | 8 | 664-2 | -45.88 | -199.49 | | 11.94 | 1.820 | 30.14 |
| Nimonic-115 | 0 | 664-3 | -8.85 | -20.28 | | 0.3654 | 0.1267 | 1.633 |
| | 5 | 664-4 | -.43 | -.99 | | ----- | .004836 | .0967 |
| | 10 | 664-5 | -3.13 | -6.13 | | ----- | .03142 | .6284 |
| | 15 | 664-6 | -.95 | -1.01 | | ----- | .00712 | .1425 |
| U-720 | 0 | 655-1 | -83.50 | -150.82 | | 4.697 | 1.150 | 16.19 |
| | 7.5 | 655-2 | -98.23 | -242.90 | | 10.17 | 19.65 | 29.82 |
| | 14.7 | 655-3 | -135.17 | -313.46 | | 9.556 | 2.278 | 32.33 |
| SX-R-150 | 0 | 614-1 | -177.91 | -243.81 | | ----- | 1.404 | 28.07 |
| | 6 | 614-2 | -24.96 | -180.74 | | 13.36 | 1.797 | 31.33 |
| | 12 | 614-3 | -381.61 | -596.41 ^a | | 3.777 | 4.124 | 45.01 |

^aValue at 160 Hr-test terminated

TABLE V. - SUMMARY OF CYCLIC OXIDATION SPECIFIC WEIGHT CHANGE DATA
INCLUDING DERIVED RATE AND ATTACK PARAMETERS AT 1150 °C

| Alloy | Percent Co | Run no. | Specific weight change, mg/cm ² | | | $k_1^{1/2}$ | k_2 | K_a |
|-------------|------------|---------|--|--------|--------|-------------|--------|--------|
| | | | 100 hr | 200 hr | 500 hr | | | |
| U-700-cast | ~0.1 | 423-1 | -30.50 | | | ----- | 0.2570 | 5.140 |
| | .1 | 454-2 | -34.79 | | | ----- | .2919 | 5.839 |
| | 4.3 | 423-2 | -50.07 | | | 2.333 | .6793 | 9.127 |
| | 8.6 | 423-3 | -104.39 | | | 6.677 | 1.501 | 21.69 |
| | 12.8 | 423-4 | -197.77 | | | 16.33 | 3.513 | 51.46 |
| | 17.0 | 423-5 | -230.72 | | | 16.44 | 3.922 | 55.66 |
| | 17.0 | 438-2 | -243.21 | | | 15.21 | 3.965 | 54.86 |
| | 17.0 | 654-5 | -214.42 | | | 14.14 | 3.559 | 49.72 |
| U-700-(P/M) | 0 | 449-2 | -31.85 | | | ----- | 0.2692 | 5.384 |
| | 4.3 | 449-3 | -50.76 | | | ----- | .3984 | 7.968 |
| | 8.55 | 449-4 | -64.70 | | | 3.565 | .9398 | 12.96 |
| | 12.77 | 449-5 | -142.16 | | | 11.64 | 2.395 | 35.59 |
| | 17.0 | 449-6 | -174.85 | | | 15.04 | 3.100 | 46.04 |
| Waspaloy | 0 | 438-3 | -94.17 | | | 8.070 | 1.692 | 24.99 |
| | 0 | 613-6 | -155.16 | | | ----- | 1.554 | 31.08 |
| | 4.5 | 438-4 | -103.75 | | | 9.172 | 1.893 | 28.10 |
| | 4.5 | 482-4 | -139.96 | | | 14.03 | 2.696 | 40.98 |
| | 9.0 | 438-5 | -186.13 | | | 15.33 | 3.311 | 48.44 |
| | 9.0 | 482-5 | -9.99 | | | 1.330 | .2268 | 3.598 |
| | 13.5 | 438-6 | -165.20 | | | 14.14 | 2.919 | 43.33 |
| | 13.5 | 482-6 | -27.53 | | | 3.583 | .6077 | 9.660 |
| | 13.5 | 613-5 | -226.73 | | | 19.24 | 4.155 | 60.80 |
| MAR-M-247 | 0.1 | 454-3 | -15.26 | | | ----- | 0.1596 | 3.193 |
| | .1 | 482-1 | .16 | | | .1548 | .01463 | .3012 |
| | 5 | 454-4 | -7.95 | | | ----- | .09237 | 1.847 |
| | 5 | 482-2 | -2.65 | | | ----- | .02734 | .5468 |
| | 9.76 | 454-5 | -19.46 | | | .4067 | .2250 | 2.657 |
| | 9.76 | 482-3 | -30.86 | | | 1.146 | .4054 | 5.200 |
| | 9.76 | 656-1 | -.30 | | | .07611 | .01139 | .1901 |
| IN-738 | 0 | 661-6 | -116.59 | | | 6.789 | 1.661 | 23.40 |
| | 4 | 663-1 | -187.51 | | | 15.20 | 3.288 | 48.08 |
| | 8 | 663-2 | -183.28 | | | 13.57 | 3.106 | 44.63 |
| Nimonic-115 | 0 | 663-3 | -32.78 | | | 0.9616 | 0.4246 | 5.207 |
| | 5 | 663-4 | -4.13 | | | ----- | .04835 | .9670 |
| | 10 | 663-5 | -8.93 | | | ----- | .1045 | 2.091 |
| | 15 | 663-6 | -7.24 | | | ----- | .08198 | 1.640 |
| U-720 | 0 | 654-1 | -206.15 | | | 12.26 | 3.326 | 45.52 |
| | 7.5 | 654-2 | -245.92 | | | 9.295 | 3.453 | 43.82 |
| | 14.7 | 654-3 | -313.42 | | | 4.811 | 3.676 | 41.58 |
| SX-R-150 | 0 | 613-1 | -296.50 | | | ----- | 2.998 | 59.97 |
| | 6 | 613-2 | -427.42 | | | ----- | 4.357 | 87.13 |
| | 12 | 613-3 | -667.04 ^a | | | ----- | 15.74 | 314.86 |

^aTest discontinued after 45 hr.

TABLE VI. - SUMMARY OF X-RAY DIFFRACTION RESULTS OF SELECT Ni-BASE SUPERALLOYS
 AT 1000 °C FOR SURFACE OXIDE PHASES AFTER VARIOUS EXPOSURE TIMES IN CYCLIC
 OXIDATION. PHASE LISTED IN DECREASING ORDER OF INTENSITY

