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Progress Report

to

The International Nickel Company, Inc.

on

# THE EFFECTS OF LEAD ON THE CREEP-RUPTURE PROPERTIES AND HOT-WORKABILITY OF TWO NICKEL-CHROMIUM-TITANIUM-ALUMINUM ALLOYS

by

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# INTRODUCTION

Experimental research was conducted with the objective of quantitatively determining the adverse effects of lead on the creep-rupture properties and hot-workability of two vacuum-induction melted nickel-chromium-titanium-aluminum alloys. Lead contents up to 100 ppm (0.01-percent) were thoroughly investigated. The alloys studied had the following nominal compositions in weight percent:

| C | Ti | Al | Cr | Ni | Mn and Si |
|---|---|---|---|---|---|
| 0.04-0.06 | 2.5 | 0.8 | 20.0 | balance | low as possible |
| 0.05-0.07 | 3.8 | 1.2 | 20.0 | balance | low as possible |

The heats were split with 0.0035-percent boron and 0.045-percent zirconium being added to one of the two ingots from each heat.

Lead was studied because it can be an inadvertant contaminant which could be detrimental to both creep-rupture properties and hot-workability. It had been suggested that lead would be highly deleterious in nickel-base heat-resistant alloys[1, 2*] and implications were that vacuum melting could readily remove lead by distillation and thereby improve properties[3, 4]. Accordingly, a program was devised where varied amounts of lead ranging from 10 to 100 ppm were added directly to heats and, in selected instances, the heats were subsequently held in vacuum. These heats were completely processed and tested with the objectives of accurately determining the effects of lead and to what extent it could be removed by vacuum refining and properties restored; thus delineating how a harmful trace element acts in nickel-base alloys.

About the time this work was completed, Peter and Spitzer[5] and Wood and Cook[6] published papers containing limited quantitative data on the effects of lead in typically commercial nickel-base heat-resistant alloys. Their data is in excellent agreement with the results obtained here.

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*The superscripts refer to literature listed under REFERENCES.

A split-heat procedure was adopted to provide material with and without the combined boron and zirconium additions. Past experience[7] had demonstrated that boron and zirconium were essential to produce good quality materials to the types being studied. On the other hand, any effects of lead would have been difficult to interpret without data from boron and zirconium-free material. The split-heat procedure consisted of pouring the first ingot with no boron and zirconium addition and pouring the second ingot after simultaneously adding 0.0035-percent boron and 0.045-percent zirconium to the melt.

The ingots were hot rolled to bar-stock and the bar-stock heat treated. Sufficient creep-rupture tests were conducted at 1200° and 1500°F to establish the 100-hour rupture strength as obtained from stress-rupture time curves. Effects of lead on hot-workability were qualitatively estimated by behavior during hot rolling.

The compositions of the alloys were selected to provide a 1.8 to 1 atomic ratio of titanium to aluminum at two levels of the $Ni_3(Al, Ti)$, $\gamma'$, strengthening precipitate. Decker[8] found this ratio to result in the largest difference between the lattice parameters of the $\gamma$ matrix and the $\gamma'$ precipitate in nickel-titanium-aluminum alloys and it was expected to optimize creep-rupture properties for a given combined total of titanium and aluminum.

# EXPERIMENTAL PROCEDURE

## General Approach

All melting was done in a vacuum-induction furnace using high-purity alumina crucibles. The heats weighed approximately 5500 grams (12 pounds). The general program of heat preparation was as follows:

a) Make base alloys, splitting each heat without and with a combined boron and zirconium addition, to establish melting procedures for composition control and to delineate base properties.

b) Establish the influence of lead by adding up to 1000 ppm lead to different heats, again splitting each heat without and with a combined boron and zirconium addition.

c) Perform vacuum-distillation-rate experiments on heats having prior intentional lead additions to establish the utility of vacuum melting for removing lead.

All ingots were hot rolled to bar-stock, the bar-stock sectioned for chem samples and test specimen slugs, and the specimen slugs heat treated.

The evaluation of the influence of lead on the creep-rupture properties of the two alloys was based on the results of creep-rupture tests at 1200° and 1500°F. Two or more tests were run for each heat to establish stress-rupture time curves defining the 100-hour rupture strengths. Hot-workability was qualitatively estimated by observations of working characteristics during rolling at 2150°F.

## Melting Stock and Crucibles

The raw materials used were selected by mutual agreement with representatives of the International Nickel Company, Inc. Generally, the purest materials available in the quantities needed for the investigation were selected.

### Nickel

Mond carbonyl nickel pellets were used for all heats. Three 200-pound pails were furnished by the International Nickel Company, Inc. A chemical analysis made on a sample from one pail of pellets was as follows, in weight percent:

| C | S | Pb | B | Si | Mn | Fe | Co |
|---|---|---|---|---|---|---|---|
| 0.013 | 0.0030 | <0.001 | <0.001 | <0.005 | <0.005 | 0.011 | <0.01 |

| Cu | P | Mg | Al | Ti | Zr |
|---|---|---|---|---|---|
| <0.005 | 0.002 | <0.005 | <0.01 | <0.01 | <0.01 |

### Chromium

Shieldalloy aluminum-reduced "VMG" chromium metal was used in all heats. Past experience at the University of Michigan indicated that this type of chromium was easily alloyed during vacuum melting and was of as high a purity feasible in the quantities needed. Four different pails were purchased from the Shieldalloy Corporation with the chemical analysis of each being almost identical. A typical analysis is as follows, in weight percent:

| C | S | Si | Fe | Al | Cr |
|---|---|---|---|---|---|
| 0.01 | 0.012 | 0.12 | 0.21 | 0.06 | 99.43 |

### Titanium

On advice from representatives of the International Nickel Company, Inc., Dupont sponge titanium was used throughout the investigation. The analysis supplied with the pail purchased reported only 0.06-percent iron as an impurity. A sulfur analysis made by the International Nickel Company, Inc., indicated 0.0020-percent of that element.

### Aluminum

The aluminum was supplied gratis by the Aluminum Company of America as 99.99-percent aluminum ingot-1 pound notch bars with the following analysis, in weight percent:

| Cu | Fe | Si | Mg |
|---|---|---|---|
| 0.001 | 0.001 | 0.003 | 0.002 |

### Boron

Boron additions were made using a nickel-boron master alloy purchased from the Electromet Division of the Union Carbide Corporation. It had the following analysis, in weight percent:

| Ni | B | Al | Fe | C | Si |
|---|---|---|---|---|---|
| 77.25 | 16.38 | 0.11 | 2.98 | 0.44 | 1.01 |

### Zirconium

Zirconium additions were made with Reactor Grade chunks purchased from the Mallory Sharon Metals Company. The following impurity content was reported, in weight percent:

| C | Si | Mn | Pb | Fe |
|---|---|---|---|---|
| 0.025 | 0.006 | 0.002 | 0.002 | 0.0309 |

### Carbon

Spectrographically-pure graphite rods were turned on a lathe to produce coarse powder which was used for carbon additions.

