

BNL-52620  
Formal Report

**OXIDATION OF INCONEL 718 IN AIR  
AT TEMPERATURES FROM 973 K TO 1620 K**

by

**G.A. Greene and C.C. Finfrock  
Brookhaven National Laboratory  
Upton, New York 11973**

**OCTOBER 2000**

**ENERGY SCIENCES AND TECHNOLOGY DEPARTMENT**

**Brookhaven National Laboratory  
Brookhaven Science Associates  
Upton, Long Island, New York 11973**

**Under Contract No. DE-AC02-98CH10886 with the**

**UNITED STATES DEPARTMENT OF ENERGY**

BNL-52620  
UC- 704

**OXIDATION OF INCONEL 718 IN AIR  
AT TEMPERATURES FROM 973 K TO 1620 K**

by

G.A. Greene and C.C. Finfrock  
Brookhaven National Laboratory  
Upton, New York 11973

October 2000

Energy Sciences and Technology Department

Brookhaven National Laboratory  
Brookhaven Science Associates  
Upton, Long Island, New York 11973

Under Contract No. DE-AC02-98CH10886 with the

UNITED STATES DEPARTMENT OF ENERGY

## DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency, contractor, or subcontractor thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency, contractor or subcontractor thereof.

Printed in the United States of America  
Available from  
National Technical Information Service  
U.S. Department of Commerce  
5285 Port Royal Road  
Springfield, VA 22161

## ABSTRACT

As part of the APT project, it was necessary to quantify the release of tungsten from the APT spallation target during postulated accident conditions in order to develop accident source terms for accident consequence characterization. Experiments with tungsten rods at high temperatures in a flowing steam environment characteristic of postulated accidents revealed that considerable vaporization of the tungsten occurred as a result of reactions with the steam and that the aerosols which formed were readily transported away from the tungsten surfaces, thus exposing fresh tungsten to react with more steam. The resulting tungsten release fractions and source terms were undesirable and it was decided to clad the tungsten target with Inconel 718 in order to protect it from contact with steam during an accident and mitigate the accident source term and the consequences.

As part of the material selection criteria, experiments were conducted with Inconel 718 at high temperatures to evaluate the rate of oxidation of the proposed clad material over as wide a temperature range as possible, as well as to determine the high-temperature failure limit of the material. Samples of Inconel 718 were inserted into a preheated furnace at temperatures ranging from 973 K to 1620 K and oxidized in air for varying periods of time. After oxidizing in air at a constant temperature for the prescribed time and then being allowed to cool, the samples would be reweighed to determine their weight gain due to the uptake of oxygen. From these weight gain measurements, it was possible to identify three regimes of oxidation for Inconel 718: a low-temperature regime in which the samples became passivated after the initial oxidation, an intermediate-temperature regime in which the rate of oxidation was limited by diffusion and exhibited a constant parabolic rate dependence, and a high-temperature regime in which material deformation and damage accompanied an accelerated oxidation rate above the parabolic regime.

At temperatures below 1173 K, the rate of oxidation of the Inconel 718 surface was found to decrease markedly with time; the parabolic oxidation rate coefficient was not a constant but decreased with time. This was taken to indicate that the oxide film on the surface was having a passivating effect on oxygen transport through the oxide to the underlying metal. For temperatures in the range 1173 K to 1573 K, the time-dependent rate of oxidation as determined once again by weight-gain measurements was found to display the classical parabolic rate behavior, indicating that the rate of transport of reactants through the oxide was controlled by diffusion through the growing oxide layer. Parabolic rate coefficients were determined by least-squares analysis of time-dependent mass-gain data at 1173 K, 1273 K, 1373 K, 1473 K and 1573 K.

At temperatures above 1540 K, post test examination of the oxidized samples revealed that the Inconel 718 began to lose strength and to deform. At 1540 K, samples which were suspended from their ends during testing began to demonstrate axial curvature as they lost strength and bowed under their own weight. As the temperatures of the tests were increased, rivulets were seen to appear on the surfaces of the test specimens; damage became severe at 1560 K. Although melting was never observed in any of these tests even up to 1620 K, it was concluded from these data that the Inconel 718 clad should not be expected to protect the underlying tungsten at temperatures above 1540 K.

## TABLE OF CONTENTS

	<u>Page</u>
Abstract .....	iii
List of Figures .....	v
List of Tables .....	vi
Introduction .....	1
Experimental Apparatus and Procedures .....	2
Discussion of Results .....	3
Parabolic Oxidation Rate (24-hour tests) Series, 1173 K-1573 K .....	4
Correlations of Parabolic Oxidation Rate Data .....	5
Ultra-High Temperature (single-point) Data, 1474 K-1614 K .....	6
Low-Temperature, Long-Term (9-14 days) Oxidation Rate Series, 973 K-1223 K .....	8
Conclusions .....	9
Authors' End Note .....	10
References .....	11

## LIST OF FIGURES

	<u>Page</u>
Figure 1	Square of the mass gain per unit area vs. time for Inconel 718 oxidized in air between 1173 K and 1573 K for up to 36 hours at atmospheric pressure. . . . . 13
Figure 2	Correlations of the parabolic oxidation rate coefficients $K_p$ vs. $1/T$ for Inconel 718 oxidation in air at temperatures between 1173 K and 1573 K. . . . . 14
Figure 3	High temperature parabolic oxidation rate coefficients $K_p$ vs. $1/T$ for Inconel 718 oxidation in air at temperatures between 1473 K and 1620 K. . . . . 15
Figure 4	Square of the mass gain per unit area vs. time for Inconel 718 oxidized in air between 973 K and 1223 K for nine to 15 days at atmospheric pressure (linear fit to the 1223 K data is for illustration purposes only). . . . . 16

## LIST OF TABLES

	<u>Page</u>
Table 1	Constituents of Inconel 718 ..... 17
Table 2	Data Sets for Parabolic Oxidation Rate Correlations for Inconel 718 in Air ... 18
Table 3	Parabolic Oxidation Rate Coefficients $K_p$ for Oxidation of Inconel 718 in Air Used to Construct the Oxidation Correlations in Figure 2 ..... 19
Table 4	Ultra-High Temperature 24-Hour Oxidation Rate Data ..... 20

## INTRODUCTION

The vaporization of tungsten from the APT spallation target has been shown to be the dominant contributor to the accident source term for unmitigated target overheating accidents [1]. Reactions with steam which persist for tungsten temperatures as low as 800°C result in the formation of a hydrated tungsten-oxide which has a high vapor pressure and is readily convected in a flowing atmosphere. This low-temperature vaporization reaction essentially removes the oxide film that forms on the tungsten surface as soon as it forms, leaving behind a fresh surface for continued oxidation and vaporization. Since a protective oxide scale is not formed, a continual process of oxidation and vaporization occurs until either the tungsten temperature decreases below the vaporization threshold and a protective oxide scale forms, the gas phase is starved of steam in which case only oxidation will occur, or the tungsten metal is consumed. In an air environment, tungsten vaporization does not occur.

