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FORMATION AND BREAKDOWN OF A PROTECTIVE LAYER OF CHROMIUM SESQUIOXIDE ON L-605 ALLOY AT 1100° C

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16. Abstract

The formation and breakdown of a protective layer of Cr_2O_3 on the cobalt-base alloy L-605 during oxidation at 1100° C was evaluated. Deformed surface layers, pressure, and moisture were variables. Increasing surface deformation, decreasing pressure, and decreasing moisture increased time to breakdown. A model was deduced. The breakdown of the Cr_2O_3 layer was caused by a combination of growth stresses and volatilization. Breakdown exposed a chromium-depleted alloy to air, resulting in very rapid oxidation, which continued until consumption of the depletion zone was complete.

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SUMMARY

The formation and breakdown of a protective layer of Cr_2O_3 on L-605 during oxidation at 1100° C was investigated. The effects of surface deformation, pressure, and water vapor on the breakdown time were evaluated. It was found that increasing surface deformation increased the time to breakdown. Decreasing pressure below 1 atmosphere, to 1×15^{-3} torr increased the time until breakdown, as did decreasing water content from 25 000 to 2.5 parts per million.

By metallographic and microprobe examinations of samples during breakdown, a model was deduced. Surface deformation promotes Cr_2O_3 formation, while increasing pressure and moisture increases the volatility of Cr_2O_3. Thus, the Cr_2O_3 grows for a time which is determined by these three factors. At the end of this time the growth stresses cause the oxide to crack, exposing a chromium-depleted metal surface to the oxidizing gas. The resultant rapid oxidation of this surface lifts the remaining Cr_2O_3, exposing more depleted metal. When the depletion zone is consumed and a very thick oxide has formed, the rate of oxidation slows and no further disruption is noted.

INTRODUCTION

The cobalt-base superalloy L-605 is used in air-breathing engines. Its oxidation behavior has been reviewed extensively by Wlodek (ref. 1) and Wolf and Sandrock (ref. 2). Wlodek found that cobalt chromite ($CoCr_2O_4$) was the controlling oxide formed. In the temperature range of 1700° to 2200° F (925° to 1204° C), he found that the oxidation was characterized by nearly parabolic growth of the scale.

Wolf and Sandrock attempted to determine the effect of silicon (Si) content on the oxidation of L-605. They found that some heats of the alloy with low Si or manganese (Mn) contents exhibited excessive oxidation, which was typified by what they termed

"an anomalous weight increment" at 1100° C. By this they meant that these alloys started to oxidize parabolically and then at some time less than 20 hours the rate increased abruptly by more than 100 times. After a period of less than an hour, the rate once again slowed to approximately the initial rate. The authors were able to reproduce the effect only qualitatively. Identical compositions would differ in the time of the increase by a factor of 10. The primary explanation given for this effect was that, during the first stages of oxidation, chromium sesquioxide (Cr_2O_3) formed under stress caused by the mechanism of growth. At some point these stresses were relieved by failure of the metal, exposing unoxidized metal with a surface area very much larger than the original area to an oxidizing atmosphere and resulting in the observed rapid oxidation. They stated that once the new areas were oxidized the rate again dropped. As pointed out by Wolf and Sandrock, this phenomenon appears to be quite general and has been found in other cobalt-base superalloys (e.g., WI-52 (ref. 3)) and in simple binaries (e.g., iron-chromium (Fe-Cr) (ref. 4), nickel-chromium (Ni-Cr) (ref. 5), and cobalt-chromium (ref. 6)). However, these other investigations associated the rapid weight change with a breakdown of the Cr_2O_3 scale, which allowed a chromium-depleted alloy to oxidize rapidly (ref. 7). In all cases, the time of breakdown was erratic, making this alloy difficult to study.

Work on WI-52, also a cobalt-base superalloy with similar chromium and tungsten (W) contents, indicated that the rapid oxidation effect might be somewhat dependent on surface preparation (ref. 3). This work indicated that some initial surface deformation was needed to form a single-phase layer of Cr_2O_3. Reference 8 indicates that this probably derived from the formation of small grains with attendant enhanced Cr diffusion along the grain boundaries. Without the stress, CoO and Cr_2O_3 formed and immediately reacted to form $CoCr_2O_4$; thus, there was no stressed Cr_2O_3 to break down.

The purpose of the current work was to determine which, if either, proposed mechanism for the "anomalous weight increment" is the correct one, in hopes of controlling the effect. To do this it was first necessary to control the variables causing the apparently erratic behavior so that its occurrences could be predicted and the phenomenon studied while in progress.

This work inv/ed the isothermal oxidation of L-605, with the composition found by Wolf and Sandrock to be most prone to the "anomalous weight increment." The oxidation temperature was 1100° C for times to 100 hours. The surface preparation of the samples was varied from a metallographic polish to abrasion with 120-grit paper. The moisture content of the air was controlled, and the pressure was varied from 1×10^{-3} to 760 torr although the bulk of the work was done at near-atmospheric pressure.