### Lead

Lead additions were made using laboratory grade L. P. lead metal shot.

### Crucibles

On advice from a representative of the International Nickel Company, Inc. all melting was carried out in "Norton Alundum A 99% fused alumina crucibles" with an I. D. of 3½-in. and a height of 7-in. A typical analysis of this crucible material is as follows, in weight percent:

| $Al_2O_3$ | $SiO_2$ | $Fe_2O_3$ | $Na_2O$ | Other |
|---|---|---|---|---|
| 99.00 | 0.58 | 0.11 | 0.17 | balance |

A personal communication from the Norton Company stated that this grade of crucible material is spectrographically free from boron or boron compounds.

## Melting Procedure

All melting was done in a vacuum-induction melting furnace designed and built at the University of Michigan. This furnace has a 30 cubic foot cylindrical tank and a 6-in. booster-diffusion pump and mechanical fore pump combination capable of maintaining pressures of less than 10 microns during the melting of the usual twelve-pound charges.

The following melting procedure was established to provide reproducible results:

1) All chromium and carbon and as much nickel as possible were charged into the crucible. The remaining additions were placed in the charging buckets in the vacuum tank. The boron and zirconium were wrapped in aluminum foil to prevent losses during addition.

2) The tank was evacuated to a maximum pressure of two microns with a maximum apparent leak-up rate of four microns-per-minute.

3) The power was then turned on and the complete melting of the initial charge and remainder of the nickel was completed in about 45 minutes. Shorter melting times were not possible due to the lack of power handling capacity of the furnace. Generally, after the melt surface finally became visible, little if any carbon boil was observed; most of the deoxidation apparently took place during meltdown.

4) The titanium and aluminum were added and the melt superheated 200°F as measured by an optical pyrometer.

5) One half of the heat was poured.

6) Boron and zirconium additions were made, superheat re-extablished, and the second half of the heat was poured. On the basis of past experience, these additions were made assuming 100 percent recovery.

7) The total elapsed time from the start of melting was about one hour. The molds used were 4¼-in. sections of 2-in. I.D. steel pipe. A 2-in. high hot-top made from insulating brick was mounted on each mold.

When lead was to be added to the heat, the above procedure was modified as follows:

1) After the titanium and aluminum additions were made, argon was introduced into the tank and the pressure allowed to rise to one-half an atmosphere. Previous work[9] demonstrated that the addition of argon after vacuum melting did not influence properties.

2) The lead, which had been peened into holes in the nickel pellets, was then added to the melt. The melt was kept as cool as possible to prevent excessive vaporization of the lead before it dissolved. This was

accomplished by freezing the melt surface during the pressure build-up, remelting after reaching the desired one-half atmosphere of argon pressure, and then adding the leaded pellets.

3) A superheat of about 100°F was obtained and the melting completed as in steps (5) through (7) above. A 100°F superheat was used after adding lead instead of the usual 200°F to prevent excessive losses.

## Lead Distillation Study

The techniques used to investigate the effectiveness of vacuum-induction melting for lead removal included the following:

1) Two heats of the 20Cr-3.8Ti-1.2Al alloy with intentional 100 ppm lead additions under argon, held and successively sampled at varied times under vacuum, were made to define the rate of lead distillation. Five small samples were taken from one heat for chemical analysis. Four small chemical analysis samples were taken from the other heat and a fifth sample taken from bar-stock obtained by rolling a full-sized ingot poured from the material remaining.

2) Two heats of the 20Cr-2.5Ti-0.8Al alloy, with intentional 100 ppm lead additions under argon and held respectively for 10 and 60 minutes under vacuum, were made to define the effect of lead removal on creep-rupture properties. Two normal-size ingots were poured from each heat with and without boron and zirconium additions. These ingots were rolled to bar-stock and creep-rupture tested.

3) Three heats of the 20Cr-3.8Ti-1.2Al alloy, with intentional 100 ppm lead additions under argon and held respectively for 10, 30, and 60 minutes under vacuum, were made to define the effect of lead removal on creep-rupture properties. Two normal-size ingots were poured from each heat with and without boron and zirconium additions. The ingot made in step (1) was also included here as being identical to the first half of the heat held 30 minutes under vacuum. These ingots were rolled to bar-stock and creep-rupture tested.

The variables in the vacuum-induction refining process that were felt to have a significant influence on the rate of lead removal were the rate or violence of induction stirring, the percentage of total holding time under vacuum that induction stirring took place, and the degree of superheat. To keep the investigation as simple as possible, it was decided to perform all vacuum distillation studies at a superheat of 100°F as maintained by the minimum amount of power possible from the equipment. This kept the rate of induction stirring about constant and allowed the power to be on from 80 to 100-percent of the holding time.

## Hot Working Procedure

The ingots were hot rolled directly to bar-stock. The practice used for most of the ingots consisted of soaking for two hours at the rolling temperature, 2150°F, and rolling to ½-in. square bar-stock. This required 22 passes with a 10 minute reheat between each pass to maintain the temperature at 2150°F. The reduction-per-pass for the first 12 passes was about 10 percent and about 15 percent for the last 10 passes. The last pass was followed by air cooling.

A deviation from this practice arose when excessive cracking was noted. The ingot from Heat 1261A was stopped after 13 passes to grind off the cracks, reheated to 2000°F and given five passes which produced 0.65-in. square stock. The ingots from Heats 1321A and 1321C were stopped at 0.65-in. square stock after 18 passes to prevent excessive cracking that would give difficulty in obtaining sound creep-rupture specimens. The ingots from Heats 1287A and 1287B (1000 ppm lead added) cracked so badly after five passes that they were stopped at that point.

## Chemical Analysis

Sampling procedure for chemical analysis consisted of taking a 1-in. section from the center of each piece of bar-stock and machining chips from that section. This provided a 1-in. by ½-in. sample for spectrographic analysis and sufficient chips for wet analyses. Additional samples were taken from each end of the bars from Heats 1257A and 1257B to check over-all homogeniety. All analyses were made at the Research Laboratory of the International Nickel Company, Inc.

## Creep-Rupture Testing

Specimens having a gage section 1-in. long and 0.250-in. in diameter were machined from the heat treated bar-stock. The specimens were then placed in creep-rupture furnaces already at temperature and allowed a maximum of four hours to reach the required temperature and temperature distribution. Variation in temperature along the gage lengths of the specimens was held to within $\pm 3°F$ by furnace adjustments and the nominal test temperature was held to within $\pm 3°F$ by automatic controllers. The load was applied within the necessary four hours either directly or through a lever system depending on the magnitude of the load. Rupture time was automatically measured with a timer. Creep measurements were made during the tests using an optical extensometer system. These creep measurements are available as a matter of record, but they are not used in this report.