| Alloy set | Percent Co | 1 hr | 100 hr | 200 hr | 500 hr |
|-----------------|------------|-------------------------|-------------------------------|-----------------------------|---------------------------------|
| U-700 (cast) | ~0.1 | 1,3 | 1,4,3 | 1,4,6,3 | 1,6,4,2 |
| | 4.3 | 1,3,2 | 1,4 | 1,4 | 1,4 |
| | 8.6 | 1,3,2 | 1,4,3 | 1,4,3 | 1,4 |
| | 12.8 | 1,3,2 | 1,4,3 | 1,3,4 | 1,4 |
| | 17.0 | 1,3,2 | 1,3,4 | 1,4,3 | 1 |
| U-700 (P/M) | 0 | 1,3,4 | 1,3,4 | 1,3,4 | 1,2,6,3 |
| | 4.3 | 1,3 | 1,3,4 | 1,3 | 1,6,4,3 |
| | 8.55 | 1,3 | 1,3,4 | 1,3 | 1,6,4,3 |
| | 12.77 | 1,3 | 1,3 | 1,2,6,3 | 1,4,2,6,3 |
| | 17.0 | 1,3 | 1,3,4 | 1,3 | 6,1,2,5,3 |
| Waspaloy | 0 | 1,3 | 1,4 | 1,4 | 6,1,4 |
| | 4.5 | 1,3 | 1,3,4 | 1,4,3,6 | 6,1,4 |
| | 9.0 | 1,3 | 1,3,4 | 1,3,4 | 6,1,4 |
| | 13.5 | 1,3 | 1,4 | 1,3,7 | 6,1,4 |
| MAR-M-247 | 0.1 | 7/ ^a 7,2,1 | 2,1,7,3/ ^a 2,4,7,6 | 2,4,3/ ^a 2,1,7,3 | 2,7,3,1/ ^a 2,6,1,3,7 |
| | 5 | 7/ ^a 7,2,1,3 | 2,7,3,1/ ^a 2,4,7,1 | 2,7,4,3 | 2,7,3,1 |
| | 9.76 | 7,2,1,3 | 2,7,3,1,4 | 2,7,3,1 | 2,7,3,1 |
| IN-738 | 0 | 1,3 | 1,3 | 1,3 | 1,3,6 |
| | 4 | 1,3 | 1,3,4 | 1,3,4 | 1,3 |
| | 8 | 1,3 | 1,3,4 | 1,3,4 | 1,6,3,2 |
| Nimonic-115 | 0 | 2,1 | 2,1,4 | 1,2,4 | 2,1 |
| | 5 | 1,3 | 2,1,3,4 | 1,4,2,3 | 6,1 |
| | 10 | 1,3,2 | 2,1,3 | 2,3 | 3,2 |
| | 15 | 1,3,2 | 3,2,1,4 | 4,1,3 | 4,2,1,3/ ^a 6,1,4 |
| U-720 | 0 | 1,3 | 1,3 | 1,6,3 | 6,4,1,3 |
| | 7.5 | 1,3 | 1,3 | 1,3,6 | 6,4,1,3 |
| | 14.7 | 1,3 | 1,3,4 | 1,6,3 | 6,1,4,3 |
| SX-R-150 | 0 | 2,3,1 | 2,3/ ^a 2,3,6 | 2,3 | 6,2,3 |
| | 6 | 3,2,1 | 2,3 | 2,3 | 2,3 |
| | 12 | 2,3,1 | 2,3 | 2,3 | 2,6,3,1 |

Phases: 1 Cr₂O₃/Chromite spinel
 2 Al₂O₃/Aluminate spinel
 3 Tri-rutile-Ni(Ta,Cb,W,Mo)₂O₆
 4 Titanates-(Ni,Co,Fe)TiO₃;Cr₂Ti₁₃O₂₉;TiO₂
 5 Ni(W,Mo)O₄
 6 NiO
 7 Misc oxides - ZrO₂,HfO₂

^aReplicate samples - significantly different results

TABLE VII. - SUMMARY OF X-RAY DIFFRACTION RESULTS OF SELECT Ni-BASE SUPERALLOYS
AT 1100 °C FOR SURFACE OXIDE PHASES AFTER VARIOUS EXPOSURE TIMES IN CYCLIC
OXIDATION. PHASE LISTED IN DECREASING ORDER OF INTENSITY

| Alloy set | Percent Co | 1 hr | 100 hr | 200 hr | 500 hr |
|-----------------|------------|-----------------------------|---------------------------------|-------------------------------|--------|
| U-700 (cast) | ~0.1 | 1,3 | 2,6,1,3,4 | 2,4,6,1,3 | |
| | 4.3 | 1,3 | 2,4,6,3 | 2,4,3,6,1 | |
| | 8.6 | 1,3 | 2,4,6,1,3 | 2,4,6,3,1 | |
| | 12.8 | 1,3 | 2,4,6,1 | 4,2,1,6,3 | |
| | 17.0 | 1,3 | 1,6,2,4,3 | 6,1,4/ ^a 6,1,4,3,2 | |
| U-700 (P/M) | 0 | 1,3 | 2,6,4,1,3 | 2,4,1,6 | |
| | 4.3 | 1,3 | 2,4,6,3,1 | 2,4,6,1 | |
| | 8.55 | 1,3 | 2,4,6,3,1 | 6,4,1,2,3 | |
| | 12.77 | 1,3 | 4,2,1,6,3 | 6,1,5 | |
| | 17.0 | 1,3 | 4,2,1,6,3 | 6,1,4/ ^a 4,6,2,1,3 | |
| Waspaloy | 0 | 1,3,4 | 1,4 | 1,3,6/ ^a 1,3,4 | |
| | 4.5 | 1,3,4 | 1,4,3 | 1,3,6 | |
| | 9.0 | 1,3 | 1,4 | 1,3,6 | |
| | 13.5 | 1,3 | 1,4 | 1,3,6 | |
| MAR-M-247 | 0.1 | 6,1,5,3,7 | 2,6,3,7,5/ ^a 2,3,7,4 | 2,6,5,3,7/ ^a 2,3,7 | |
| | 5 | 1,5,3,7 | 2,3,6,7/ ^a 2,3,7,5 | 2,6,5,3,7/ ^a 2,3,7 | |
| | 9.76 | 1,5,3,7/ ^a 3,7,1 | 2,3,1,7,5 | 2,7,6 | |
| In-738 | 0 | 1,3 | 6,1,4,3 | 6,1,3,2 | |
| | 4 | 1,3 | 1,3 | 6,1,3 | |
| | 8 | 1,3 | 6,1,3 | 6,1,4,3 | |
| Nimonic-115 | 0 | 2, | 2,1 | 1,2 | |
| | 5 | 1,3 | 2,3 | 2,1,3 | |
| | 10 | 1,3 | 2,3 | 2,3 | |
| | 15 | 1,3 | 2,3 | 2,3 | |
| U-720 | 0 | 1,3 | 6,4,1,3 | 6,1,4,2,5,3 | |
| | 7.5 | 1,3 | 6,1,4,3 | 6,1,4 | |
| | 14.7 | 1,3 | 6,1,4,3 | 6,1,4,3 | |
| SX-R-150 | 0 | 2,3,6 | 6,5,2,3 | 6,2,5,3 | |
| | 6 | 6,2,7 | 6,2,7,5 | 6,2,7 | |
| | 12 | 6,2,7 | 6,5,2,7 | 6,2,5,3 ^b | |

- Phases: 1 Cr₂O₃/Chromite spinel
2 Al₂O₃/Aluminate spinel
3 Tri-rutile-Ni(Ta,Cb,W,Mo)₂O₆
4 Titanates-(Ni,Co,Fe)TiO₃;Cr₂Ti₁₃O₂₉;TiO₂
5 Ni(W,Mo)O₄
6 NiO
7 Misc oxides - ZrO₂,HfO₂