In addition to gathering weight change data, the samples were evaluated by X-ray diffraction, metallography, and electron microprobe. A model for the mechanism of this phenomenon is presented.

EXPERIMENTAL PROCEDURE

Material and Preparation

All samples were cut from a sheet of hot-rolled and mill-annealed (1230° C solution treated, rapid air cooled) material about 1 millimeter thick. This sheet is from heat 8 of reference 2, which was found to have a pronounced tendency for the "anomalous weight increment." The composition of this heat is given in table I. Samples were cut from the sheet into 1.9-centimeter by 2.2-centimeter pieces. A variety of surface roughnesses and surface deformations were introduced by grinding and polishing, as shown in table II. Surface roughness was measured with a profilometer, while surface deformations were ranked by standard X-ray diffraction line-broadening techniques. Finally, the samples were ultrasonically cleaned in trichloroethylene, followed by a methanol rinse.

Oxidation

Two oxidation balances were used. While these balances had different operating characteristics, oxidation under similar atmospheres in each gave similar results. They are shown in figures 1(a) and (b). Both balances have automatic recording to a precision of ±0.2 milligram. There are two differences between them: the method of starting the heating cycle and the atmosphere control. The initiation of oxidation for the balance in figure 1(a) is accomplished by raising the furnace; and there is no atmosphere control, the sample being fully exposed when the furnace is lowered. The balance in figure 1(b) requires the sample to be lowered and/or gas admitted at the start of the run. The atmosphere can be closely controlled, and pressures less than 10^{-5} torr can be maintained. The main advantage of the former system is that the sample can be withdrawn within a few minutes of the termination of the run. The main advantage of the latter is the control over the oxidizing atmosphere. Temperature control in both was better than ±5° C.

Runs made in the apparatus in figure 1(a) were started by raising the furnace. Most runs were carried on with no further changes. Some runs were made in which moist air was introduced at either the start of the test or at a predetermined point during the test. Samples were cooled by lowering the furnace. Because of the tendency for spallation of the oxide, some samples were quickly (less than 30 sec) transferred to a mold, where a low-melting-point liquid metal was poured over the sample. This step helped retain the scale for metallographic examination. When samples were not encased in metal, they were allowed to cool with a beaker surrounding them to collect the spall for analysis.

Runs in the apparatus of figure 1(b) were begun with the sample raised from the hot zone and the system evacuated. The desired gas was then allowed to flow into the system. After the pressure and temperature had reached equilibrium, the sample was lowered into the hot zone and the run begun. At the end of the run, the sample was retracted, the system opened, and the sample removed.

Analysis

Where possible, in-situ oxide and spalled oxide were examined by X-ray diffraction. Samples mounted in metal were sectioned and examined metallographically. Selected samples were examined in the electron microprobe with both raster scan and line scan techniques.

RESULTS AND DISCUSSION

Weight Change

A typical plot of isothermal weight change against exposure time is shown in figure 2. It has three distinct portions. First, there is a region of slow, parabolic growth with a low rate constant K_p. This region is abruptly terminated by an oxidation rate increase of over 100 times. This rate then gradually decreases to a rate close to the initial rate, 0.20 to 0.30 mg^2 cm^{-4} hr^{-1}. Phalnikar, Evans, and Baldwin (ref. 9) have shown K_p values of approximately 0.20 mg^2 cm^{-4} hr^{-1} in the cobalt-chromium system at 1100° C for compositions containing about 22-weight-percent chromium and K_p values of 50 mg^2 cm^{-4} hr^{-1} at about 17-weight-percent chromium.

Although the moisture content of the oxidizing medium was not controlled, it is felt that the data of reference 9 are reasonably valid in showing the kind of K_p ranges in this system. The major point is that for relatively small compositional changes (5 to 10 percent), the value of K_p can vary over several orders of magnitude.

A characteristic, easy-to-measure parameter of curves such as figure 2 is the point at which the sudden increase begins. This time is, in effect, the life of the protective Cr_2O_3 layer. It is used in this report to show the effects of water vapor, surface deformation, and pressure. Figure 3 is a plot of the effect of both water vapor and prior surface deformation (as indicated by X-ray line broadening) on the lifetime of the Cr_2O_3 layer. The curve for tests run at 2.5 parts per million (dewpoint = -70° C) H_2O indicates a very strong function of Cr_2O_3 layer life on surface deformation (the lifetimes vary from less than 1 hr to about 15 hrs). This indicates that increasing surface de-

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formation promotes the tendency for Cr_2O_3 formation, perhaps by increased diffusion of chromium along the increased grain boundaries of the fine grains formed during abrasion. Note that if the data were plotted against surface roughness (see table II), the ground sample would fall well off the curve. Thus, deformation, not roughness, is the important variable.