Inconel 718 alloy has been proposed as a clad for the water-cooled APT tungsten target for several reasons, not the least of which is to prevent the contact of tungsten target rods with steam during postulated accident conditions, thus depriving the tungsten of steam and preventing the chemical reactions which result in tungsten-oxide vaporization [2]. Inconel 718 was chosen from among other superalloy candidate materials because of its superior performance in irradiated water-cooled environments and its resistance to oxidation at elevated temperatures in air and steam. Detailed physical and mechanical properties of Inconel 718 have been provided by The International Nickel Company in Reference 3.

For the APT target overheating accidents in which tungsten vaporization is an issue, the sequence of events which occur is a loss of flow followed by a loss of coolant with the shutdown of the proton beam. Dry-out of the target rungs would result in the loss of all axial heat transfer to the engineered heat sink; the only heat transfer mechanism which would remain to remove decay heat from the irradiated tungsten cylinders would be thermal radiation from the inner most of the nested cylinders radially outward to the outer housing of the target rung, and then by radiative heat transfer to the surrounding blanket modules which may themselves still be cooled by their independent cooling loop. The tungsten cylinders are nested within each other in a configuration which is not favorable for thermal radiation as a cooling mechanism. An illustrative analogy would be a series of nested thermal radiation shields around a heated cylinder, the purpose of which would be to present a resistance to heat transfer from the inner heat source to the colder surroundings. Such a geometry would be self-insulated, and the inner cylinder could easily achieve extremely high temperatures with little heat input. The APT target assemblies which consist of just such nested cylinders inside a structural housing have been calculated to achieve very high temperatures without forced cooling, even without beam energy deposition in the target, approaching or even exceeding 1200°C shortly following beam shutdown and loss of coolant. Such a transient would be short and these elevated temperatures would decrease rapidly within tens of minutes following the start of the transient.

The material which is chosen as a clad for the APT tungsten spallation target must have other attributes in addition to its tolerance for the severe accident conditions just outlined. These include

weldability and machineability, resistance to erosion by flowing water and structural strength to resist mechanical failure. Inconel 718 satisfies these criteria well. The severe accident attributes which the clad must possess are as follows: oxidation resistance at high temperatures during an overheating transient such that the penetration of the oxide into the clad does not compromise its function, a sufficiently high failure temperature limit which will not be exceeded by maximum temperatures in the target during the transient, and long-term oxidation resistance at lower temperatures which may persist for many hours or even days in the target following the peak of the transient into the tail of the cool down phase. The experiments to be discussed in this report are designed to investigate the viability of Inconel 718 under these conditions.

## **EXPERIMENTAL APPARATUS AND PROCEDURES**

The experimental apparatus and procedures for this investigation were similar to those for other investigations reported in the literature [4-8] and will be described in the following discussion. Test samples were prepared from a stock of mill-certified Inconel 718 rod material of 3 mm diameter (see Table 1 for the alloy composition). The rod was cut into pieces nominally 1.5 inches long. The ends were deburred and the pieces were cleaned in a detergent solution. The samples were then rinsed well with distilled water and then rinsed with ethanol. After drying, the samples were stored in polyethylene zip-lock bags. As samples were taken for use, their lengths and diameters were measured. The samples' lengths were measured to a precision of 0.001 inch with a recently calibrated dial indicating caliper. The diameters of the samples were measured in three places along the length to a precision of 0.0001 inch with a recently calibrated micrometer. The three diameter measurements were averaged to provide the sample diameter used in the surface area calculation. Sample mass determinations were made using a Sartorius analytical balance capable of weighing to a precision of 0.1 mg. The balance calibration was frequently checked using both its internal 100 g standard mass and an external 50 g standard mass. Prior to weighing, all samples were held overnight in glass desiccators to eliminate any effect of humidity on the sample mass determination. The dimensional and mass specifications for each sample were recorded on the preprinted run sheet for the sample, and this run sheet served as a traveler for the sample throughout its testing. The run sheets were eventually reviewed, signed and archived after a quality assurance check to verify the validity of the experimental data.

Sample oxidations were conducted in fused quartz or high-purity alumina crucibles as temperature requirements dictated. Prior to use, these crucibles were heated to their service temperature to drive out any volatile materials that would show up as mass loss on weighing. Two crucibles were repeatedly heated, cooled in a desiccator and then weighed until no mass change was observed. The cumulative duration to the state of no mass change was then used as the required heating time for all other crucibles used in this study. Two standard laboratory muffle furnaces were used to oxidize the samples. A furnace with electric resistance elements similar to Nichrome was used up to 1273 K and a furnace with silicon carbide elements was used for tests above 1273 K. Temperature measurements of the furnace chambers were made with a Leeds and Northrop Pt/Pt-10%Rh thermocouple mounted in a high-purity alumina protection tube. The Pt/Pt-10%Rh thermocouple

was inserted into the furnace chamber through an access hole in the rear or top of the furnace and positioned so that the active element was in the proximity of the samples. The thermocouple voltage was read through an ice point by a Hewlett Packard 3456A digital voltmeter. Temperature was calculated using a fifth-order polynomial fit applied to the voltage vs. temperature calibration data that accompanied the Pt/Pt-10%Rh thermocouple. The same precision thermocouple was used in both furnaces. All experiments were performed in atmospheric air at atmospheric pressure; data in the literature [9] indicate that the rate of oxidation of superalloys is not sensitive to the oxygen partial pressure.

Several approaches were used to accumulate data based on the particular phenomenon being studied. For the twenty-four-hour parabolic rate tests and the long-term low-temperature oxidation tests, an array of six to eight samples was accurately weighed and then placed into tare-weighed crucibles. The array was then placed in a refractory holder and the entire array and holder inserted into the muffle furnace that had already been preheated to the required temperature. At appropriate time intervals, single samples were withdrawn from the furnace. Prior to withdrawal of a crucible from the furnace, a cover was placed on the crucible to prevent any mass loss due to flaking of the oxide as the samples cooled. After cooling to slightly above room temperature, the crucible containing the sample was placed in a desiccator overnight prior to weighing. The crucible and sample were then weighed as a unit, the sample was then removed and the crucible cleaned of debris, and then the crucible was weighed empty. Combining this data with the pre run data allowed us to isolate the mass change in the sample material due to reactions with oxygen in the air from any unexpected mass change in the crucible. At the conclusion of post test measurements, all data were input to a spreadsheet for quality assurance and analysis. For the high-temperature single-point data, samples were installed into and removed from the furnace individually. Similar steps were taken to accurately measure the mass changes of the samples and crucibles. Due to the high temperatures involved, alumina crucibles and covers were used exclusively for these tests. A discussion of the test series and their results follows.