## Metallography

Metallographic examination of the materials in this investigation has been confined mostly to optical techniques. Several pallodium-shadowed collodion replicas of high lead content samples were examined in an electron microscope with negative results.

In all cases specimens were mechanically polished and electrolytically etched before examination. The etchant used was a 12 parts $H_3PO_4$ – 47 parts $H_2SO_4$ – 41 parts $HNO_3$ mixture developed at the University of Michigan for nickel-base alloys of the type in question. It tends to stain alloys containing large amounts of $Ni_3(Al, Ti) - \gamma'$, but delineates the overall structure well.

# RESULTS

## Chemical Composition

Chemical analyses of the heats of the 20Cr-2.5Ti-0.8Al alloy and the 20Cr-3.8Ti-1.2Al alloy are presented in Tables 1 and 2 respectively. All values were determined by the Research Laboratory of the International Nickel Company, Inc.

Carbon, titanium, and aluminum contents varied between heats of a given alloy. In several instances boron was reported to be present in halves of heats to which none was added. There were also several cases where considerably more than 100-percent recovery of intentional additions of boron and zirconium was reported.

Lead analyses of early heats having intentional lead additions indicated about 100-percent recovery (Heats 1257 and 1261) and a 10 ppm lower limit of analysis (Heats 1279 and 1280). Analyses of later heats indicated about a 60-percent recovery of lead (Heat 1321) and a lower limit of analysis of about 1 ppm (Heats 1322 and 1323). In view of these findings, samples of early heats of questionable lead content were re-submitted for analysis. These later results are presented in separate columns in Tables 1 and 2 and are used throughout the remainder of the report in preference to the earlier results. Where re-analyses for lead were not obtained, the original values are considered sufficiently accurate.

To check for possible segregation in the rolled bar-stock, separate samples from each end and the center of the bars from Heats 1257A and 1257B were submitted for analysis. These samples were coded T, M, and B corresponding to the top, middle and bottom of the original ingot. The results of the analyses (Table 1) showed no indication of segregation from end to end in the bar-stock.

The heats held in vacuum and periodically sampled to define lead removal rates (Heats 1304 and 1322) were analyzed completely, as shown in Table 2. These results indicated that holding for up to 30 minutes in vacuum did not significantly change the base composition of the alloys; only lead content changed.

Possible effects of variations in chemical composition will be discussed in following sections.

## The Effect of Lead on Creep-Rupture Properties

The creep-rupture test data obtained for the 20Cr-2.5Ti-0.8Al alloy at 1200° and 1500°F are presented in Table 3 and those obtained for the 20Cr-3.8Ti-1.2Al alloy are presented in Table 5. Figures 1, 2, 5, 6, and 7 show the stress-rupture time curves based on these data. The 100-hour rupture strengths derived from the stress-rupture time curves are summarized in Tables 4 and 6.

Generally, reduction-of-area measurements gave the most uniform results as a measurement of ductility in rupture tests. The varying degree of cracking adjacent to the fractures together with the difficulties encountered in matching fractures to measure elongation introduced a great deal of uncertainty into elongation values. For these reasons, and since reduction-of-area values generally showed no consistant change with time for rupture, the effect of lead on ductility in creep-rupture tests has been expressed in Tables 4 and 6 in terms of the average reduction-of-area for all tests conducted on a heat at a given temperature.

The influence of lead on the 100-hour rupture strength and average reduction-of-area of both alloys is summarized in Figures 3, 4, 8, and 9 by plotting each property as a function of analyzed lead content. Where analyzed lead contents were reported only as "less than 10 ppm", the points were plotted at 10 ppm lead with arrows pointing to a lower lead content. Associated curves were extrapolated below 10 ppm lead with some uncertainty due to the range in properties existing in most cases at that level of lead.

### 20Cr-2.5Ti-0.8Al Alloy with No Boron and Zirconium Added

As shown in Figure 1, the level of the stress-rupture time curves at 1200° and 1500°F decreased with increasing lead content. At 1500°F the stress-rupture time curves also became steeper with increasing lead. This change in slope at 1500°F indicated that lead was more detrimental to rupture strength the lower the stress and the longer the rupture time considered.

The influence of lead on 100-hour rupture strength is summarized by Figure 3. Generally, about 10 ppm lead was required at 1200°F and 20 ppm lead at 1500°F to noticeably reduce rupture strength below the level of the base heats. The heat containing 110 ppm lead (Heat 1257A) was weaker than the two heats containing about 35 ppm lead (1301A and 1303A) at either test temperature. A lead recovery of greater than 100-percent in Heat 1257A was inconsistent with recoveries in other heats and a somewhat lower lead content would have made the rupture strength data more consistent.

Holding a heat under vacuum for 60 minutes to distill off lead (Heat 1279A-100 ppm Pb added) resulted in the complete recovery of strength at 1200°F and only partial recovery at 1500°F, as shown in Figure 1. Complete lead removal to a level comparable to that of the base heats was obtained. Holding a base heat (Heat 1259A – no lead added) for 60 minutes under vacuum resulted in no change of strength at 1200°F and increased strength at 1500°F. There was an unfortunate variation of carbon content in these heats. If this was responsible for the strength variation at 1500°F, low carbon was beneficial (Heat 1259A) and high carbon was detrimental (Heat 1279A).

Ductility in creep-rupture tests was reduced by lead as indicated by average reduction-of-area values in Table 4 and Figure 4. About 35 ppm lead was required to produce a significant effect at 1200°F while only about 10 ppm was necessary at 1500°F. There was also a trend for the individual reduction-of-area values of the higher lead heats (Table 3) to decrease with increasing rupture time at 1500°F. Note that this occurred where stress-rupture time curves tended to change slope and "fan out". For a given lead content, heats held under vacuum to distill off lead had higher ductility than those not exposed to vacuum after a lead addition. The average reduction-of-area data then suggest an effect due to holding under vacuum over and above lead removal; but it is difficult to see why holding 10 minutes (Heat 1303A – 100 ppm lead added) should be beneficial.

Difficulties were experienced due to erratic test results within a given heat, uncertainties of chemical analyses, and real variations in chemical composition. It was most significant that the general trends of the data indicating a loss in rupture strength and ductility with increasing lead were not greatly influenced by these difficulties.

### 20Cr-2.5Ti-0.8Al Alloy with 0.0035% Boron and 0.045% Zirconium Added

Stress-rupture time curves and 100-hour rupture strength were only slightly influenced by lead additions up to 100 ppm, as shown in Figures 2 and 3. There was less effect from lead in the presence of boron and zirconium than for the same alloy with no boron and zirconium added. Also, stress-rupture time curves at 1500°F did not exhibit an increase in slope with increasing lead, as observed in boron and zirconium-free heats (compare Figs. 1 and 2). About 30 ppm lead was necessary at 1200°F and 40 ppm lead at 1500°F to reduce 100-hour rupture strength below that of the base material containing less than 10 ppm lead (Fig. 3).