As the moisture content was raised to 8000 parts per million (dewpoint = 0° C), the lifetimes were cut by 1/2 to 1/3. A further increase to 25 000 parts per million (dewpoint = 25° C) reduced the times to less than 1 hour. This phenomenon is associated with the loss of Cr_2O_3 as vapor by a reaction with H_2O (see ref. 10). It presents a problem in data interpretation to the oxidation investigator who does not control the moisture content of the atmosphere during oxidation of Cr_2O_3-scale-forming alloys. However, it also offered a tool for use in the present investigation (see the following section, Metallography).

Kohl and Stearns (ref. 10) have also shown the strong effect of oxygen pressure on the loss of CrO_3 vapor from Cr_2O_3. In this case the loss increases with O_2 pressure as CrO_3 formation increases. Thus, one would expect the Cr_2O_3 layer to last longer at low pressures. This is indeed the case, as shown in figure 4.

Diffraction, metallography, and microprobe X-ray diffraction confirmed the data of Wolf and Sandrock (ref. 2). Cr_2O_3 was the only oxide before the rapid increase in rate, while CoO, $CoCr_2O_4$, $CoWO_4$, and Cr_2O_3 were present afterward.

Metallography

As indicated in the preceding section, Weight Change, the water vapor effect was used to investigate the breakdown of the Cr_2O_3 layer. Samples with sufficient prior surface deformation to achieve long lives were initially oxidized in dry air. Then, at the desired moment, air saturated with water at 25° C was introduced. Within a few minutes, breakdown of the Cr_2O_3 layer began. This allowed samples to be removed and mounted just before, during, or after the breakdown.

The results of this work are illustrated in figures 5 to 8. Figure 5(a) is the microstructure prior to breakdown. It shows the poorly adherent, blistered Cr_2O_3 seen by Wolf and Sandrock on L-605 (ref. 2) and by Caplan, Harvey, and Cohen on chromium (ref. 11). The microprobe trace of this sample (fig. 6) clearly shows the Cr_2O_3 layer and also indicates extreme chromium depletion in the metal beneath, with corresponding enrichment of cobalt and tungsten. Note that this depletion-zone composition appears low enough in chromium to produce a very high K_p (up to 600 mg^2 cm^{-4} hr^{-1} from ref. 11) upon oxidation.

During the first stages of breakdown (fig. 5(b)), CoO is seen pushing up the Cr_2O_3. Note the thinner portions of Cr_2O_3 at the top of the blister. At the metal-oxide interface, rapid growth of CoO, $CoCr_2O_4$, and $CoWO_4$ occurs. The microprobe results help confirm this view (fig. 7).

Figures 5(c) and 8 show the sample in the third stage of Cr_2O_3 scale breakdown. CoO has grown completely around the Cr_2O_3, reacted with it, and formed $CoCr_2O_4$. As noted by Wolf and Sandrock (ref. 2) the shape of the original Cr_2O_3 is retained by the $CoCr_2O_4$. Also, as shown in figure 5(b) the nodes of the blisters are very thin. Figure 8 shows a very shallow chromium depletion zone beneath the scale.

Oxidation Model

Figure 9 represents a proposed model for the formation and breakdown of Cr_2O_3 on L-605. It is believed that the model has some general validity for most systems where this phenomenon has been observed.

The first step (fig. 9(a)) is the initial growth of Cr_2O_3 with its corresponding compressive stresses. As the stresses build, the oxide blisters begin to form and pull the oxide away from the metal in some spots (fig. 9(b)). The oxide grows by diffusion and diminishes by the reaction of Cr_2O_3 with O_2 to form CrO_3 vapor or with H_2O to form $CrO_2(OH)$. At points where the oxide is not in contact with the metal it is thinner (fig. 9(c)) because the chromium would have to be vapor transported across the void, which must be a slower step. Next, the oxide cracks at these thin points either because the stress exceeds the load capabilities of the thin cross section or, less likely, because the vapor loss proceeds to open a hole. In either case, oxide cracking exposes chromium-depleted alloy to the oxidizing environment. CoO forms rapidly beneath the Cr_2O_3, lifting the loosely adhering oxide and allowing more depleted metal to be exposed (figs. 9(d) and (e)). Finally, the CoO envelopes the Cr_2O_3 layer, and with the thickening of the total layer and the consumption of the depleted metal the reaction slows down. The magnitude of the abrupt increase is somewhat proportional to the extent of the depleted zone at breakdown and is therefore related to the life of the initial Cr_2O_3 scale layer.

In cases where Cr_2O_3 is not allowed to form because of either high water content at the start of oxidation or insufficient stress, CoO forms from the start. This eliminates the breakdown phenomenon but at the expense of a much higher initial oxidation rate.