^aReplicate samples - significantly different results

^bTest terminated - 160 hr

TABLE VIII. - SUMMARY OF X-RAY DIFFRACTION RESULTS OF SELECT Ni-BASE SUPERALLOYS AT 1150 °C FOR SURFACE OXIDE PHASES AFTER VARIOUS EXPOSURE TIMES IN CYCLIC OXIDATION. PHASE LISTED IN DECREASING ORDER OF INTENSITY

| Alloy set | Percent Co | 1 hr | 100 hr | 200 hr | 500 hr |
|-----------------|------------|---------------------------------|-----------------------------|--------|--------|
| U-700 (cast) | ~0.1 | 1,3 | 6,4,1,2,3 | | |
| | 4.3 | 1,3 | 6,4,1,2 | | |
| | 8.6 | 1,3 | 6,1,4,5,2 | | |
| | 12.8 | 1,3 | 6,1,2,5 | | |
| | 17.0 | 1,3 | 6,1,2,5 | | |
| U-700 (P/M) | 0 | 1,3 | 6,4,1 | | |
| | 4.3 | 1,3 | 6,4,1 | | |
| | 8.55 | 1,4,3 | 6,4,1 | | |
| | 12.77 | 1,3 | 6,1,5 | | |
| | 17.0 | 1,3,4 | 6,1,5 | | |
| Waspaloy | 0 | 1,3 | 1,6,3 | | |
| | 4.5 | 1,3,4 | 1,6,3/ ^a 6,1,5 | | |
| | 9.0 | 1,3,4 | 6,1,4,5/ ^a 6,1 | | |
| | 13.5 | 1,3,4 | 6,1,5 | | |
| MAR-M-247 | 0.1 | 6,3,5,1,7/ ^a 1,5,3,7 | 2,3,7,4,1 | | |
| | 5 | 6,3,5,1,7/ ^a 1,5,3 | 2,3,7/ ^a 2,6,7,1 | | |
| | 9.76 | 1,3,5,7 | 2,3,7,6,5 | | |
| In-738 | 0 | 1,3 | 6,1,4,3 | | |
| | 4 | 1,3 | 6,1,4,3 | | |
| | 8 | 1,3 | 6,1,4,3 | | |
| Nimonic-115 | 0 | 2 | 1,2,6 | | |
| | 5 | 1,4,2,3,2,6,7 | 2,3 | | |
| | 10 | 1,7,4,2 | 2,3 | | |
| | 15 | 1,3,4 | 2,3 | | |
| U-720 | 0 | 1,3,7 | 6,4,1,2,5,3 | | |
| | 7.5 | 1,3 | 6,1,4,2,5,3 | | |
| | 14.7 | 1,3 | 6,1,4,5,3 | | |
| SX-R-150 | 0 | 2,3 | 6,5,2,3 | | |
| | 6 | 6 | 2,6,5,3 | | |
| | 12 | 6,2,3 | 6 | | |

Phases: 1 Cr₂O₃/Chromite spinel
 2 Al₂O₃/Aluminate spinel
 3 Tri-rutile-Ni(Ta,Cb,W,Mo)₂O₆
 4 Titanates-(Ni,Co,Fe)TiO₃;Cr₂Ti₁₃O₂₉;TiO₂
 5 Ni(W,Mo)O₄
 6 NiO
 7 Misc oxides - ZrO₂,HfO₂; Tri-rutiles > 3.30
^aReplicate samples - significantly different results

TABLE A-I. - SUMMARY OF STATISTICAL REGRESSION ANALYSIS RESULTS OF SPECIFIC WEIGHT CHANGE CYCLIC OXIDATION DATA FOR SELECTED ALLOYS AT 1000 °C. REJECTION LEVEL AT 0.90 LENGTH OF TEST-500. ONE HOUR EXPOSURE CYCLES.

| Alloy | Percent Co | Run No. | $k_1^{1/2}$ | k_2 | R^2 | S.E.E | Final $\Delta W/A$ values, mg/cm^2 | |
|-------------|------------|---------|-------------|-----------------------|-------|-------|--------------------------------------|------------|
| | | | | | | | Observed | Calculated |
| U-700-cast | ~0.1 | 424-1 | 0.4851 | 0.02011 | 0.995 | 0.18 | 0.41 | 0.79 |
| | .1 | 452-2 | .7208 | .04675 | .949 | .72 | -6.73 | -7.26 |
| | 4.3 | 424-2 | .4840 | .02329 | .967 | .38 | -.67 | -.82 |
| | 8.6 | 424-3 | .4440 | .02312 | .956 | .37 | -2.24 | -1.63 |
| | 12.8 | 424-4 | .5657 | .03619 | .931 | .65 | -7.20 | -5.45 |
| | 17.0 | 424-5 | .4720 | .02892 | .979 | .28 | -4.38 | -3.91 |
| U-700-(P/M) | 0 | 447-2 | 0.5231 | 0.02470 | 0.953 | 0.51 | 0.30 | -0.66 |
| | 4.3 | 447-3 | .4811 | .02299 | .902 | .54 | -1.04 | -1.92 |
| | 8.55 | 447-4 | .4170 | .02343 | .833 | .71 | -4.25 | -2.39 |
| | 12.77 | 447-5 | .2912 | .02086 | .781 | .81 | -3.94 | -3.92 |
| | 17.0 | 447-6 | .4038 | .02434 | .634 | 1.19 | -7.15 | -3.14 |
| Waspaloy | 0 | 436-3 | 3.029 | 0.2300 | 0.744 | 11.57 | -76.46 | -29.20 |
| | 0 | 615-6 | 3.514 | .2733 | .834 | 9.99 | -74.13 | -58.10 |
| | 4.5 | 436-4 | 3.472 | .2591 | .734 | 13.01 | -77.84 | -51.90 |
| | 4.5 | 480-4 | 3.663 | .2900 | .885 | 8.77 | -70.03 | -63.07 |
| | 9.0 | 436-5 | 4.356 | .3398 | .852 | 12.64 | -92.16 | -72.48 |
| | 9.0 | 480-5 | 3.662 | .2852 | .848 | 9.92 | -74.49 | -60.68 |
| | 13.5 | 436-6 | 3.668 | .2862 | .854 | 10.56 | -75.73 | -61.11 |
| | 13.5 | 480-6 | 3.061 | .2450 | .900 | 6.96 | -57.36 | -54.04 |
| | 13.5 | 615-5 | 1.702 | .1253 | .667 | 6.75 | -44.80 | -24.61 |
| | | | | | | | | |
| MAR-M-247 | 0.1 | 452-3 | 0.04138 | 0.0008771 | 0.988 | 0.04 | 0.50 | 0.49 |
| | .1 | 480-1 | .05390 | .002017 | .976 | .05 | .18 | .20 |
| | 5 | 452-4 | .03696 | .001094 | .981 | .04 | .30 | .28 |
| | 5 | 480-2 | .03170 | .0008846 | .913 | .08 | .42 | .27 |
| | 9.76 | 452-5 | .04708 | .001215 | .991 | .04 | .46 | .45 |
| | 9.76 | 480-3 | .03427 | .001221 | .954 | .05 | .24 | .16 |
| IN-738 | 0 | 674-1 | 1.005 | 0.06670 | 0.921 | 1.31 | -12.38 | -10.87 |
| | 4 | 674-2 | .9427 | .06107 | .947 | .96 | -11.28 | -9.46 |
| | 8 | 674-3 | 1.028 | .06705 | .953 | .95 | -12.65 | -10.54 |
| Nimonic-115 | 0 | 675-1 | 0.06846 | 0.005765 | 0.968 | 0.10 | -1.44 | -1.35 |
| | 5 | 675-2 | .3182 | .01786 | .762 | .67 | -3.36 | -1.82 |
| | 10 | 675-3 | .06514 | .001709 | .990 | .05 | .65 | .60 |
| | 15 | 675-4 | .1930 | .01242 | .825 | .38 | -1.47 | -1.89 |
| | 15 | 675-5 | 0.3220 | 0.01779 | 0.542 | 1.12 | -4.60 | -1.70 |
| U-720 | 0 | 674-4 | 2.915 | 0.2690 | 0.984 | 3.49 | -70.44 | -69.32 |
| | 7.5 | 674-5 | 2.847 | .2335 | .890 | 7.25 | -56.84 | -53.06 |
| | 14.7 | 674-6 | 2.959 | .2821 | .978 | 4.48 | -77.59 | -74.94 |
| | 14.7 | 675-6 | 3.569 | .3431 | .973 | 6.10 | -93.57 | -91.75 |
| SX-R-150 | 0 | 615-1 | 0.2002 | 0.01463 | 0.686 | 0.75 | -4.94 | -2.84 |
| | 6 | 615-2 | .01349 | Not sig. ^a | .920 | .05 | .25 | .30 |
| | 12 | 615-3 | .6394 | .05139 | .827 | 2.01 | -16.78 | -11.40 |