Creep-rupture test ductility was reduced by lead, as shown in Table 4 and Figure 4. At 1200°F, as little as 10 ppm lead produced a significant effect and the relative loss of ductility was about the same for a given lead content as that experienced in the boron and zirconium-free heats; although the actual values were higher. At 1500°F, possibly less than 10 ppm lead reduced ductility and increasing lead had a relatively greater influence than on the same alloy without boron and zirconium; however, the actual average reduction-of-area values at 1500°F (Table 3) did not show a trend of being reduced at longer rupture times as did the boron and zirconium-free material and neither did the stress-rupture time curves "fan out" (Fig. 2).

With the exception of the tendency for heats held in vacuum in order to distill off lead to have higher ductility in creep-rupture tests, the properties generally correlated with lead contents whether obtained by direct addition or by partial removal upon holding under vacuum.

Limited data again suggests some benefit from holding under vacuum over and above the removal of lead.

## 20Cr-3.8Ti-1.2Al Alloy with No Boron and Zirconium Added

Four base alloy heats were tested at 1200° and 1500°F. These heats had differences in composition and the test results showed significant scatter within a given heat and between heats. For these reasons, and since the amount of data that could be intelligibly presented on a single set of stress-rupture time curves was limited, it appeared best to define the stress-rupture time properties of the base alloys as bands, as shown in Figure 5. The only correlation of strength variations with compositional differences was the tendency for the higher carbon heats to be somewhat stronger at 1200°F.

The stress-rupture time relationships in Figure 6 show a marked detrimental effect of lead. The bands developed for the base alloy in Figure 5 are also reproduced in Figure 6. Heats containing more than 30 ppm lead had very erratic rupture properties at 1200°F, with higher stresses producing what appeared to be premature failure. Rupture data was much more consistant at 1500°F, although erratic results were still a problem at higher lead contents. Generally, increasing amounts of lead lowered the levels and increased the slopes of rupture curves at 1500°F.

The 100-hour rupture strengths (Table 6) estimated from Figures 5 and 6 were plotted as a function of lead content in Figure 8. This figure shows that increasing lead from 10 to 65 ppm caused a continual and large loss of rupture strength at both 1200° and 1500°F.

Heats held in vacuum to distill off lead had about the same rupture strengths at 1200°F as those with a similar lead content obtained by direct addition (Figs. 6 and 8). At 1500°F, strengths were similar only for the higher lead heats where holding time was short (Heat 1302A – held 10 min. in vacuum); while the heats held for 30 to 60 minutes under vacuum for 60 minutes did not improve rupture strength at 1500°F as it did for Heat 1259A (no lead added) of the 20Cr-2.5Ti-0.8Al alloy (Figs. 1 and 5).

Average reduction-of-area values were generally low for the base alloy and lead additions reduced ductility in creep-rupture tests still further (Table 6 and Fig. 9). At 1200°F as little as 10 ppm lead reduced ductility and at 1500°F a lead content of more than 10 ppm resulted in less than 1-percent reduction-of-area. For a given lead content, heats held in vacuum to distill off lead had ductility values of about the same magnitude as those obtained for heats not held under vacuum. Note also in Table 6 that the low lead heats having the highest ductility at 1500°F also had high carbon contents (Heat 1314A – 0.10% C, Heat 1280A – 0.09% C).

Split Heat 1321 was made to obtain information on the effect of carbon because of the carbon content variations experienced. After a direct addition of 100 ppm lead under argon, Heat 1321A was poured with a 0.07-percent final carbon content and Heat 1321C with a 0.11-percent final carbon content. Boron and zirconium were not added to this split heat so the two halves are comparable to Heat 1261A. The pertinent results were:

| | | | 100-Hour Rupture Strength (psi) | | Average Reduction of Area (%) | |
|---|---|---|---|---|---|---|
| Heat | C (%) | Pb (ppm) | 1200°F | 1500°F | 1200°F | 1500°F |
| 1261A | 0.04 | 60 | 41,000 | 8,900 | <1 | <1 |
| 1321A | 0.07 | 64 | 41,000 | 11,100 | 1 | <1 |
| 1321C | 0.11 | 62 | 53,900 | 10,600 | 3 | 1 |

These data suggest that increasing carbon in the presence of lead increased rupture strength and possibly ductility at both 1200° and 1500°F. The rupture curves at 1200° and 1500°F for Heat 1321C (0.11% C) also had lesser slopes and less erratic data scatter than the lower carbon heats with high lead contents (Fig. 6).

### 20Cr-3.8Ti-1.2Al Alloy with 0.0035% Boron and 0.045% Zirconium Added

When 0.0035-percent boron and 0.45-percent zirconium were added to the base alloy, creep-rupture properties were greatly improved and the scatter in the stress-rupture time properties was reduced in comparison to the alloy without the additions (Fig. 5). Holding the base alloy in vacuum for an hour did not appreciably influence the rupture strength.

The effects of lead on the stress-rupture time curves and 100-hour rupture strengths as derived from these curves, are shown in Figures 7 and 8 respectively. At 1200°F, rupture data for the heats containing up to 40 ppm lead fell within the band developed for the base alloy (Fig.7); while Heat 1261B, containing 54 ppm lead, showed a very large decrease in strength. The relationship for 100-hour rupture strength (Fig. 8) indicated a slight loss in strength within the base alloy band between 20 and 40 ppm lead followed by a very large decrease at 54 ppm.

The stress-rupture time curves at 1500°F (Fig. 7) showed significant reductions in strength from lead contents as low as 10 ppm. This was not as evident in the 100-hour rupture strengths (Table 6 and Fig. 8) as it was in the stress-rupture time curves because of the changing slope of the curves at about 40,000 psi or between 10 and 50 hours. These changes in slope indicated that lead was more detrimental the lower the stress and the longer the time for rupture. Again, Heat 1261B with 54 ppm lead showed a marked loss in strength, as shown in Figures 7 and 8.

Rupture strengths were the same for a given lead content whether the heat was held under vacuum to distill of lead or had a direct addition of lead.

Average reduction-of-area values of rupture tests (Table 6 and Fig. 9) were increased by boron and zirconium at any level of lead when compared to the same alloy without boron and zirconium. At 1200°F, the loss of ductility from lead additions was proportionally about the same with or without boron and zirconium (Fig. 9); but at 1500°F, lead had a proportionally greater effect when boron and zirconium were present. Individual reduction-of-area values for the higher lead heats (Table 5) showed a pronounced tendency to decrease with increasing time for rupture at 1500°F and the stress-rupture time curves (Fig. 7) showed breaks at intermediate rupture times and "fanned" out markedly. Holding heats in vacuum to remove lead did not result in improved rupture ductility over that experienced in normally melted heats having similar lead contents.

## Vacuum Removal of Lead

The investigation provided data on the rate at which lead was removed from molten heats held in a vacuum of about one micron. Two heats were successively samples at increasing time. Since only a limited number of samples were possible, they were taken at holding times of 30 minutes or less to accurately define the portion of the distillation process where lead analyses would be most reliable. Plotting the logarithm of the analyzed lead content versus holding time developed the curve shown in Figure 11.