SUMMARY OF RESULTS

This work deals with the formation and breakdown of a protective layer of Cr_2O_3 on

L-605 at 1100° C. The material was oxidized for times up to 100 hours in air at pressures from 10^{-3} to 760 torr with water vapor contents from 2.5 to 25 000 parts per million. The Cr_2O_3 layer formation and breakdown sequence was evaluated by weight change, X-ray diffraction, metallography, and electron microprobe. The major findings were as follows:

1. A protective layer consisting only of Cr_2O_3 formed on surfaces deformed prior to oxidation. On those surfaces without residual stress (as indicated by X-ray line broadening) a complex layer of CoO, $CoCr_2O_4$, and Cr_2O_3 formed immediately.

2. Samples having greater initial surface deformation retained the exclusive protective layer of Cr_2O_3 for longer times.

3. Water vapor in the air reduced the lifetime of the Cr_2O_3 layer drastically, that is, from a maximum 15 hours to less than 1/2 hour, when sufficient surface deformation was present to allow Cr_2O_3 to form initially.

4. As the pressure of the low-water-content air is reduced, the rate of formation of Cr_2O_3 is reduced; but its lifetime as a single layer is extended.

5. The basic sequence of the formation and breakdown of the Cr_2O_3 layer on a deformed surface is as follows:

    a. Cr_2O_3 is formed preferentially on a deformed surface. The oxide grows in compression.

    b. The Cr_2O_3 deforms by wrinkling, causing much of the contact between oxide and metal to be lost.

    c. The parts of the oxide not in contact with the metal grow thinner because of the loss of oxide through the formation of gaseous CrO_3.

    d. The Cr_2O_3 cracks in the thinned area, exposing a chromium-depleted alloy.

    e. This alloy oxidizes rapidly mostly to CoO, pushing the rest of the Cr_2O_3 away from the metal and exposing more fresh surface.

    f. The chromium-depleted surface of the alloy is oxidized; the rate slows as this zone is consumed. As a thick mixed scale forms, the diffusion path is lengthened and the rate of oxidation slows.

CONCLUDING REMARKS

The object of this investigation was to determine the mechanism of failure of the protective Cr_2O_3 layer formed on L-605 and to discover some of the factors affecting this breakdown. The failure of the chromium scale was caused by the lifting of the scale in the shape of blisters from the metal by the compressive growth stresses, followed by vaporization of the oxide. When the scale cracks at the blister tops, rapid oxidation of the chromium-depleted alloy follows the failure of Cr_2O_3 and is noted as

a large increase in the oxidation rate. Therefore, anything that promotes Cr_2O_3 formation (i.e., silicon or prior surface deformation) will prolong the life of the protective layer. Conversely, anything that increases volatilization (i.e., water vapor) decreases the life of this layer.

There appear to be several ways of avoiding the Cr_2O_3 layer breakdown. The first is to use a higher silicon concentration to further stabilize the Cr_2O_3 layer. This, however, has the disadvantage of seriously decreasing the post-aging ductility of the alloy. Another possibility would be the use of alloying additions to act as dopants of the Cr_2O_3 scale to reduce its volatility. Changing volatility without altering the single-phase Cr_2O_3 nature of the scale or drastically increasing the rate of oxidation appears a remote possibility. An extension of this approach would be the addition of Mn to change the scale to $MnCr_2O_4$, which might be a desirable adherent scale as has been shown in the simple Ni-Cr and Co-Cr binary systems. One final alloying approach might be to increase the chromium content so as to mitigate depletion-zone formation. A different approach would be to polish the surface prior to oxidation. This would eliminate the Cr_2O_3 formation entirely, but the resultant scale would have a higher rate constant K_p and would spall readily upon cooling.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, February 15, 1973,
501-01.

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TABLE I. - ANALYSIS OF L-605

HEAT 8 (REF. 2)

| Element	Content, wt.%
Silicon	0.12
Manganese	1.10
Iron	3.06
Chromium	20.06
Tungsten	14.50
Carbon	.12
Nickel	10.27
Cobalt	Balance
Sulphur	.005