## **DISCUSSION OF RESULTS**

Three series of experiments were performed to investigate the oxidation resistance of Inconel 718 in air at high temperatures. The first of these series was intended to study the parabolic oxidation rate behavior of Inconel 718 in the range of temperature from 1173 K to 1573 K and to evaluate the temperature bounds of the diffusion-controlled oxidation range. The second series of experiments was performed to investigate the oxidation rate at very high temperatures, from the upper limit of the parabolic rate range to beyond failure, to establish a conservative failure limit for accident analyses. The third series of experiments was performed at lower temperatures, from 973 K to 1223 K, to examine the lower limit of the parabolic oxidation rate range. These three series of experiments will be discussed in order below.

## **Parabolic Oxidation Rate (24-hour tests) Series, 1173 K-1573 K**

Six series of experiments were conducted to measure the oxidation resistance of Inconel 718 in the temperature range of most interest for postulated loss-of-coolant accident conditions in the APT target. The temperature range of interest and the temperature range of these experiments was 1173 K to 1573 K (900°C to 1300°C) in increments of 100 K. There was considerable evidence in the published literature that, over this range of temperature, the rate of oxidation of superalloys exhibits a parabolic rate dependence [4-5, 8-12]. Parabolic rate dependence refers to a phenomenon in which transport (mass, momentum or energy) is diffusion-controlled and the solution to the parabolic differential equation results in a time-dependence in which the rate of change of the dependent variable is proportional to the square root of time. As a result, comparison of the square of the oxidation mass increase vs. time should be linear if the oxidation rate behavior is indeed parabolic. In order to test this hypothesis, six series of experiments were performed, each with from six to seven samples, in a temperature-controlled laboratory furnace for various time intervals. The procedure that was followed was to install all the test specimens into the preheated furnace at the same time, then to remove them after the prescribed elapsed time interval which ranged in duration from one to 36 hours. For the test series at 1173 K, 1273 K, 1373 K and 1473 K, samples were removed from the furnace at intervals of one, three, five, eight, 16 and 24 hours (the 1473 K test series had a seventh sample which was removed from the furnace after 36 hours). Handling of the samples for the test series at 1573 K was extremely difficult due to the temperature of the furnace; therefore, for this test series, samples were instead removed at intervals of three, five, 12 and 24 hours.

Upon removal from the furnace, each sample was labeled and placed, along with its crucible, into a desiccator for humidity-free storage prior to post test weighing. The oxidized samples were first weighed in their crucibles, then the samples were removed and placed in zip-lock bags for archival storage. The empty crucibles were then reweighed. When the data just collected was input to a spreadsheet, these data allowed for the mass gain of the samples to be precisely calculated to an accuracy of  $\pm 0.1$  mg. This procedure was identical for all the tests conducted. The data for mass gain per unit area vs. oxidation time for the six series of parabolic oxidation rate tests are listed in Table 2. Two series of tests were conducted at 1173 K with the designations "maximum" and "minimum." These designations simply indicate that one set of data resulted in an upper-limit oxidation rate and the other set of data resulted in a lower-limit oxidation rate at 1173 K. Two sets of data were taken at 1173 K because it is generally found that oxidation rate behavior can be more variable at lower temperatures. Additional data which was taken at the other temperatures but not reported here demonstrated less variability at higher temperatures.

The data for these parabolic oxidation rate test series are presented in Figure 1 as the square of the mass gain per unit area vs. time of oxidation to evaluate the parabolic dependence of the measured oxidation data. The data exhibit some scatter at early times as be expected. Over the duration of each of the five series of data presented in Figure 1, parabolic dependence of the oxidation rates is manifest as demonstrated by the apparently linear dependence of the square of the mass gain per unit area vs. time. The data for the 1173 K series shown in Figure 1 are the set designated as "maximum"

in Table 2. The numerical values for the parabolic oxidation rate coefficients which were determined by linear least-squares analyses of these data are also shown on Figure 1 as  $K_p$  ( $\text{mg}^2/\text{cm}^4\cdot\text{s}$ ). These coefficients and the analysis of the data which determined these coefficients will be discussed in the following section.

### Correlations of Parabolic Oxidation Rate Data

The data for five of the six sets of parabolic oxidation rate tests are shown graphically in Figure 1 and the results of all six series are listed in Table 2. The data in Figure 1 clearly demonstrate the expected parabolic rate behavior; the square of the mass gain per unit area is shown to increase linearly with increasing time, in one case (1473 K) for up to 36 hours of oxidation in air. Each set of parabolic oxidation data was curve fit by a linear least-squares algorithm to a function of the form,

$$(m'')^2 = K_p \cdot t \quad (1)$$

where  $m''$  is the oxidation mass gain of the sample per unit surface area ( $\text{mg}/\text{cm}^2$ ),  $t$  is the oxidation time in seconds and  $K_p$  is the parabolic oxidation rate coefficient to be determined.

The parabolic oxidation rate coefficients for five of the six series of experiments so calculated are shown on Figure 1 along with linear lines which represent the least-squares curve fits to the data; the coefficients for all six sets of experiments are listed in Table 3. Over the temperature range from 1173 K to 1573 K ( $900^\circ\text{C}$  to  $1300^\circ\text{C}$ ), the parabolic oxidation rate coefficients and thus, the oxidation rates of Inconel 718 in air, vary by almost three orders of magnitude from a low of  $2.67 \times 10^{-6} \text{ mg}^2/\text{cm}^4\cdot\text{s}$  at 1173 K to a high of  $2.42 \times 10^{-3} \text{ mg}^2/\text{cm}^4\cdot\text{s}$  at 1573 K. It is worth noting again that the mass gain calculated by application of the parabolic oxidation rate coefficient in Table 3 with Eq. (1) result in the mass of oxygen gained by the test sample due to oxidation. What was measured in these tests is mass gain by oxygen uptake. This can be converted to the mass of Inconel 718 consumed by the oxidation by a simple conversion which will be discussed shortly. The data in Table 3 are single-temperature parabolic oxidation rate coefficients and, for practical applications, should be generalized over the entire range of temperature from 1173 K to 1573 K. An Arrhenius relationship of the functional form,

$$K_p = A \cdot \exp[-B/RT] \quad (2)$$

was sought where  $K_p$  is the parabolic oxidation rate coefficient,  $A$  and  $B$  are integration constants,  $R$  is the universal gas constant and  $T$  is the temperature. In a thermodynamic sense,  $B$  would be the activation energy but, in the present formulation, that will not be the case. The data from these tests were not precise enough to calculate the activation energy by regression analysis. In order to evaluate the appropriate ranges of temperature over which to fit  $K_p$  as a function of temperature, the values in Table 3 were plotted vs. temperature and are shown in Figure 2. On this basis, three ranges of temperature were selected for three curve fits for  $K_p$ : 1173 K to 1273 K, 1273 K to 1473 K, and 1473 K to 1573 K. A linear regression analysis of the form of Eq. (2) resulted in the following relationships for  $K_p$  as a function of temperature:

$$K_p = (5.59 \times 10^5) \exp[-29559/T] \quad \text{for} \quad T = 1173 \text{ K} - 1273 \text{ K} \quad (3a)$$

$$K_p = (2.19) \exp[-13754/T] \quad \text{for} \quad T = 1273 \text{ K} - 1473 \text{ K} \quad (3b)$$

$$K_p = (3.93 \times 10^{13}) \exp[-58690/T] \quad \text{for} \quad T = 1473 \text{ K} - 1573 \text{ K} \quad (3c)$$