Two heats were held for 10 minutes in vacuum and one heat held for 30 minutes; all having an intentional additions of 100 ppm lead. Four heats were held for 60 minutes in vacuum; two having no intentional lead additions and two having intentional additions of 100 ppm lead. The analyzed lead contents of these heats, averaged between two ingots because the heats were split, as well as the analyzed lead contents of two heats poured immediately after melting with no lead addition, were also plotted on Figure 11. These data agreed with the curve developed for the successively sampled heats for shorter holding times, but showed a marked deviation from a straight line extrapolation of the curve at 60 minutes. Also, the data available for heats with no lead additions indicate that holding for 60 minutes did not appreciably change the lead content at about the 4 ppm level.

A possible explaination for the change in lead removal rate apparent in Figure 11 for the portion of the curve beyond 30 minutes that was estimated to deviate from a straight line, other than the obvious fact that the limit of chemical analysis had been approached, was that the partial pressure of lead in the melt was approaching the atmosphere pressure above the melt. Raoult's Law for an ideal solution of lead in a liquid is

$$p = Np^{\circ}$$

where $p^{\circ}$ is the vapor pressure of pure lead, $N$ the atom fraction of lead in the alloy, and $p$ the partial pressure of lead in the alloy. At about 2700°F and 5 ppm lead:

$$p^\circ = 4 \times 10^{5} \text{ micron} \qquad N = 1.35 \times 10^{-6}$$

and

$$p = (1.35 \times 10^{-6})\,(4 \times 10^{5}) = 0.54 \text{ micron}$$

So that even with the probable deviations from ideality and the inaccuracy of the vapor pressure data, it is likely that the rate of lead distillation would be reduced at lead contents between 1 and 10 ppm for a tank pressure of one micron.

The equation defining the straightline segment of the curve in Figure 11 is:

$$\log C = -0.0376 + \log C_o$$

where C is the lead content in ppm, t the time in minutes, and $C_o$ the initial lead content in ppm. The figure also shows a curve developed by the Research Laboratories of the International Nickel Company, Inc. for the vacuum distillation of lead from nickel. This curve was nearly parallel to the curve for nickel-chromium alloys developed in the present investigation and is defined by the equation:

$$\log C = -0.0392t + \log C_o$$

The agreement between the curves for nickel-chromium alloys and nickel, determined in two separate laboratories, is remarkable. The curves indicate that holding molten heats of nickel of nickel-chromium alloys initially containing 100 ppm lead in a vacuum of one micron for 30 minutes would reduce the lead content to about 10 ppm. However, the apparent deviation in the curve developed by the authors indicates a rapid decrease in removal rate below 10 ppm lead, assuming the lead analyses are correct.

The agreement between the two laboratories for the rate of lead removal from nickel and nickel-chromium alloys tends to suggest that it is independent of the apparatus used. Since the details of the International Nickel Company's procedure are not yet available, no conclusions can be drawn about this. It should be pointed out, though, that melt surface-to-volume ratio, degree of vacuum possible, and the severity of induction stirring should affect the rate of lead removal.

## Influence of Lead on Hot-Working Characteristics

Behavior of the materials during rolling was closely observed and the only effect noticed was corner cracking. All ingots apparently rolled the same as a function of lead content except for the degree of corner cracking. Additions of lead increased corner cracking in the manner detailed in the following sections.

### 20Cr-2.5Ti-0.8Al Alloy

The addition of lead did not cause significant corner cracking of the 20Cr-2.5Ti-0.8Al alloy until the analyzed lead content was about 35 ppm (Heat 1301 with 60 ppm lead added), as shown in Figure 12. The severity of the cracking increased when the lead was increased to 100 ppm (Heat 1257).

The corner cracking was not as severe in vacuum distilled Heat 1303 having 34 to 45 ppm lead, as in Heat 1301 with about the same amount of lead by direct addition. Heat 1280 (7 ppm lead), held for an hour under vacuum after an addition of 100 ppm of lead, was not subject to cracking. However, base Heat 1259, held for an hour in vacuum with no lead addition, was initially very prone to cracking in the "A" half which had no boron or zirconium added. The "B" half of Heat 1259 with boron and zirconium added was normal.

Most of the data suggest improved resistance to cracking upon lead removal by vacuum refining. Boron and zirconium additions did not seem to change the corner cracking characteristics.

### 20Cr-3.8Ti-1.2Al Alloy

The base heats were rolled to ½-in. square bars with very little or no corner cracking whether or not boron and zirconium were added, as shown in Figure 13. Holding Heat 1260 under vacuum for an hour did not change the cracking tendency.

No cracking was observed in heats with intentional lead additions until reaching a lead content of 40 ppm (Heat 1286), where a small amount of corner cracking occurred (Fig. 14). The bar from Heat 1261A, with 54 ppm lead (100 ppm added) and no boron or zirconium, cracked severly (Fig. 15). Grinding off the cracks after 13 passes and reducing the rolling temperature to 2000°F still resulted in severe cracking. The bar from Heat 1261B, with 60 ppm lead and boron and zirconium added, rolled directly to ½-in. square bar-stock with only slight cracking. Heats 1321A and 1321C, with 60 and 64 ppm lead (100 ppm Pb added) and no boron and zirconium, also cracked severely (Fig. 15). These halves of a split heat were made with two carbon levels and the variation in carbon content had no observable influence.

A split heat with a 1000 ppm lead addition (Heat 1287A and 1287B), used only for hot-workability evaluation, cracked so severely after five passes that further rolling was stopped, as shown in Figure 16. The addition of boron and zirconium had no observable influence on this very high lead material.

Holding under vacuum for 10 minutes reduced the lead in Heat 1302 (100 ppm added) to 35-42 ppm. The cracking severity noted for this heat was the same as noted for Heat 1286 having about the same analyzed lead content by a direct addition (compare Figs. 14 and 17). Holding in vacuum for 30 or 60 minutes reduced lead below 10 ppm and eliminated cracking (Fig. 17).

The data indicated that about 40 ppm lead was necessary in the 20Cr-3.8Ti-1.2Al alloy to induce corner cracking during hot rolling. There seemed to be no effect from boron and zirconium additions except in Heat 1261 (54 and 60 ppm lead). In this case, boron and zirconium seemed to reduce the corner cracking severity. Cracking appeared to be about the same for a given lead content whether obtained by direct addition or by partial removal from holding a melt in vacuum.

## Microstructural Investigation

Microstructures of the materials included in this investigation were examined optically to ascertain how and where lead was acting to reduce properties. No evidence of lead or lead compounds was found in any sample of either alloy and there were no other striking structural differences that could be attributed directly to the presence of lead. At this time only results giving examples of general microstructural features are presented.