TABLE II. - SURFACE CONDITIONS OF L-605

PRIOR TO OXIDATION

Surface treatment	Roughness (rms), μm	X-ray line breadth (degrees 2θ), corrected
0.5-μm diamond	5×10^{-3} to 15×10^{-3}	0
600-Grit SiC	.02 to .05	.292
400-Grit SiC	.05 to .10	.335
320-Grit SiC	.08 to .12	.410
240-Grit SiC	.12 to .17	.535
120-Grit SiC	.25 to .33	.707
Ground	.02 to .15	.626
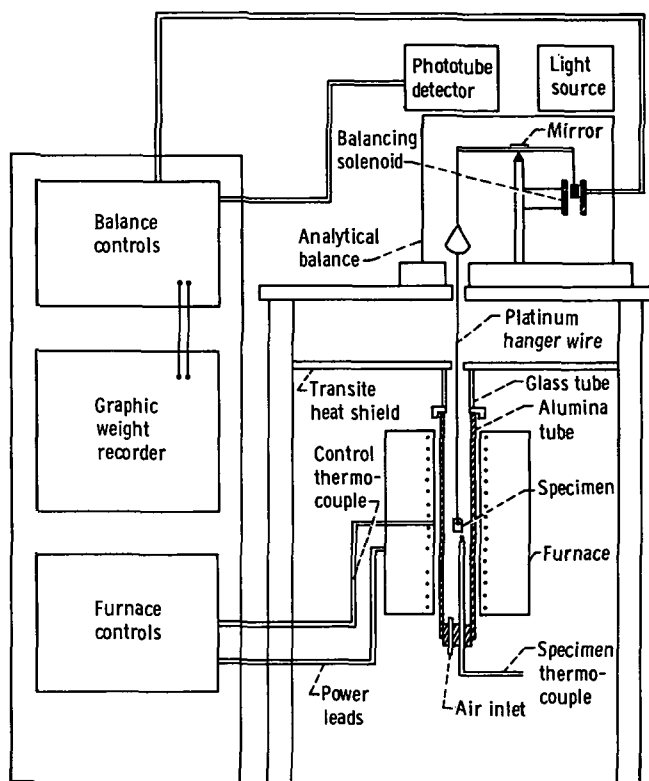
(a) Open to ambient pressure.
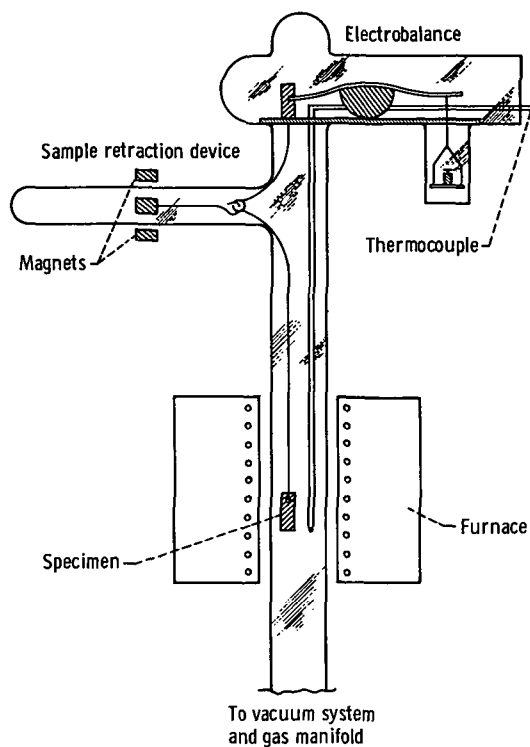
(b) With controlled atmosphere.