^aSpecial parabolic case (i.e., parabolic).

TABLE A-II. - SUMMARY OF STATISTICAL REGRESSION ANALYSIS RESULTS OF SPECIFIC WEIGHT CHANGE CYCLIC OXIDATION DATA FOR SELECTED ALLOYS AT 1100 °C. REJECTION LEVEL AT 0.90 LENGTH OF TEST-200. ONE HOUR EXPOSURE CYCLES.

| Alloy | Percent Co | Run No. | $k_1^{1/2}$ | k_2 | R^2 | S.E.E | Final $\Delta W/A$ values, mg/cm^2 | |
|-------------|------------|---------|-------------|---------|-------|-------|--------------------------------------|------------|
| | | | | | | | Observed | Calculated |
| U-700-cast | ~0.1 | 422-1 | 0.8799 | 0.1879 | 0.988 | 1.65 | -23.59 | -25.13 |
| | .1 | 453-2 | 1.455 | .3826 | .992 | 2.98 | -60.17 | -55.94 |
| | 4.3 | 422-2 | -Force- | .1143 | .986 | 1.68 | -23.97 | -22.86 |
| | 8.6 | 422-3 | .4837 | .2639 | .996 | 1.83 | -48.89 | -45.94 |
| | 12.8 | 422-4 | Not sig. | .3530 | .978 | 6.62 | -87.71 | -70.60 |
| | 17.0 | 422-5 | 8.551 | 1.405 | .982 | 12.51 | -174.2 | -160.1 |
| | 17.0 | 437-2 | 10.93 | 1.662 | .982 | 13.46 | -88.97 | -91.09 |
| | 17.0 | 610-5 | Not sig. | .2383 | .993 | 2.54 | -53.16 | -47.65 |
| | 17.0 | 655-5 | 7.377 | .9441 | .914 | 13.93 | -111.2 | -84.50 |
| U-700-P/M | 0 | 448-2 | Not sig. | 0.1465 | 0.984 | 2.34 | -33.62 | -29.29 |
| | 4.3 | 448-3 | Not sig. | .1544 | .970 | 3.38 | -37.19 | -30.89 |
| | 8.6 | 448-4 | Not sig. | .1565 | .961 | 3.91 | -39.47 | -31.31 |
| | 12.8 | 448-5 | 5.641 | .8016 | .906 | 14.34 | -109.8 | -80.54 |
| | 17.0 | 448-6 | 5.677 | .7721 | .869 | 15.74 | -108.3 | -74.14 |
| | 17.0 | 610-6 | -Force- | .1048 | .931 | 3.52 | -17.31 | -20.96 |
| Waspaloy | 0 | 437-3 | 1.512 | 0.2038 | 0.950 | 2.43 | -24.26 | -19.37 |
| | 0 | 614-6 | .3660 | .04550 | .938 | .54 | -4.57 | -3.92 |
| | 4.5 | 437-4 | 1.843 | .2431 | .941 | 3.08 | -28.77 | -22.56 |
| | 4.5 | 481-4 | 2.195 | .2632 | .934 | 3.03 | -27.16 | -21.60 |
| | 9.0 | 437-5 | 3.564 | .4604 | .961 | 4.51 | -49.97 | -41.68 |
| | 9.0 | 481-5 | -Poor fit- | | | | | |
| | 13.5 | 437-6 | 1.663 | .2097 | .930 | 2.72 | -23.91 | -18.42 |
| | 13.5 | 481-6 | -Poor fit- | | | | | |
| | 13.5 | 614-5 | 1.099 | .1271 | .869 | 2.01 | -14.48 | -9.88 |
| MAR-M-247 | 0.1 | 453-3 | 0.1437 | 0.04744 | 0.998 | 0.20 | -7.38 | -7.46 |
| | .1 | 481-1 | .08980 | .005592 | .900 | .10 | .12 | .15 |
| | 5 | 453-4 | .04398 | .02908 | .996 | .21 | -5.05 | -5.19 |
| | 5 | 481-2 | .09107 | .007895 | .757 | .11 | -.31 | -.29 |
| | 9.76 | 453-5 | Not sig. | .02800 | .993 | .30 | -5.30 | -5.60 |
| | 9.76 | 481-3 | .07888 | .03204 | .993 | .27 | -4.92 | -5.29 |
| | 9.76 | 657-1 | .2228 | .03335 | .998 | .09 | -3.50 | -3.52 |
| IN-738 | 0 | 662-6 | 7.243 | 1.096 | 0.972 | 11.16 | -139.1 | -116.8 |
| | 4 | 664-1 | 4.160 | .5094 | .949 | 5.33 | -52.50 | -43.06 |
| | 8 | 664-2 | 11.94 | 1.820 | .992 | 10.01 | -199.5 | -195.1 |
| Nimonic-115 | 0 | 664-3 | 0.3654 | 0.1267 | 0.999 | 0.20 | -20.28 | -20.18 |
| | 5 | 664-4 | Not sig. | .004836 | .915 | .18 | -.99 | -.97 |
| | 10 | 664-5 | Not sig. | .03142 | .992 | .36 | -6.13 | -6.28 |
| | 15 | 664-6 | -Force- | .007126 | .736 | .53 | -1.01 | -1.43 |
| U-720 | 0 | 655-1 | 4.697 | 1.150 | 0.989 | 10.43 | -150.8 | -163.5 |
| | 7.5 | 655-2 | 10.17 | 1.965 | .996 | 8.73 | -242.9 | -249.2 |
| | 14.7 | 665-3 | 9.556 | 2.278 | .999 | 6.67 | -313.5 | -320.4 |
| SX-R-150 | 0 | 614-1 | -Force- | 1.404 | 0.982 | 23.78 | -243.8 | -280.7 |
| | 6 | 614-2 | 13.36 | 1.797 | .979 | 13.64 | -180.7 | -170.4 |
| | 12 | 614-3 | 3.777 | 4.124 | .999 | 8.52 | -596.4 | -612.0 |