The two-stage heat treatment used throughout this investigation, consisting of an 8-hr. solution anneal at 1975°F and a 16-hr. age at 1300°F followed by air cooling to room temperature, resulted in the structures shown in Figures 18, 19, 20, and 21 for the 20Cr-2.5Ti-0.8Al and 20Cr-3.8Ti-1.2Al alloys. The as-heat treated microstructures were generally the same for both alloys, although the 20Cr-3.8Ti-1.2Al alloy tended to react more within the grains to the etchant used due to its higher $\gamma'$ content. Grain sizes were mixed, with the higher carbon heats tending to have a finer grain size and the lower carbon heats a coarser grain size (Figs. 18 and 20). Carbides, probably $Cr_{23}C_6$ and/or $Cr_7C_3$, were apparent in the grain boundaries and within the grains (Figs. 19 and 21) and primary Ti(C, N) particles were distributed randomly throughout the structure. The chromium carbides tended to be somewhat segregated, resulting in mildly banded structures (Figs. 18 and 20). Consequently, high magnification photomicrographs (Figs. 18 and 21) are difficult to compare with one another. The $Ni_3$ (Al, Ti) — $\gamma'$ intragranular precipitate was extremely fine due to the 1300°F aging temperature and could not be resolved optically.

No influence was observed of boron and zirconium additions on grain size (Figs. 18 and 20) or microconstituents (Figs. 19 and 21) in either alloy in the as-heat condition.

Longitudinally sectioned rupture specimens were also examined. The structural changes that occurred during creep were those to be expected in the type of alloy being studied. Figure 22 illustrates, for the 20Cr-3.8Ti-1.2Al alloy, the following structural changes at 1500°F:

1. The $\gamma'$ intragranular precipitate coarsened during prolonged exposure, in some cases (Fig. 22 e and f) being almost visible optically.
2. Regions along grain boundaries were depleted of $\gamma'$ leaving conspicuously white precipitate-free areas, particularly transverse to the direction of testing. This depletion increased with exposure time (Fig. 22 e and f).
3. Microcracks formed along grain boundaries and carbide particles transverse to the direction of testing (Fig. 22 a, c, and e).

The same general structural changes occurred in both alloys (compare Figs. 22 and 23) and, as would be expected, these structural changes were more extensive for the higher temperature of testing (Fig. 23) and longer exposure times (Fig. 22).

Fractures were intergranular in all cases at both test temperatures. Figure 24 illustrates the intergranular fractures and a case of severe transverse cracking adjacent to the fracture.

Presently it is thought that boron and zirconium are beneficial because they slow down the structural changes occurring during creep that lead to rupture. Lead, however, is thought not to influence the phase reactions as such, but limits them by causing early crack formation and propogation that leads to premature fracture with limited ductility. The structural observations to date have not well documented these concepts. Possibly the investigation of specimens with similar rupture times at different stresses will clarify the mechanisms, although structural studies of interrupted creep tests might be more fruitful.

# DISCUSSION

This investigation was undertaken to provide data on degree of damage to creep-rupture properties and hot-workability that could be introduced by trace amounts of lead in two nickel-chromium-titanium-aluminium alloys. The alloys were investigated without and with combined boron and zirconium additions in order to explore the relationships between the beneficial effects of these elements and the harmful effects of lead. Data were also obtained on properties after lead was removed by vacuum refining.

## Amount of Lead Required to Damage Creep-Rupture Properties

Data scatter and uncertainties of lead analyses required a minimum of 10 ppm lead to show significant reductions of rupture strength and/or ductility. However, when rupture strength and ductility were plotted versus lead content, there appeared to be damage to these properties when lead was considerably less than 10 ppm. In fact the curves suggested that any lead at all started to reduce properties with the degree of damage increasing with lead content. These results agree with those of Wood and Cook[6] which showed a like sensitivity of two wrought and one cast nickel-base heat-resistant alloys to small amounts of lead. Data now available do not define a limit below which lead will not damage creep-rupture properties.

The very practical problem does then remain of how much lead can be tolerated in commercial engineering alloys. The data from this investigation demonstrated that the degree of damage to properties from a given amount of lead varied between alloys and whether or not boron and zirconium were present. The damage also varied with test temperature, test time period or stress, and whether rupture strength or rupture ductility was used to measure the effects. Therefore the maximum lead content tolerable in an alloy depends both on the response of the alloy to lead and the requirements of the application. If one were to require of the alloys included in this investigation that lead contents be low enough to not cause any reduction of creep-rupture properties in the temperature range of 1200°F to 1500°F, a figure of less than 10 ppm lead would have to be specified.

## Role of Boron and Zirconium in Creep-Rupture Properties

The presence of boron and zirconium apparently changed the "environment", or more properly, produced a "new" alloy to which lead was added rather than interacting with lead. More lead was necessary to cause damage in the 20Cr-2.5Ti-0.8Al alloy when it contained boron and zirconium than when it did not. On the other hand, the main effect of boron and zirconium on the 20Cr-3.8Ti-1.2Al alloy was to raise the level of strength with the damage from lead simply occurring at this higher level. There was also an added effect in both alloys in that boron and zirconium retarded lead damage to creep-rupture properties at 1500°F. Attempts to place the combined effects of lead and boron and zirconium on an interaction basis were generally unsuccessful.

## Mechanism of Damage from Lead

Direct evidence of lead or effects of lead was not found during microstructural studies. The large influence of trace amounts of lead, like boron and zirconium effects, must presently be explained indirectly with circumstantial evidence.

Lead presumably segregates to grain boundaries and damages properties by melting or inducing effects similar to melting. It is generally accepted that boron and zirconium also segregate to grain boundaries and there improve properties. Therefore, boron and zirconium could be considered to have effects opposite to those of lead. The data provide indirect evidence suggesting this concept. Extensive consideration of the data suggests that boron and zirconium change the conditions in the grain boundaries rather than interacting directly with lead.

The compositional effects can be understood if two assumptions are made:

1) Due to a greater amount of $\gamma'$-intragranular precipitate, the 20Cr-3.8Ti-1.2Al alloy had grains considerably stronger than the grain boundaries and grain boundary conditions controlled properties.

2) Due to the lesser amount of the $\gamma'$-intragranular precipitate, the relative strengths of the grains and grain boundaries in the 20Cr-2.5Ti-0.8Al alloy were nearly balanced and grain boundary conditions did not control

strength to as great a degree as in the 20Cr-3.8Ti-1.2Al alloy. From these assumptions and the postulated grain boundary segregation, lead and boron and zirconium should have had the most influence, good or bad, in the 20Cr-3.8Ti-1.2Al alloy. This was observed to be the case. Furthermore, at higher temperatures and longer exposure times where creep is more dependent on grain boundary properties these elements should have had greater effects, as was again observed. These concepts also seem to account for the "fanning out" of the stress-rupture time curves at 1500°F and the retardation of lead damage by boron and zirconium.