Figure 1. – Continuous weight gain apparatus (ref. 2).
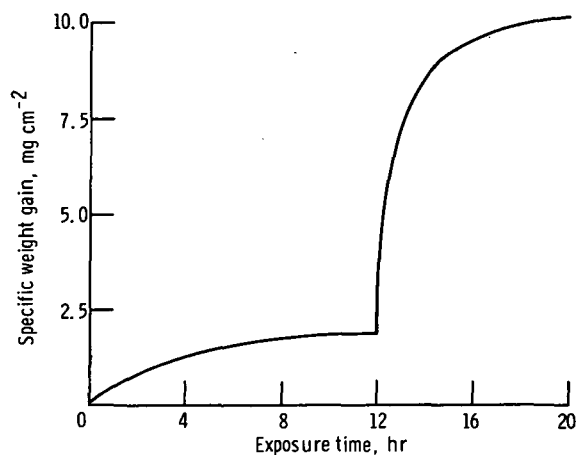
Figure 2. – Typical oxidation behavior of L-605 with low silicon content in air at 1100° C. Isothermal, 700-torr air containing 2.5 ppm H_2O. Sample abraded with 120-grit SiC prior to oxidation.
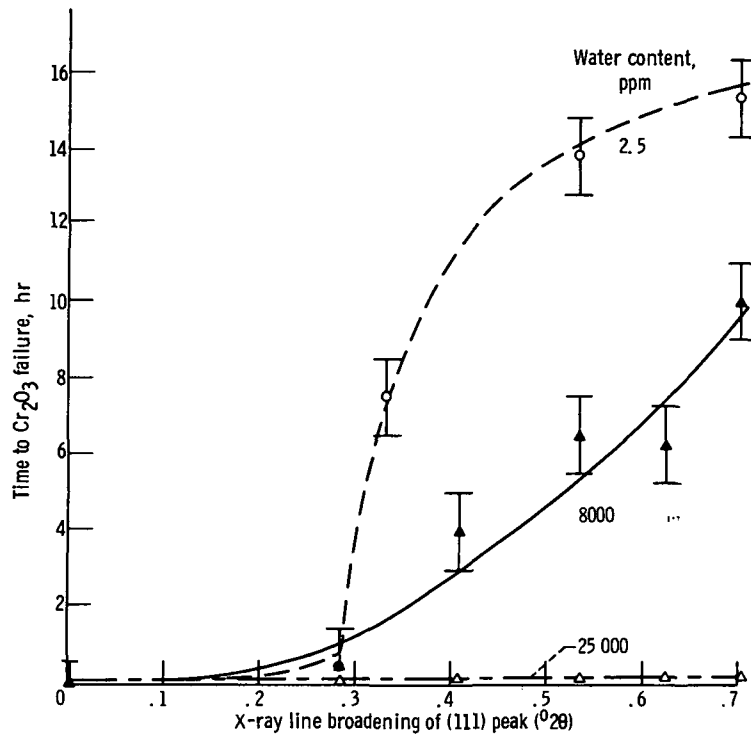
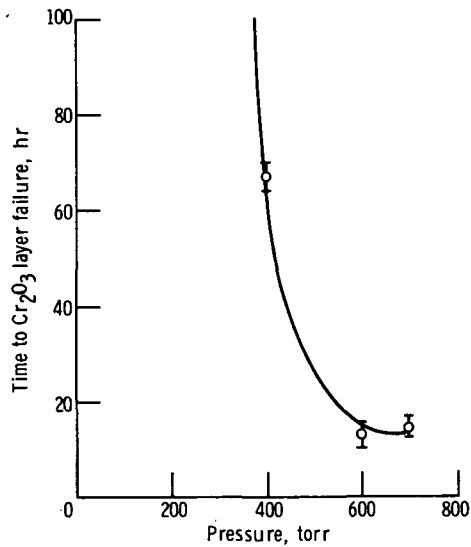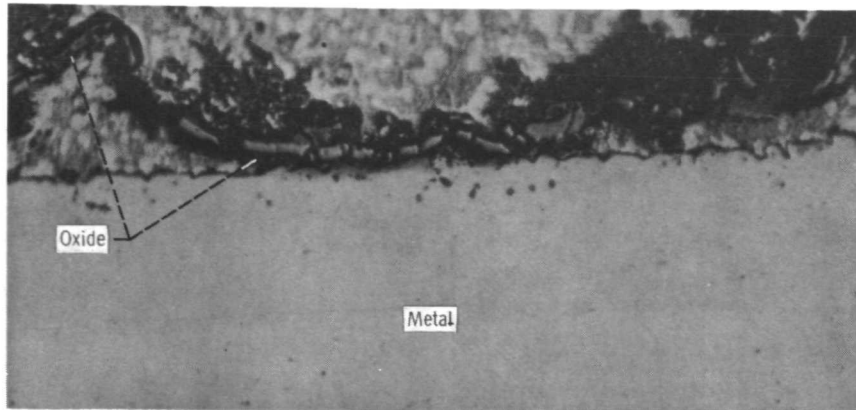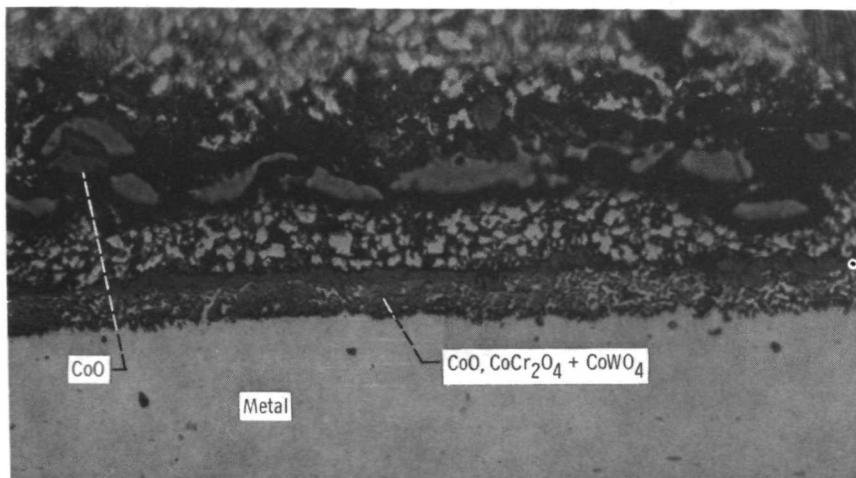Figure 3. – Effect of water vapor and prior surface deformation as indicated by line broadening on the loss of Cr_2O_3 protection on L-605 at 1100° C in still air. Ambient pressure.

Figure 4. – Effect of pressure on breakdown of protective layer of Cr_2O_3 formed on L-605 in air containing 2.5 ppm H_2O at 1100° C.

(a) Before breakdown and after 4 hours in air containing 8000 ppm H_2O.