The values of  $K_p$  so calculated are in units of ( $\text{mg}^2/\text{cm}^4 \cdot \text{s}$ ). When  $K_p$  from Eq. (3) is input into Eq. (1), the square of the oxygen mass gain per unit surface area over the time interval of oxidation (in seconds) is computed as ( $\text{mg}^2/\text{cm}^4$ ). Of course, the square root of Eq. (1) yields the oxygen mass gain per unit surface area of the sample.

As a first approximation, Inconel 718 can be considered as a pseudo element with a composition of roughly 58% Ni, 21% Cr and 21% Fe, resulting in a mixture with an effective atomic mass of 56.7 AMU. Oxygen has an atomic mass of 16.00 AMU. Assuming that during oxidation all oxides formed are mono-atomic (ie., CrO, NiO and FeO) which would be the worst-case oxidation rate, then the ratio of the mass of Inconel 718 oxidized to the mass of oxygen gained due to oxidation would be 3.54. In other words, if the sample gained one gram of mass during oxidation due to oxygen uptake, then 3.54 grams of Inconel would have been oxidized. Multiplying Eq. (1) by  $(3.54)^2$  will give the mass of Inconel 718 consumed by oxidation; using the Inconel 718 density of  $8.22 \text{ gm}/\text{cm}^3$ , the average depth of penetration of the oxidation into the surface of the metal can be calculated. Penetration depth estimates made by this procedure will result in maximum penetration estimates due to the conservative assumptions in the oxidation chemistry stoichiometry.

#### **Ultra-High Temperature (single-sample tests) Data, 1474 K-1614 K**

The parabolic oxidation rate coefficients in Eq. (3) can be input to Eq. (1) to calculate the extent of the mass gain of Inconel 718 during oxidation by the uptake of oxygen, assuming mono-atomic stoichiometry of the oxide products. This mass gain can be readily converted to the mass of Inconel 718 consumed by oxidation simply by multiplying the oxygen mass gain per unit surface area by the conversion factor of 3.54, to convert from oxygen mass gain to Inconel mass consumed. The parabolic oxidation rate coefficients in Eq. (3) are applicable over the temperature range from 1173 K to 1573 K. Simple extrapolation to temperatures outside this temperature range is not recommended. A prescription for extrapolation to lower temperatures will be discussed in a subsequent section. To evaluate oxidation resistance of Inconel 718 at higher temperatures, another series of tests was performed which will now be discussed.

A series of high-temperature oxidation tests was performed over the temperature range of 1474 K to 1614 K ( $1201^\circ\text{C}$  to  $1341^\circ\text{C}$ ). The purpose of these tests was to evaluate the upper-temperature limit of Eq. (3c) and to prescribe a conservative upper-temperature threshold, above which the Inconel 718 would fail to provide any structural function, especially the function of a clad on the tungsten cylinders of the APT target. A total of 14 single-sample tests was performed; ten of these tests were performed in the temperature range 1473 K to 1573 K in nominal increments of 10 K, and four tests were performed at temperatures above 1573 K. The duration of each of the tests in this series was nominally 24 hours. This methodology was adopted in order to "walk through" the upper limit of the parabolic oxidation rate range for Inconel 718 to observe any deviations from the

oxidation-rate coefficient correlation previously derived [Eqs. (3a-c)], and to pinpoint the thermal failure threshold for the material. The numerical data for all 14 tests are listed in Table 4; thirteen of these points are plotted on Figure 3 which is identical to Figure 2 with the addition of the 13 data points superimposed for contrast. [Note: the 14<sup>th</sup> data point at 1614 K was excluded from Figure 3 because the test sample was essentially consumed during the experiment and destroyed. It would be incorrect to infer an oxidation rate from this datum.]

Single-sample oxidation data would be expected to show some variability from the integrated  $K_p$  correlation for the 1473 K to 1573 K temperature range, just as the individual data points on Figure 1 demonstrate variation from the linear least-squares lines plotted there. What is impressive about the single-sample data on Figure 3 in the 1473 K-1573 K temperature range is the remarkably close agreement between the individual single-sample data points and the curve fit [Eq. (3a)] from 1473 K to 1573 K which is based on the parabolic oxidation rate series listed in Table 2. In fact, most of the data points actually "hit" the 1473 K-1573 K line on Figure 3, the differences being credited to unresolved (but small) temperature fluctuations in the laboratory furnaces used for these tests. The single-sample high-temperature data clearly validate the correlation for the parabolic oxidation rate coefficient,  $K_p$ , in this temperature range.

Once the temperature was increased above 1573 K, the values of  $K_p$  inferred from the data (see Table 4) began to increase asymptotically, suggesting the onset of catastrophic oxidation in this range of temperature for Inconel 718. The cylindrical test specimens were visually inspected after oxidation for evidence of morphological changes which could be used as the basis for the determination of a high-temperature service limit for the Inconel 718. In all cases, the surfaces of the Inconel 718 samples blackened during oxidation and, with the exception of some very light, dispersible oxide powder on the surfaces, the oxide layers appeared rugged and dense. These oxide scales did not flake off the samples but adhered to the surfaces and protected the underlying metal from direct contact with the oxidizing atmosphere. This is the fundamental reason why the oxidation was parabolic, i.e. growth was limited by diffusion through the growing oxide scale [7-8, 11-13]. Those samples which were oxidized at temperatures in excess of 1540 K (~1270°C) were observed to have sagged into a bow-shape under their own weight (the samples were placed into a laboratory furnace in refractory crucibles; the samples were supported in the crucibles only at their ends to insure that none of the surface area would be obscured from oxidation). At temperatures in excess of 1560 K (~1285°C), prominent rivulets were observed to have formed on the surfaces of the cylindrical samples running parallel to the cylinder axis. These rivulets became deeper as the temperatures increased, soon becoming deeper than the 0.005 inch clad thickness proposed for the APT target. The oxide layers in those tests which developed rivulets became mechanically unstable and spalled off easily. It was not possible to determine if the spalling occurred at temperature or upon cooling due to contraction (the latter behavior would be non prototypic for the APT case). Regardless, the spalling behavior could not be tolerated in an application as a clad material for APT. The sample in the final test in this series at 1614 K (1341°C) was essentially consumed into a twisted mass of oxide with some evidence of liquefaction and void formation present in the debris. It was concluded from these observations that the Inconel 718 samples exhibited unacceptable morphological deformations at temperatures above 1538 K (1265°C). A conservative temperature limit for the

application of Inconel 718 as a clad in a high-temperature oxidizing environment (for APT) was assigned at 1523 K (1250°C). Above this temperature, the clad is assumed to provide no protection for the underlying tungsten from contact with the oxidizing/vaporizing atmosphere.