As can be seen from the above discussion, the authors have not stated a mechanism for lead damage, but have merely indicated where the mechanism takes place. As with the elusive boron and zirconium mechanism, much quantitative metallographic work appears to be necessary before the actual mechanism of lead damage will be delineated.

## Evaluation on a Time for Rupture Basis

Consideration was given to evaluating the data by time for rupture at specific stresses; a procedure commonly used in investigations of this type. At 1200°F a comparison of this kind could be made for the 20Cr-2.5Ti-0.8Al alloy with and without boron and zirconium; but for the 20Cr-3.8Ti-1.2Al alloy at 1200°F, where lead effects were far greater, any attempts at evaluation at a common stress were rather meaningless or impossible with the available data. This resulted from the facts that differences in rupture time at a specific stress were very large and the lowest stress used for the stronger heats was higher than the highest stress used for the weaker heats. The use of different stresses in this situation to allow comparison on a time for rupture basis had two short comings; 1) It was difficult to properly understand the effect of lead in terms of the presence or absence of boron and zirconium and 2) The stress-rupture time curves had little slope.

For the tests at 1500°F it was posible to use rupture time at common stresses. Figure 17 shows the effect of lead on rupture time at 20,000 psi for the 20Cr-3.8Ti-1.2Al alloy. This figure indicates that lead was very damaging in the material with boron and zirconium added and comparatively only slightly damaging when boron and zirconium were not added. This is an erroneous indication as lead actually reduced rupture strength about the same in both materials (Figs. 6, 7 and 8). The variable causing Figure 17 to be misleading is the difference in level of strength for the alloy with and without boron and zirconium. The rupture times at 20,000 psi were quite short for the alloy without boron and zirconium and lead could only reduce rupture time a small amount. The rupture times were very long for the low lead heats of the alloy with boron and zirconium and the reduction in life from lead was very marked. Yet when compared on the basis of the rupture strengths, the effect of lead was nearly the same in both cases.

It is believed that actual stress-rupture time curves and rupture strengths are better than rupture time at a specific stress as evaluations of an effect such as that experienced from lead. It is necessary to use considerable care to avoid being misled in evaluation of the data when rupture times at a stress inducing rupture in a convenient range of time periods are used to compare materials.

### Effect of Lead on Hot-Workability

The data on corner cracking during hot rolling showed that 35 to 40 ppm lead was necessary to induce cracking in the two alloys studied. The 20Cr-3.8Ti-1.2Al alloy cracked more for a given lead content that the 20Cr-2.5Ti-0.8Al alloy. It is to be emphasized, however, that the severity of the hot working procedure used in this investigation relative to production conditions is not known. The limitations of temperature and the amount of reduction between reheats included in the investigation makes it important to recognize that it has not been proven that 35 to 40 ppm lead can be tolerated under production conditions. It appears quite certain that lead contents would have to be kept considerably lower for most production operations.

### Removing Lead by Vacuum Refining

The experiments involving vacuum treatment to remove lead prove that vacuum melting can definitely remove lead from a melt by vaporization and thereby improve properties. Most of the data indicated that properties were the same for a given lead content whether the lead was added directly or resulted from partial removal by holding in vacuum, although there were several anomalies as mentioned previously. These exceptions are not presently understood. The improvement in properties from holding in vacuum after lead had been added was very large compared with any changes from holding heats under vacuum without lead additions. The raw materials may have been slightly contaminated with subversive elements which vaporized during prolonged holding in vacuum. The possibility thus exists that in production melting quite widely different results could be obtained depending on the contaminants and melting conditions.

The agreement between two laboratories for the rate of removal of lead from nickel and nickel-chromium-titanium-aluminium alloys during vacuum melting was rather remarkable and probably an unusual coincidence. Certainly considerably more research would be necessary before it could be concluded that melting conditions other than the degree of vacuum do not greatly influence the rate of removal. This is particularly so because it is contrary to present theories of vacuum refining.

## Creep-Rupture Property Variations Between Heats

The heat-to-heat variations of creep-rupture properties limited the preciseness of lead damage measurements. This, however, in no way detracts from the general demonstration of damage from lead. It is only necessary to recognize that a range in properties was present due to other reasons than variations in lead.

The early analyses for lead had a lower limit of 10 ppm. This improved during the investigation. It would have helped , however, to have been more certain of the lead content of the heats containing less than 10 ppm lead when attempting to understand the reasons for the variations in properties between heats.

Early in the investigation boron was reported in the ingots to which none had been added and where boron was added, analytical results indicated greater than 100 percent recovery. An extensive check made for boron in the raw materials or crucibles showed no source of boron. During the investigation the apparent "blank" for boron in the heats disappeared and the analyses indicated about 100 percent recovery. All of the data have been treated as if boron was very low when none was added and as if there had been 100 percent recovery when it was added. There was no indication that analyized boron contents bore any relation to the heat-to-heat variations in properties.

Variations in carbon, titanium and aluminium contents between heats were larger than was desirable. Controlling carbon in the heats held in vacuum was particularly difficult. As was shown in the previous report on sulphur, carbon did have a marked ability to improve ductility in the rupture tests.

There were problems with scatter of test results for individual heats. This became worse at high lead contents, but did exist even in the base heats. The reasons for the scatter are not known. Procedures in processing the materials were kept highly uniform. Lead undoubtedly contributed to erratic test results as a characteristic effect even though the checks for lead variations did not show segregation. It is suggested that the temperature of solution treatment combined with the conditions of hot working may have encompassed conditions where properties change quite rapidly with treatment. The mixed coarse and fine grains suggest that this could be the case. It is considered, however, that changing the heat treatment and prior history could not have changed the general effects of lead. The lead would have affected properties to about the same degree, but at different strength levels as previously discussed.

# CONCLUSIONS

The results from this investigation of the effects of lead on the creep-rupture properties and hot-workability of two nickel-chromium-titanium-aluminium alloys lead to the following conclusions:

1) Creep-rupture properties of the two alloys without or with boron and zirconium were damaged by lead with the damage increasing with the amount of lead. Under the conditions of the investigation, 10 ppm of lead was required to show a significant reduction in rupture strength and/or ductility. Lesser amounts undoubtedly reduced properties, although this was obscured by data scatter and the uncertainties of lead analyses .

2) The degree of damage resulting from lead was variable depending on the alloy, whether or not boron and zirconium had been added, and the testing conditions (temperature, stress or time). Therefore, it is concluded than the amount of lead tolerable in commercial practice would vary considerably depending on the alloy and it's service requirements.

3) Lead was far more harmful in the 20Cr-3.8Ti-1.2Al alloy than in the 20Cr-2.5Ti-0.8Al alloy.

4) The combined additions of 0.0035-percent boron and 0.045-percent zirconium improved the creep-rupture properties of both alloys with or without lead and were far more beneficial in the 20Cr-3.8Ti-1.2Al alloy than in the 20Cr-2.5Ti-0.8Al alloy. Boron and zirconium did not eliminate lead damage. As far as could be judged, the effects of boron and zirconium on either alloy were separate and distinct from the lead effects. The differences noted in lead damage with or without boron and zirconium were attributed to differences in material structures such as would exist in two uniquely different alloys.