(b) During breakdown and after 6 hours in air containing 8000 ppm H_2O followed by 10 minutes at 25 000 ppm H_2O.
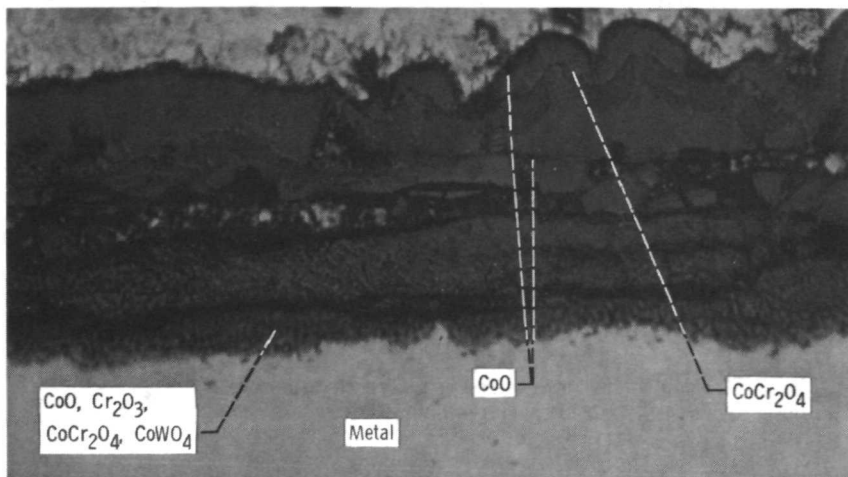
(c) After breakdown caused by 6 hours in 8000 ppm H_2O followed by 1 hour at 25 000 ppm H_2O.

Figure 5. - Microstructure of scales on L-605 oxidized at 1100^0 C in air before, during, and after breakdown of Cr_2O_3 layer by saturating the air with H_2O vapor for 6 hours after start of test. Unetched, X250.
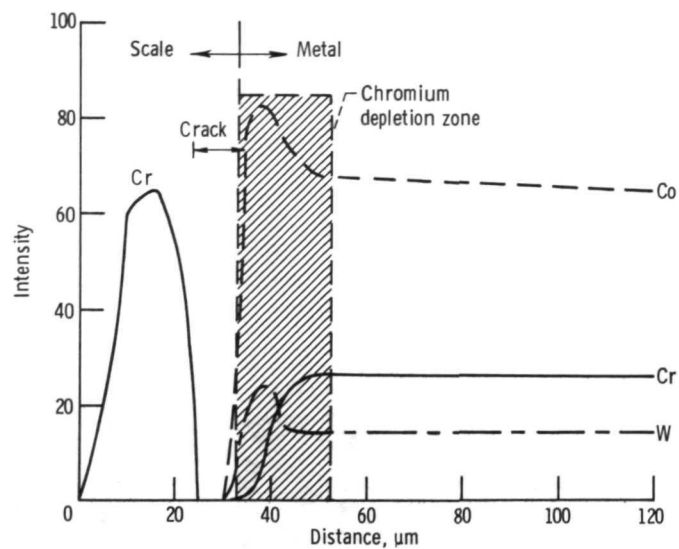
Figure 6. - Microprobe traces on L-605 before breakdown of Cr_2O_3 scale and after 4 hours at 1100^0 C in air containing 8000 ppm H_2O. Note depletion of Cr in metal at scale-metal interface.
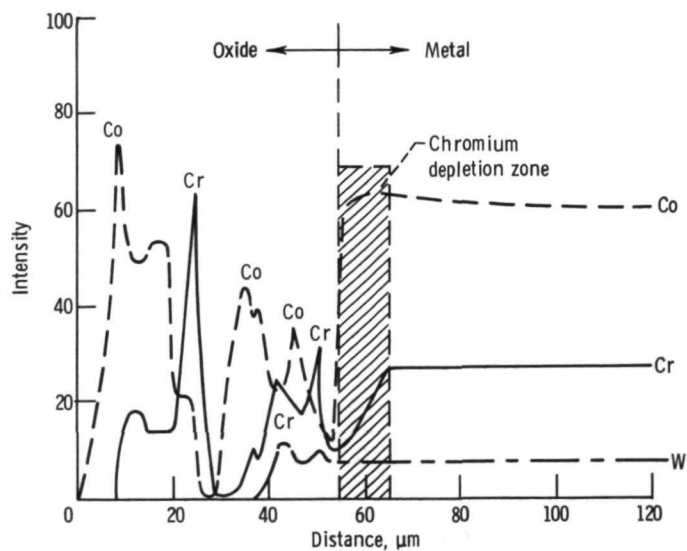