### **Low-Temperature, Long-Term (9-14 day tests) Oxidation Rate Series, 973 K-1223 K**

In addition to the parabolic oxidation rate test series which were performed over the temperature range of 1173 K to 1573 K and the ultra high temperature oxidation series (1474 K to 1614 K) which were executed to evaluate high-temperature performance, a third series of tests was conducted at low temperatures to evaluate the oxidation performance of Inconel 718 at temperatures below the parabolic range previously identified. In particular, the intent of these tests was to examine the lower limit of the parabolic oxidation rate range and to examine departure from diffusion-controlled oxidation and the onset of passivation. It was previously cautioned against extrapolation of Eq. (3c) to temperatures above 1573 K for fear that the oxidation rate could be significantly under predicted; these tests at low temperatures address the consequences of extrapolation of Eq. (3a) to temperatures below 1173 K, here the concern being over prediction of the calculated oxidation rates. In order to evaluate the low-temperature performance of Inconel 718, four series of tests were performed at temperatures 973 K, 1073 K, 1173 K and 1223 K (700°C, 800°C, 900°C and 950°C, respectively). Each series of tests at an assigned temperature consisted of from four to eight separate samples which were simultaneously inserted into a pre heated laboratory furnace in air and selectively removed at prescribed time intervals for evaluation. The procedures that were followed were identical to those described in the "Parabolic Oxidation Rate . . ." section of this report with the exceptions that the temperatures were lower as just described and the overall durations of the low-temperature tests ranged from nine to 14 days.

The mass gain data for each of these low-temperature test series are presented in Figure 4, which is identical to Figure 1 for the parabolic oxidation rate tests. In Figure 1, it is shown that parabolic oxidation behavior persists for up to 24 hours for temperatures as high as 1573 K and as low as 1173 K. The long time, low temperature data in Fig. 4 indicate that although parabolic behavior persists for long times (up to nine days) at 1223 K, a reduction of the temperature to 1173 K and below results in an apparent departure from parabolic oxidation after as little as 24 hours. It seems that at these low temperatures, Inconel 718 experiences a period of transient oxidation for approximately 24 hours of oxidation in air, followed by parabolic oxidation at a greatly reduced rate. In other words, nearly all the oxidation occurs during the first 24-hour transient of exposure to air at temperatures below 1223 K. This suggests that although parabolic oxidation growth is likely persisting for long times at low temperatures, the rate is so small as to be immeasurable and the oxide scale ceases to penetrate the metal. No further mass gains due to oxidation were measurable for up to 15 days as evidenced by the data. Since the first sample from each of the four test series was removed from the furnace at 24 hours, no details of the low temperature oxidation behavior during the first 24-hour transient were obtained. It is clear from Fig. 4 that for the purpose of calculating the depth of oxide penetration, the Inconel 718 became effectively passivated during the first 24 hours of exposure and that continued oxidation of the samples was interrupted by the oxide scale which had formed during the initial 24-hour interval.

A methodology which could be adopted to calculate the oxidation of Inconel 718 at temperatures below 1223 K is recommended as follows. The parabolic oxidation rate coefficient given by Eq. (3a) is recommended with Eq. (1) for the temperature range of 1173 K to 1273 K for long times. Once the temperature falls to 1173 K, Eq. (3a) is recommended with Eq. (1) for only 24 hours; after this 24-hour period, the surface should be considered passivated and no further oxidation considered. This procedure would allow for extrapolation of Eq. (3a) to low temperatures with the results of Figure 4 as guidance to determine the onset of passivation consistent with experimental observations. Calculations of the extent of oxidation of Inconel 718 in this low-temperature range would be slight, indeed.

## CONCLUSIONS

Several series of experiments were performed to investigate the oxidation resistance of Inconel 718 in air at elevated temperatures. The first in these series of tests was intended to study the parabolic oxidation rate behavior of Inconel 718 in air in the temperature range 1173 K to 1573 K. The second series of tests was intended to investigate the oxidation rate of Inconel 718 at very high temperatures in the range 1473 K to 1614 K to examine the departure from a parabolic oxidation rate and to determine a conservative temperature threshold, above which the Inconel 718 would fail to provide any structural function. The third series of tests was performed at low temperatures in the range 973 K to 1223 K to examine the lower limit of the parabolic oxidation rate range and the onset of passivation. The following conclusions can be drawn from the results of these tests.

1. Inconel 718 exhibits parabolic, diffusion-controlled oxidation rate dependence in air at atmospheric pressure over a wide range of temperature, from 1223 K to 1573 K, for long time intervals up to several weeks.
2. At temperatures above 1573 K, oxidation becomes catastrophic and the material becomes severely deformed. Inconel 718 begins to deform under the load of its own weight at about 1540 K. At 1560 K, deep rivulets appear in the surface of the material.
3. At 1173 K and below, Inconel 718 experiences a period of transient oxidation for approximately 24 hours of oxidation in air. After 24 hours, continued exposure to air at temperatures below 1173 K results in no further measurable weight gain due to oxidation of the metal. This suggests that, although parabolic oxidation growth is likely persisting for long times at low temperatures, the rate is so small as to be immeasurable and, for practical purposes, the growth of the oxide scale into the metal stops.
4. It is concluded from these tests that Inconel 718 would perform satisfactorily as a clad for the APT tungsten spallation target for temperatures up to 1523 K (1250°C) in air.

## **AUTHORS' END NOTE**

Three citations were retrieved during the progress of this investigation which are included with the references [14-16] but have not been discussed in the text. All three citations describe chemical/metallurgical interactions between structural materials such as Inconel and aluminum with other metals which may be in the system, such as lead, Zircaloy-4 and mercury. These three citations address other additional mechanisms to the oxidation phenomena considered in the above experiments which should be considered (if applicable) to insure that designs are not compromised.