TABLE A-III. - SUMMARY OF STATISTICAL REGRESSION ANALYSIS RESULTS OF SPECIFIC WEIGHT CHANGE CYCLIC OXIDATION DATA FOR SELECTED ALLOYS AT 1100 °C. REJECTION LEVEL AT 0.90 LENGTH OF TEST-200. ONE HOUR EXPOSURE CYCLES.

| Alloy | Percent Co | Run No. | $k_1^{1/2}$ | k_2 | R^2 | S.E.E | Final $\Delta W/A$ values, mg/cm^2 | |
|-------------|------------|---------|-------------|--------|-------|-------|--------------------------------------|------------|
| | | | | | | | Observed | Calculated |
| U-700-cast | ~0.1 | 423-1 | Not sig. | 0.2570 | 0.964 | 3.27 | -30.50 | -25.70 |
| | .1 | 454-2 | Not sig. | .2919 | .965 | 3.67 | -34.79 | -29.19 |
| | 4.3 | 423-2 | 2.333 | .6793 | .977 | 4.54 | -50.07 | -44.66 |
| | 8.6 | 423-3 | 6.677 | 1.501 | .945 | 19.79 | -104.4 | -83.32 |
| | 12.8 | 423-4 | 16.33 | 3.513 | .981 | 21.60 | -197.8 | -188.1 |
| | 17.0 | 423-5 | 16.44 | 3.922 | .992 | 13.08 | -230.7 | -227.8 |
| | 17.0 | 438-2 | 15.21 | 3.965 | .995 | 11.12 | -243.2 | -244.4 |
| | 17.0 | 654-5 | 14.14 | 3.559 | .995 | 10.35 | -214.4 | -214.5 |
| U-700-P/M | 0 | 449-2 | Not sig. | 0.2692 | 0.967 | 3.29 | -31.85 | -26.92 |
| | 4.3 | 449-3 | Not sig. | .3984 | .936 | 6.85 | -50.76 | -39.84 |
| | 8.55 | 449-4 | 3.565 | .9398 | .979 | 5.66 | -64.70 | -58.33 |
| | 12.77 | 449-5 | 11.64 | 2.395 | .964 | 15.07 | -142.2 | -123.0 |
| | 17.0 | 449-6 | 15.04 | 3.100 | .970 | 17.64 | -174.8 | -159.6 |
| Waspaloy | 0 | 438-3 | 8.070 | 1.692 | 0.990 | 5.57 | -94.17 | -88.45 |
| | 0 | 613-6 | Not sig. | 1.554 | .779 | 52.68 | -155.2 | -155.4 |
| | 4.5 | 438-4 | 9.172 | 1.893 | .990 | 23.42 | -103.8 | -97.61 |
| | 4.5 | 482-4 | 14.03 | 2.696 | .985 | 10.00 | -140.0 | -129.3 |
| | 9.0 | 438-5 | 15.33 | 3.311 | .991 | 10.96 | -186.1 | -177.7 |
| | 9.0 | 482-5 | 1.330 | .2268 | .993 | .47 | -9.99 | -9.38 |
| | 13.5 | 438-6 | 14.14 | 2.919 | .985 | 11.67 | -165.2 | -150.6 |
| | 13.5 | 482-6 | 3.583 | .6077 | .980 | 2.15 | -27.53 | -24.94 |
| | 13.5 | 613-5 | 19.24 | 4.155 | .995 | 10.34 | -226.7 | -223.1 |
| MAR-M-247 | 0.1 | 454-3 | -Force- | 0.1596 | 0.994 | 0.84 | -15.26 | -15.96 |
| | .1 | 482-1 | .1548 | .01463 | .792 | .17 | .16 | .08 |
| | 5 | 454-4 | -Force- | .09237 | .965 | 1.16 | -7.95 | -9.24 |
| | 5 | 482-2 | Not sig. | .02734 | .982 | .24 | -2.65 | -2.73 |
| | 9.76 | 454-5 | .4067 | .2250 | .996 | .81 | -19.46 | -18.44 |
| | 9.76 | 482-3 | 1.146 | .4054 | .995 | 1.42 | -30.86 | -29.07 |
| | 9.76 | 656-1 | .07611 | .01139 | .839 | .10 | -.30 | -.38 |
| IN-738 | 0 | 661-6 | 6.789 | 1.661 | .969 | 11.39 | -116.6 | -98.25 |
| | 4 | 663-1 | 15.20 | 3.288 | .981 | 15.81 | -187.5 | -176.7 |
| | 8 | 663-2 | 13.57 | 3.106 | .988 | 12.19 | -183.3 | -174.9 |
| Nimonic-115 | 0 | 663-3 | 0.9616 | 0.4246 | 0.999 | 0.39 | -32.78 | -32.84 |
| | 5 | 663-4 | -Force- | .04835 | .943 | .79 | -4.13 | -4.84 |
| | 10 | 663-5 | -Force- | .1045 | .943 | 1.70 | -8.93 | -10.45 |
| | 15 | 663-6 | -Force- | .08198 | .962 | 1.08 | -7.24 | -8.20 |
| U-720 | 0 | 654-1 | 12.26 | 3.326 | 0.996 | 8.53 | -206.1 | -210.0 |
| | 7.5 | 654-2 | 9.295 | 3.453 | .998 | 7.99 | -245.9 | -252.3 |
| | 14.7 | 654-3 | 4.811 | 3.676 | .999 | 7.98 | -313.4 | -319.5 |
| SX-R-150 | 0 | 613-1 | Not sig. | 2.998 | 0.999 | 6.55 | -296.5 | -299.9 |
| | 6 | 613-2 | Not sig. | 4.357 | .999 | 7.46 | -427.4 | -435.7 |
| | 12 | 613-3 | Not sig. | 15.74 | .993 | 42.10 | -667.0 ^a | -708.4 |

^aTest discontinued after 45 hr.

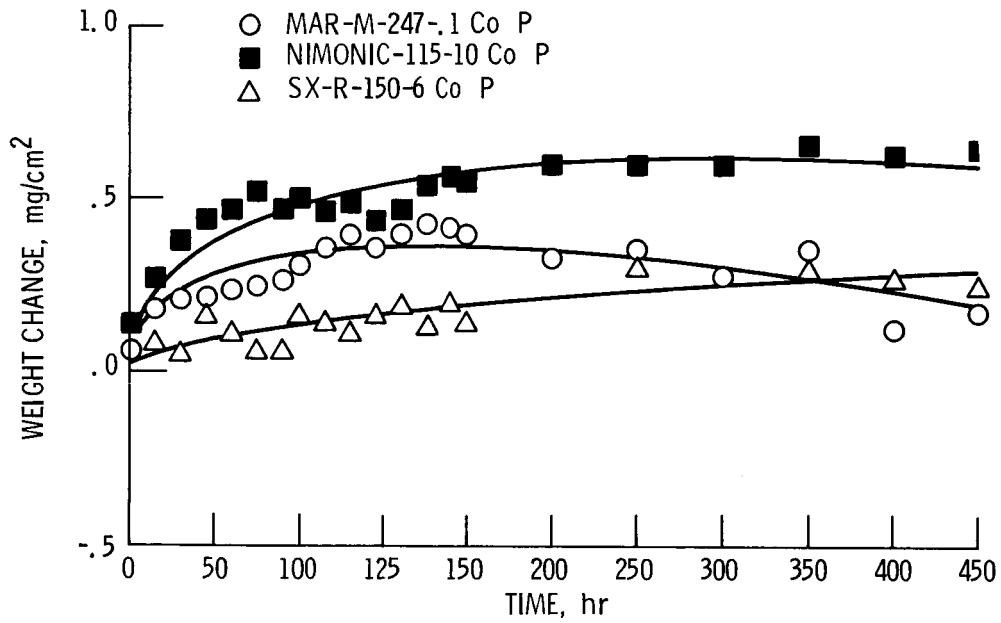


Figure 1. - Regression curve fits and observed cyclic oxidation specific weight change data for selected Ni-base superalloys at 1000 °C (P-paralinear fit).