Figure 7. - Microprobe traces on L-605 during breakdown of Cr_2O_3 scale and after 6 hours in air containing 8000 ppm H_2O followed by 10 minutes at 25 000 ppm H_2O.
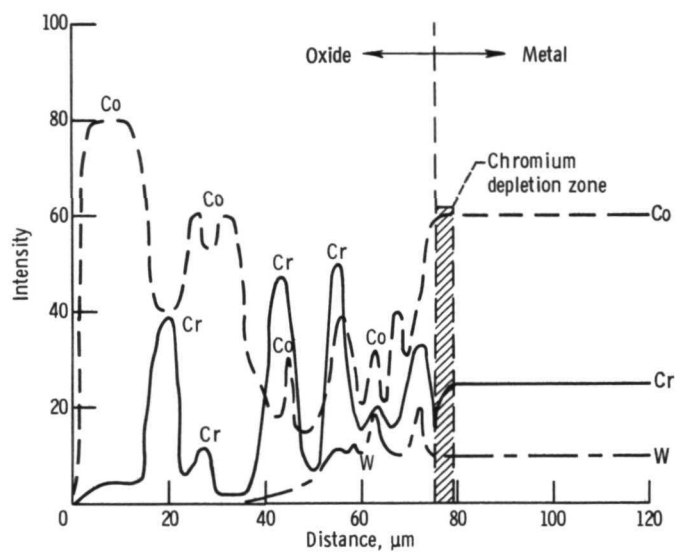
Figure 8. - Microprobe traces on L-605 after breakdown of Cr_2O_3 scale caused by 6 hours at 1100^0 C in air containing 8000 ppm H_2O followed by 1 hour at 25 000 ppm H_2O.
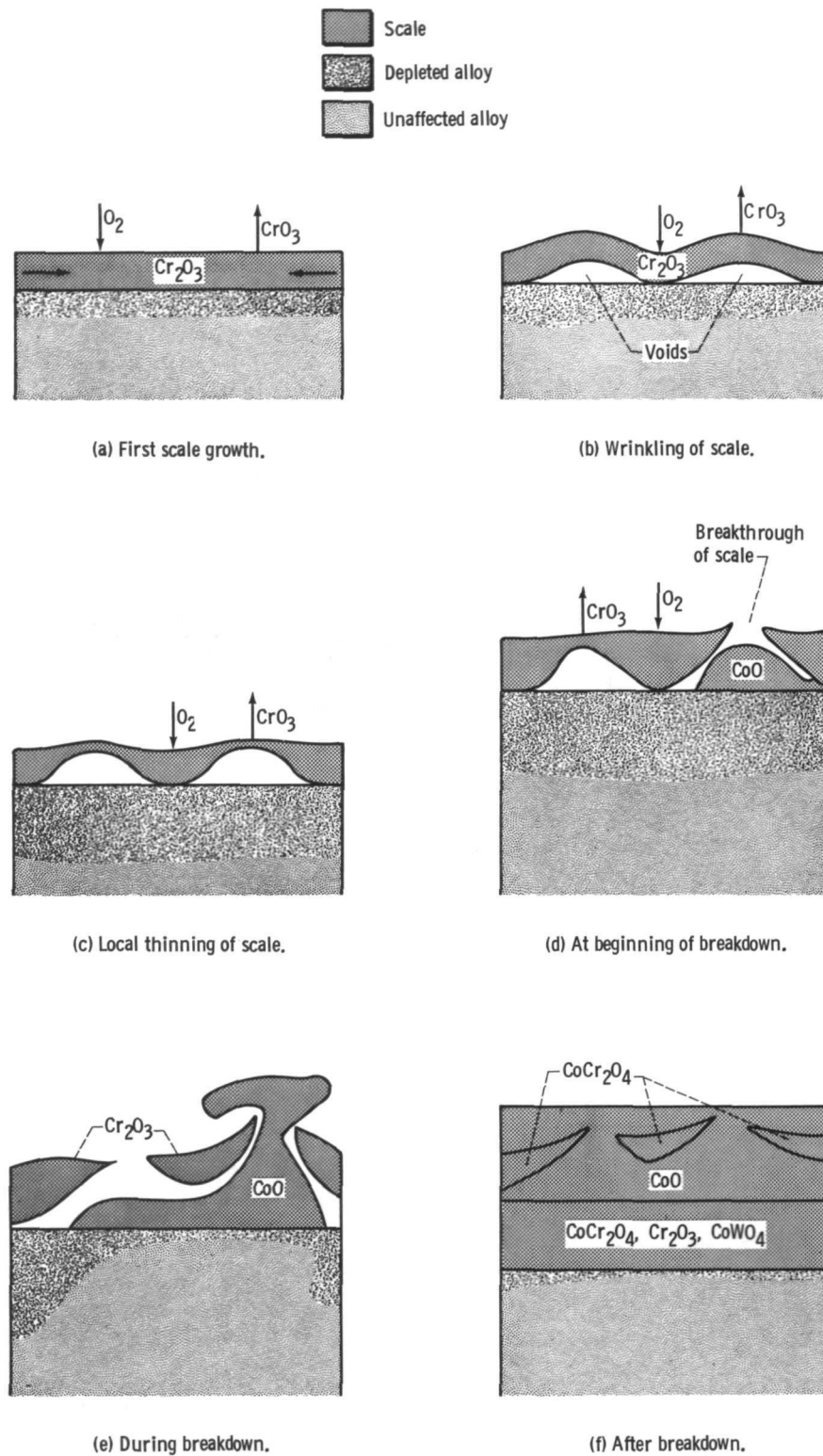
Figure 9. – Proposed model for formation and breakdown of protective oxide layer on L-605 during oxidation at 1100° C.

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—NATIONAL AERONAUTICS AND SPACE ACT OF 1958

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