## REFERENCES

1. "Preliminary Safety Analysis Report," Accelerator Production of Tritium, PPO-POO-G-PSA-X-00001, Los Alamos National Laboratory (1998).
2. "APT Conceptual Design Report," Accelerator Production of Tritium, LA-UR-97-1329, Los Alamos National Laboratory(1997).
3. "High Temperature, High Strength Nickel Base Alloys," The International Nickel Company, Inc., 3rd Edition, p. 4 (1977).
4. Hussain, N., K. A. Shahid, I. H. Khan and S. Rahman, "Oxidation of High-Temperature Alloys (Superalloys) at Elevated Temperatures in Air: I," Oxidation of Metals, 41(3/4), pp. 251-269 (1994).
5. Kumar, L., R. Venkataramani, M. Sundararaman, P. Mukhopadhyay and S. P. Garg, "Studies on the Oxidation Behavior of Inconel 625 Between 873 and 1523 K," Oxidation of Metals, 4(½), pp. 221-244 (1996).
6. Hussain, N., K. A. Shahid and S. Rahman, "Oxidation of High-Temperature Alloys (Superalloys) at Elevated Temperatures in Air: II," Oxidation of Metals, 43(3/4), pp. 363-378 (1995).
7. Zaplatynsky, I., "Volatilization of Oxides During Oxidation of Some Superalloys at 1200°C," Oxidation of Metals, 11(6), pp. 289-305 (1977).
8. Royster, D. M. and W. B. Lisagor, "Effect of High-Temperature Creep and Oxidation on Residual Room-Temperature Properties for Several Thin-Sheet Superalloys," NASA TND-6893 (1972).
9. Christ, H. J., L. Berchtold and H. G. Sockel, "Oxidation of Ni-Base Alloys in Atmospheres With Widely Varying Oxygen Partial Pressures," Oxidation of Metals, 26(½), pp. 45-76 (1986).
10. Clark, R. K. and J. Unnam, "Response of Inconel 617 to Sea-Salt and Re-Entry Conditions," J. Spacecraft, 23(1), pp. 96-101 (1986).
11. Lenglet, M., R. Guillaumet, J. Lopitiaux and B. Hannover, "Caracterisation des Premieres Etapes de l'Oxydation de l'Alliage Inconel 718 par Spectrometrie I. R. F. T.," Mat. Res. Bull., 25, pp. 715-722 (1990).

12. Allen, G. C., J. M. Dyke, S. J. Harris and A. Morris, "The Oxidation of Inconel 690 Alloy at 600K in Air," Applied Surface Science, 31(2), pp. 220-238 (1988).
13. Lorang, G., N. Jallerat, K. V. Quang and J. P. Langeron, "AES Depth Profiling of Passive Overlayers Formed on Nickel Alloys," Surface and Interface Analysis, 16(1-12), pp. 325-330 (1990).
14. Hofmann, P. and M. Markiewicz, "Chemical Interaction Between Zircaloy-4 and Inconel 718," Transactions Thirteenth International Conference on Structural Mechanics in Reactor Technology, Porto Alegre, Brazil, CONF-950804, 1, pp. 145-152 (13-18 August 1995).
15. Garcia-Mazario, M., "Effect of Lead on Inconel 600 and Incoloy 800 Oxide Layers Formed in Simulated Steam Generator Secondary Environments," Nucl. Engr. Des., 167(2), pp. 155-167 (1996).
16. Czajkowski, C. J., S. Usmani and G. A. Greene, "Effects of Mercury on Thermally Sprayed Aluminum Coatings," Materials Characterization, 43(2/3), pp. 187-194 (1999).

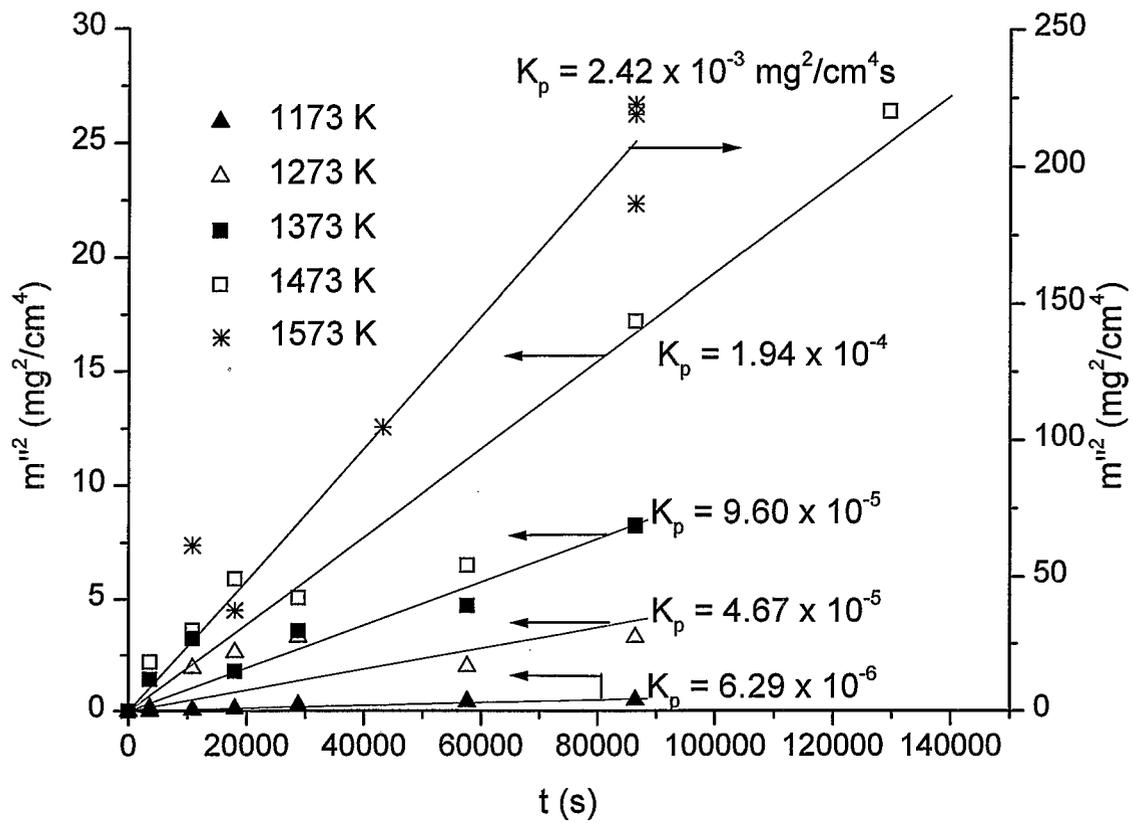


Figure 1. Square of the mass gain per unit area vs. time for Inconel 718 oxidized in air between 1173 K and 1573 K for up to 36 hours at atmospheric pressure.