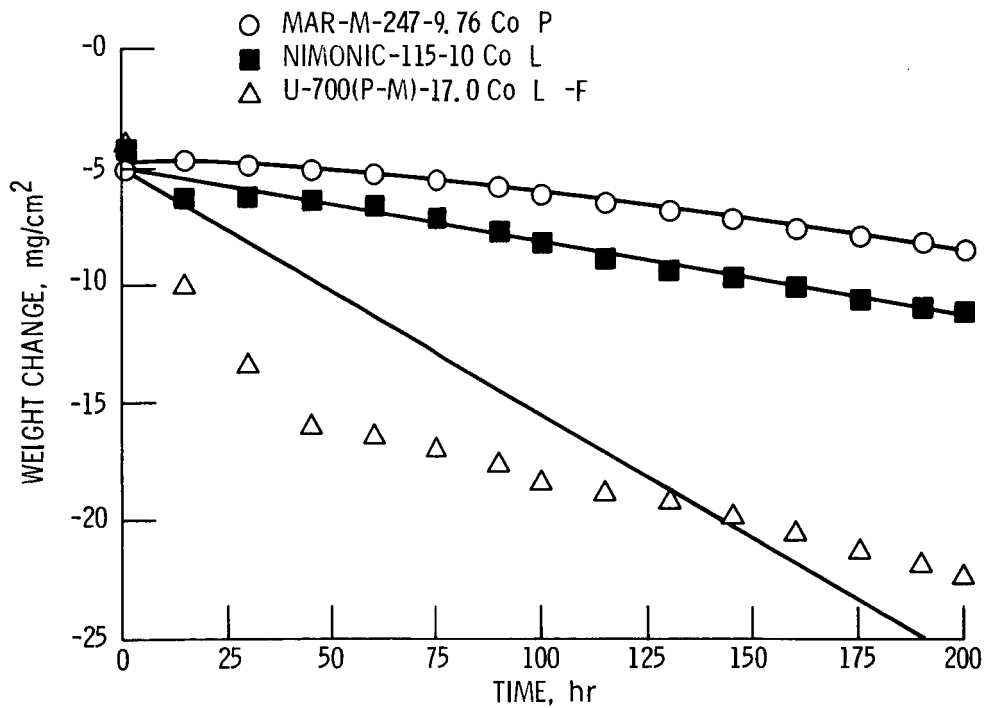


Figure 2. - Regression curve fits and observed cyclic oxidation specific weight change data for selected Ni-base superalloys at 1100 °C (P-paralinear fit; L-linear fit; F-force).

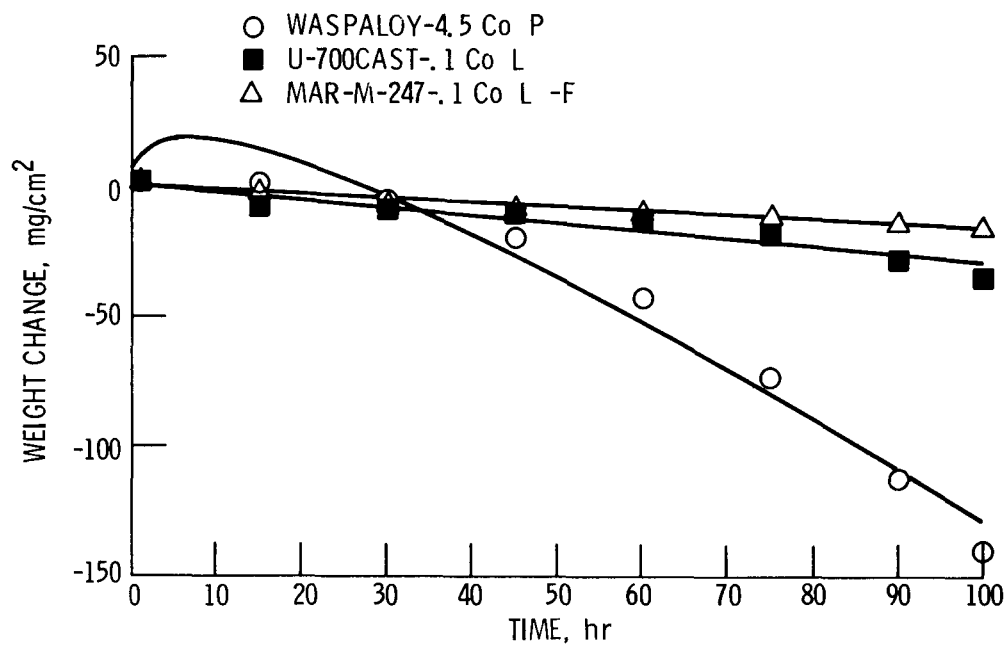


Figure 3. - Regression curve fits and observed cyclic oxidation specific weight change data for selected Ni-base superalloys at 1150 °C (P-paralinear fit; L-linear fit; F-force).

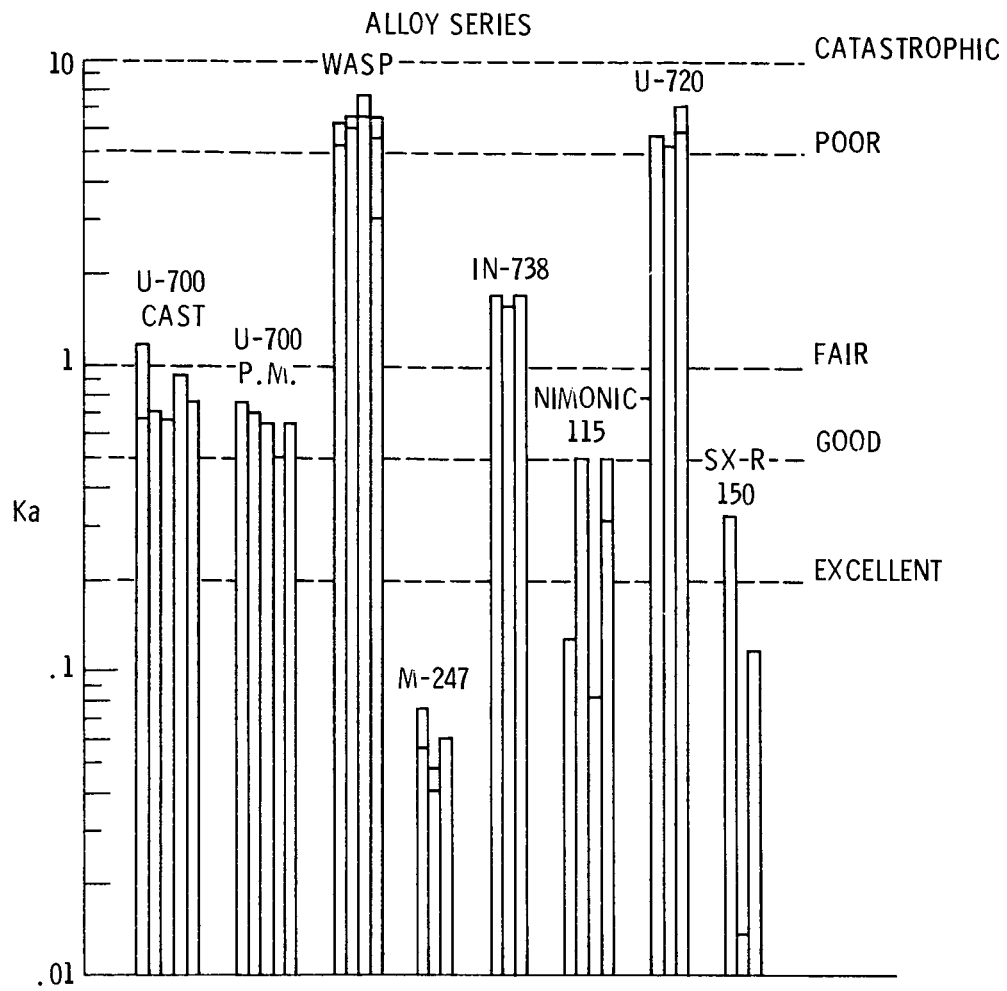


Figure 4. - Derived oxidation parameter K_a at $1000\text{ }^{\circ}\text{C}$ for selected Ni-base superalloys (cobalt level increases - left to right).

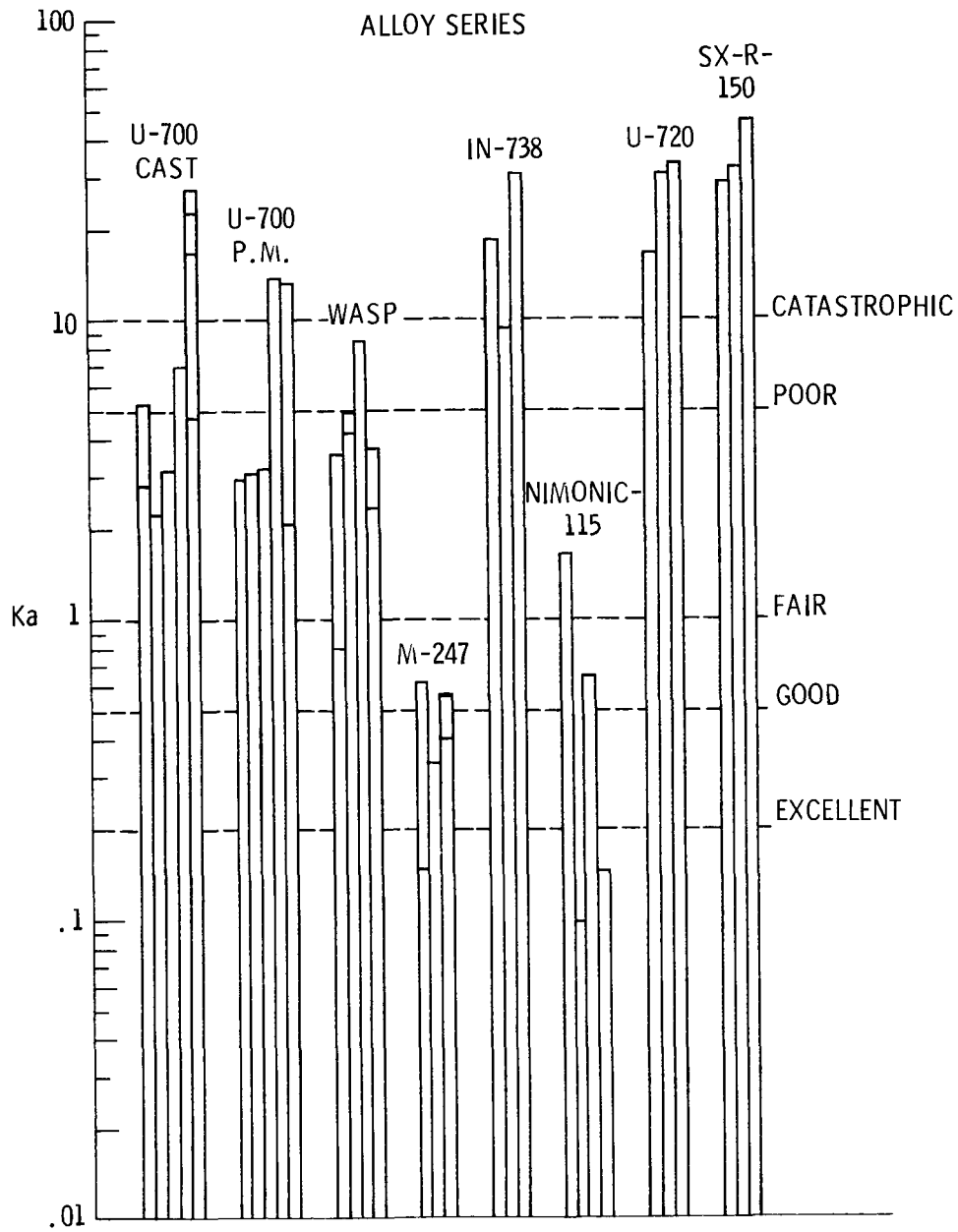


Figure 5. - Derived oxidation parameter K_a at 1100 °C for selected Ni-base superalloys (cobalt level increases - left to right).