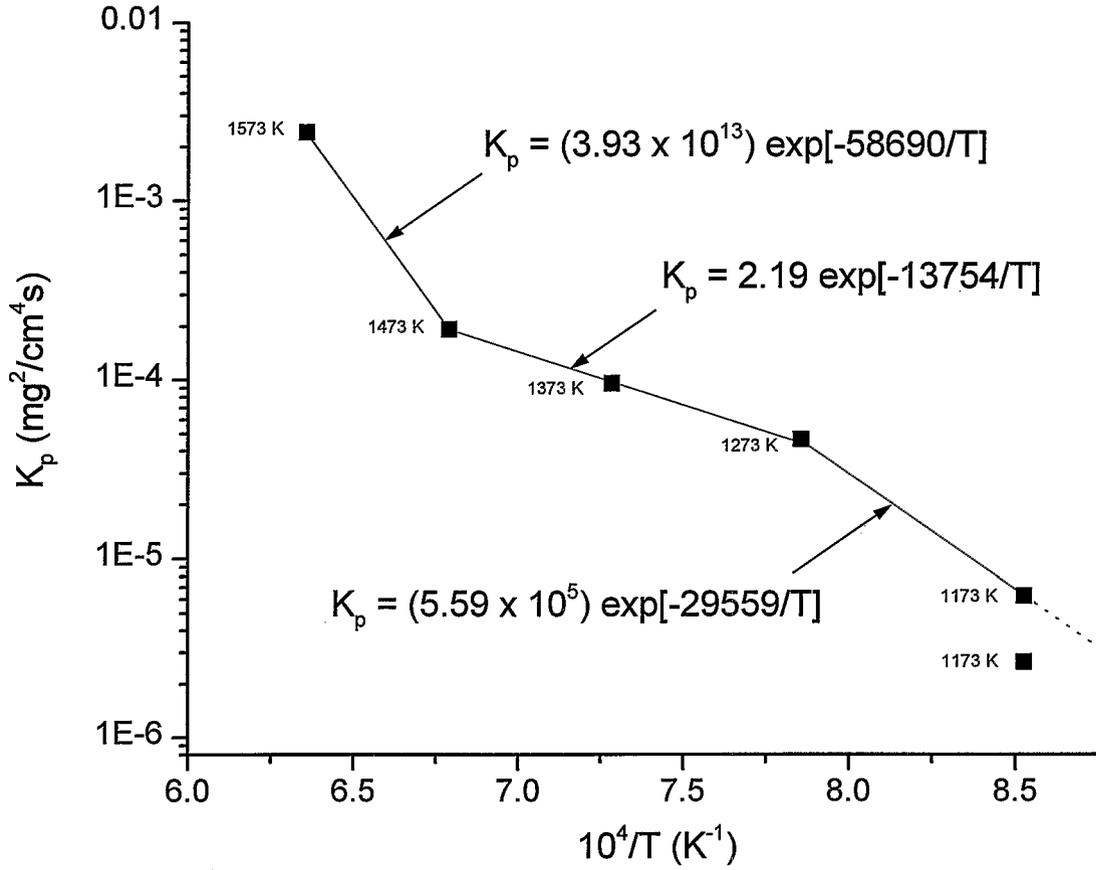


Figure 2. Correlations of the parabolic oxidation rate coefficients  $K_p$  vs.  $1/T$  for Inconel 718 oxidation in air at temperatures between 1173 K and 1573 K.

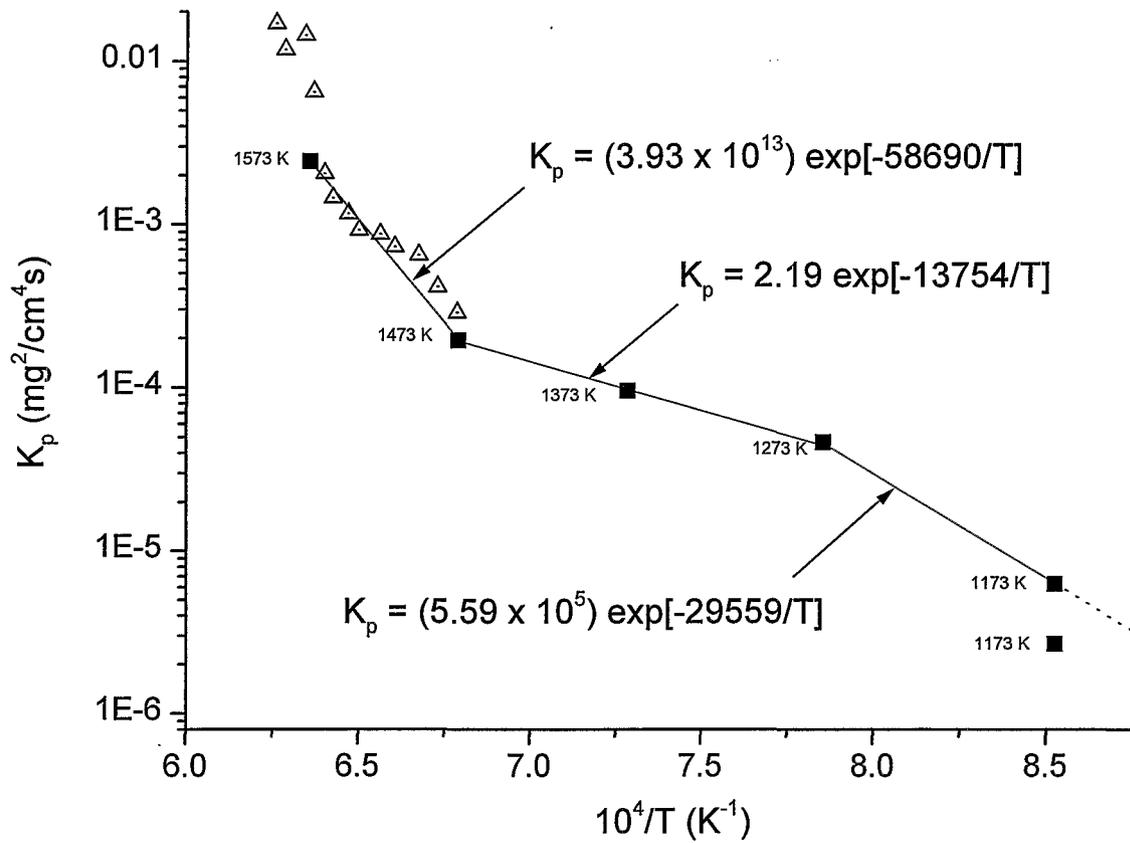


Figure 3. High temperature parabolic oxidation rate coefficients  $K_p$  vs.  $1/T$  for Inconel 718 oxidation in air at temperatures between 1473 K and 1620 K.