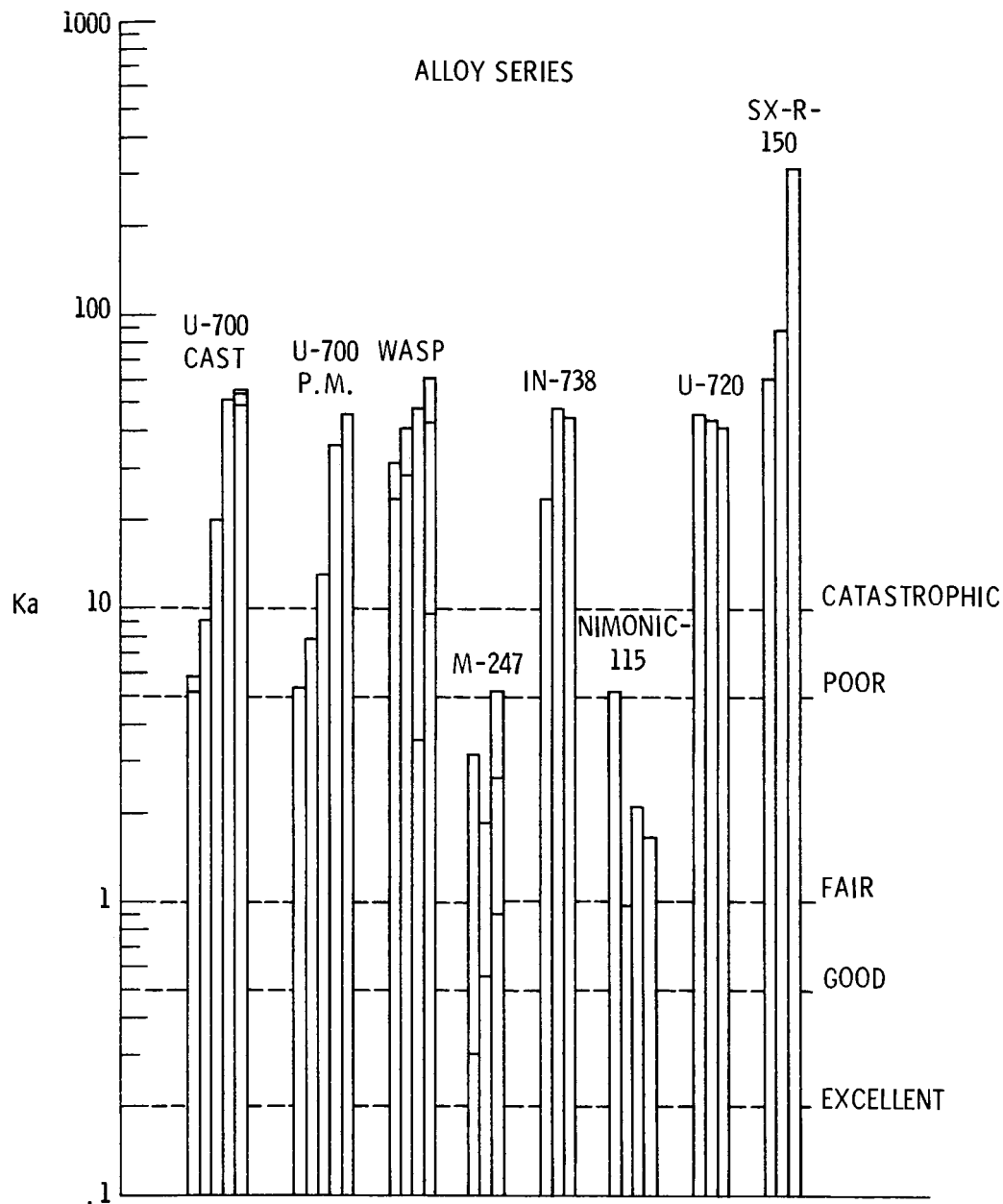
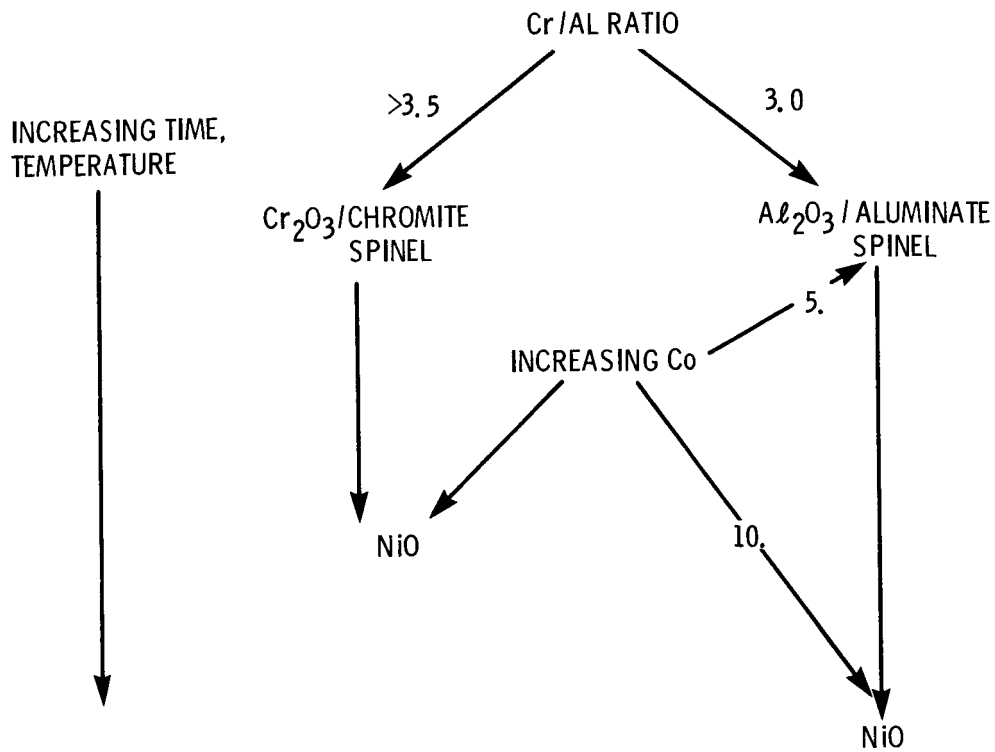


Figure 6. - Derived oxidation parameter K_a at 1150°C for selected Ni-base superalloys (cobalt level increases - left to right).



| | | | | | | |
|--------------|-----------|-------|-------------|-------|----------|-------|
| Cr/Al RATIOS | U-700 | 3.5:1 | IN-738 | 4.7:1 | SX-R-150 | 0.9:1 |
| | WASPALLOY | 15:1 | NIMONIC-115 | 3.0:1 | | |
| | MAR-M-247 | 1.5:1 | U-720 | 7.2:1 | | |

Figure 7. - Alloy scaling tendencies on high strength Ni-base superalloys.

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|--|---|--|------------|
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| 16. Abstract Cobalt levels were systematically varied in the Ni-base turbine alloys U-700 (cast), U-700 (PM/HIP), Waspaloy, Mar-M-247, IN-738, Nimonic-115, U-720, and SX-R-150. The cobalt levels ranged from 0 wt % to the nominal commercial content in each alloy. The alloys were tested in cyclic oxidation in static air at 1000, 1100 and 1150 °C for 500, 200 and 100 hr respectively. An oxidation attack parameter, K_a derived from the specific weight change versus time data was used to evaluate the oxidation behavior of the alloys along with x-ray diffraction analysis of the surface oxides. The alloys tend to form either Cr_2O_3/chromite spinel or Al_2O_3/Aluminate spinel depending on the Cr/Al ratio in the alloys. Alloys with a ratio of 3.5 or higher tend to favor the Cr oxides while those under 3.0 form mostly Al oxides. In general the Al_2O_3/aluminate spinel forming alloys have the better oxidation resistance. Increased cobalt content lowers the scaling resistance of the high Cr alloys while a 5.0 wt % Co content is optimum for the Al controlling alloys. The refractory metals particularly Ta appear beneficial to both types of oxides perhaps due to the formation of the omni-present tri-rutile $Ni(Ta,Cb,W,Mo)_2O_6$. Both scales break down as increasing amounts of NiO is formed. | | | |
| 17. Key Words (Suggested by Author(s)) Cyclic oxidation Superalloys Cobalt effect | | 18. Distribution Statement Unclassified - unlimited STAR Category 26 | |
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