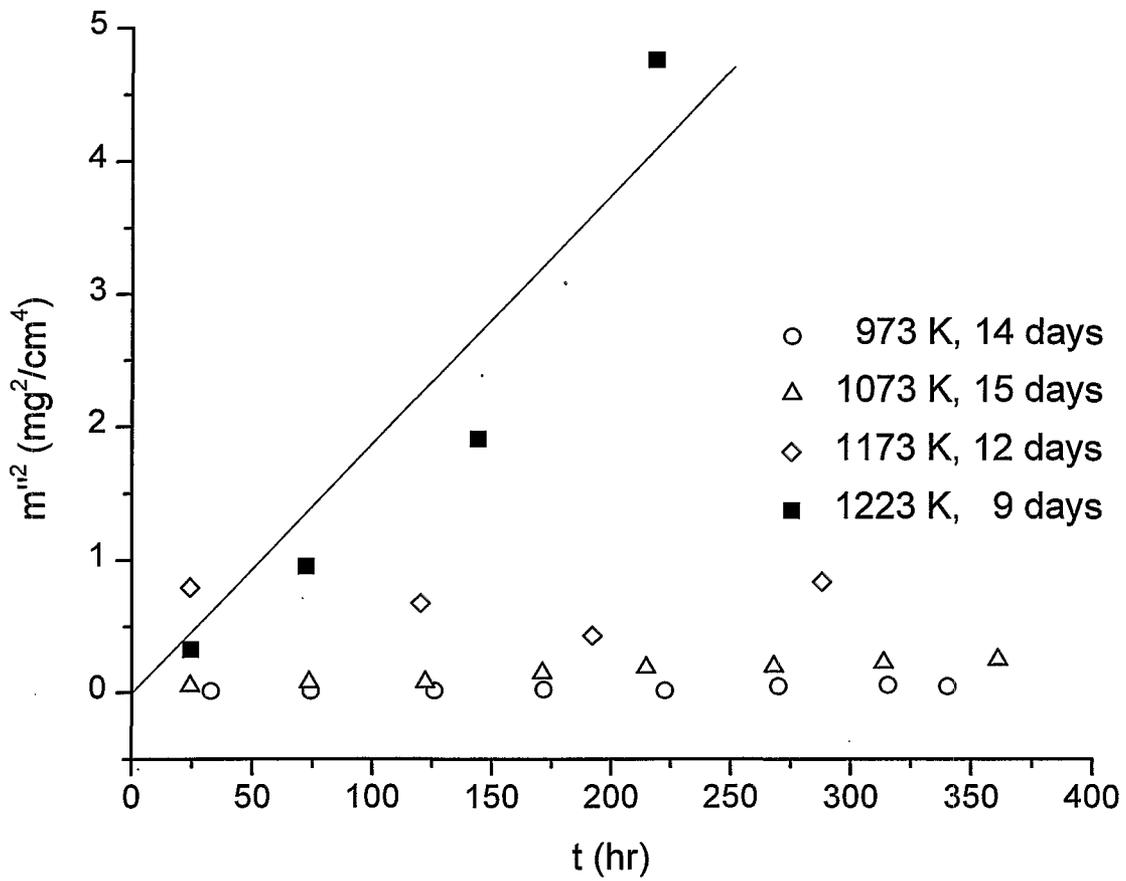


Figure 4. Square of the mass gain per unit area vs. time for Inconel 718 oxidized in air between 973 K and 1223 K for nine to 15 days at atmospheric pressure (linear fit to the 1223 K data is for illustration purposes only).

**Table 1**

**Constituents of Inconel 718 [3]**

<b>Constituent</b>	<b>% of Weight</b>
Ni	52.5
Cr	19.0
Fe	18.5
Nb	5.2
Mo	3.0
Ti	0.8
Al	0.6
Si	0.2
Mn	0.2

**Table 2**

**Data Sets for Parabolic Oxidation Rate Correlations for Inconel 718 in Air**

Temperature T (K)	Mass Gain/Unit Area $\Delta m/A$ (mg/cm <sup>2</sup> )	Oxidation Time t (s)
1173	maximum	3.6 x 10 <sup>3</sup>
	0.128	
	0.239	
	0.312	
	0.509	
	0.653	
1273	0.687	0.047
	0.139	10.8
	0.209	18.0
	0.302	28.8
	0.373	57.6
	0.487	86.4
1373	0.401	3.6
	1.378	10.8
	1.623	18.0
	1.817	28.8
	1.412	57.6
	1.815	86.4
1473	1.188	3.6
	1.794	10.8
	1.324	18.0
	1.885	28.8
	2.166	57.6
	2.862	86.4
1573	1.482	3.6
	1.899	10.8
	2.432	18.0
	2.243	28.8
	2.558	57.6
	4.134	86.4
1573	5.124	129.6
	7.852	10.8
	6.133	18.0
	10.228	43.2
	14.762	86.4
	14.916	86.4
1573	13.680	86.4

**Table 3**

**Parabolic Oxidation Rate Coefficients  $K_p$  for Oxidation of Inconel 718 in Air  
Used to Construct the Oxidation Correlations in Figure 2**

<b>Temperature (K)</b>	<b><math>K_p</math> (mg<sup>2</sup>/cm<sup>4</sup>·s)</b>
1173	$6.29 \times 10^{-6}$ (maximum)
1173	$2.67 \times 10^{-6}$ (minimum)
1273	$4.67 \times 10^{-5}$
1373	$9.60 \times 10^{-5}$
1473	$1.94 \times 10^{-4}$
1573	$2.42 \times 10^{-3}$

**Table 4****Ultra-High Temperature 24-Hour Oxidation Rate Data <sup>[1]</sup>**

<b>Temperature T (K)</b>	<b><math>\Delta m/A</math> (mg/cm<sup>2</sup>)</b>	<b>Oxidation Time t (s)</b>	<b><math>K_p</math> <sup>[2]</sup> (mg<sup>2</sup>/cm<sup>4</sup>·s)</b>
1474	4.778	79.5 x 10 <sup>3</sup>	2.87 x 10 <sup>-4</sup>
1486	5.737	80.0	4.11 x 10 <sup>-4</sup>
1499	6.967	75.1	6.47 x 10 <sup>-4</sup>
1514	7.737	82.5	7.26 x 10 <sup>-4</sup>
1524	8.244	78.3	8.68 x 10 <sup>-4</sup>
1539	8.774	83.7	9.20 x 10 <sup>-4</sup>
1546	9.849	83.7	1.16 x 10 <sup>-3</sup>
1557	10.734	79.5	1.45 x 10 <sup>-3</sup>
1563	13.125	84.3	2.04 x 10 <sup>-3</sup>
1570	23.242	83.2	6.49 x 10 <sup>-3</sup>
1576	34.467	82.5	1.44 x 10 <sup>-2</sup>
1591	30.839	80.7	1.18 x 10 <sup>-2</sup>
1598	36.982	80.7	1.69 x 10 <sup>-2</sup>
1614 <sup>[3]</sup>	194.205	80.7	4.67 x 10 <sup>-1</sup>

1. Due to the rapid oxidation rates at these ultra high temperatures, these tests were conducted with single samples and tested in nominally 24-hour exposures.

2. The parabolic oxidation rate coefficients listed here are inferred from the following relationship,  $K_p = (\Delta m/A)^2/t$ .

3. This data point has been omitted from Figure 3 because the test sample was essentially consumed during the experiment and destroyed. It would be misleading to infer an oxidation rate from this datum. This represents a temperature at which the Inconel 718 is rapidly destroyed.