5) The general pattern of data suggests that, for creep conditions, lead damages grain boundary properties while boron and zirconium improve grain boundary properties. The effects of lead (and boron and zirconium) thus probably vary depending on the relative properties of grains and grain boundaries at the test conditions. The fact that lead had a larger effect in the 20Cr-3.8Ti-1.2Al alloy than in the 20Cr-2.5Ti-0.8Al alloy could be explained by the greater amount of $\gamma'$ in the former strengthening the grains

more relative to the grain boundaries and thus increasing the susceptibility to damage from lead.

6) The only observed effect of lead on hot-workability was increased corner cracking during direct rolling of the small ingots when lead contents exceeded 35 ppm. The hot rolling conditions apparently represented a very gentle condition of hot working and the probability is that less lead would cause trouble in many production operations where the hot working is more severe.

7) Vacuum refining definitely removed lead. For the conditions used in this investigation, holding a melt initially containing 70 ppm lead for 30 minutes in a vacuum of one micron reduced the lead content to less than 10 ppm. The rate of removal was remarkably similar to that reported by The International Nickel Company Inc. for removal of lead from nickel.

8) Creep-rupture properties of the heats that underwent vacuum refining to remove lead were generally commensurate with their analyzed lead content. There were exceptions, however, for heats held for an hour in vacuum with or without prior lead additions. These anomalies were in some cases attributable to variations in carbon content; but there were indications that something else was happening during the refining operation.

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Table 1

## CHEMICAL COMPOSITION OF THE 20Cr-2.5Ti-0.8Al ALLOY HEATS

(B, Pb, and S contents are reported in parts per million, others in weight percent)

| Heat No. | Purpose of Heat | C | Cr | Ti | Al | B | Zr | Pb | Pb | S | Mn | Si | As | Sb | Sn | Bi | Mg | Zn | Cu | Fe |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Aim Composition | | 0.04-0.06 | 20.0 | 2.5 | 0.8 | (a) | (b) | (c) | (d) | LP | LP | LP | LP | LP | LP | LP | LP | LP | LP | LP |
| 1249A | base heat | 0.04 | 21.09 | 2.40 | 0.75 | 21 | <0.01 | T | 3 | 16 | -- | 0.021 | ND | T | VW | ND | VW | ND | VW | --- |
| 1249B | no Pb added | 0.04 | 21.00 | 2.56 | 0.73 | 42 | 0.044 | ND | 4 | 18 | -- | 0.033 | ND | T | ND | ND | VW | ND | VW | --- |
| 1259A | 1 hour vac. dist. | 0.017 | 21.5 | 2.45 | 0.92 | 15 | <0.01 | T | 4 | 11 | T | 0.037 | ND | T | ND | ND | VW | ND | VW | W-M |
| 1259B | no Pb added | 0.020 | 21.4 | 2.50 | 0.93 | 68 | 0.068 | T | --- | 11 | T | 0.038 | ND | T | ND | ND | VW | ND | VW | W-M |
| 1298A | 10 ppm Pb added | 0.036 | 20.40 | 2.56 | 0.77 | <10 | <0.01 | <10 | --- | 62 | -- | 0.010 | --- | --- | --- | --- | <0.01 | --- | --- | --- |
| 1298B | | 0.039 | 20.46 | 2.55 | 0.79 | 40 | 0.057 | <10 | --- | 61 | -- | <0.01 | --- | --- | --- | --- | <0.01 | --- | --- | --- |
| 1275A | 25 ppm Pb added | 0.04 | 20.46 | 2.57 | 0.85 | 10 | <0.01 | 21 | --- | 37 | -- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| 1275B | | 0.05 | 20.51 | 2.41 | 0.90 | 75 | 0.043 | 20 | --- | 38 | -- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| 1276A | 50 ppm Pb added | 0.05 | 20.62 | 2.56 | 0.87 | 12 | <0.01 | 27 | --- | 37 | -- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| 1276B | | 0.04 | 20.73 | 2.49 | 0.87 | 60 | 0.045 | 24 | --- | 36 | -- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| 1301A | 60 ppm Pb added | 0.04 | 20.45 | 2.60 | 0.79 | <10 | <0.01 | 36 | --- | 38 | <0.01 | <0.01 | --- | --- | --- | --- | <0.01 | --- | --- | --- |
| 1301B | | 0.04 | 20.42 | 2.55 | 0.75 | 41 | 0.060 | 34 | --- | 56 | <0.01 | <0.01 | --- | --- | --- | --- | <0.01 | --- | --- | --- |
| 1257A-T | 100 ppm Pb added | 0.05 | 20.7 | 2.61 | 0.91 | 13 | <0.01 | 110 | --- | 16 | T | 0.03 | ND | T | T | ND | VW | ND | VW | W-M |
| 1257A-M | | 0.04 | 20.6 | 2.46 | 0.91 | 17 | <0.01 | 92 | 110 | 15 | T | 0.04 | ND | T | T | ND | VW | ND | VW | W-M |
| 1257A-B | | 0.04 | 20.7 | 2.58 | 0.90 | 14 | <0.01 | 110 | --- | 13 | T | 0.04 | ND | T | T | ND | ND | ND | ND | W-M |
| 1257B-T | | 0.04 | 20.7 | 2.46 | 0.93 | 21 | <0.01 | 110 | --- | 16 | T | 0.04 | ND | T | T | ND | VW | ND | VW | W-M |
| 1257B-M | | 0.04 | 20.7 | 2.50 | 0.90 | 26 | <0.01 | 110 | 95 | 18 | T | 0.04 | ND | T | T | ND | VW | ND | VW | W-M |
| 1257B-B | | 0.04 | 20.7 | 2.67 | 0.92 | 25 | <0.01 | 95 | --- | 10 | T | 0.04 | ND | T | T | ND | VW | ND | VW | W-M |
| 1303A | 100 ppm Pb added | 0.04 | 20.41 | 2.57 | 0.77 | <10 | <0.01 | 34 | --- | 67 | <0.01 | 0.017 | --- | --- | --- | --- | <0.01 | --- | --- | --- |
| 1303B | 10 min. vac. dist. | 0.04 | 20.38 | 2.61 | 0.78 | 90 | 0.12 | 45 | --- | 54 | <0.01 | 0.022 | --- | --- | --- | --- | <0.01 | --- | --- | --- |
| 1279A | 100 ppm Pb added | 0.08 | 20.68 | 2.56 | 0.92 | <10 | <0.01 | <10 | 7 | 24 | -- | 0.034 | --- | --- | --- | --- | --- | --- | --- | --- |
| 1279B | 1 hour vac. dist. | 0.07 | 20.68 | 2.42 | 0.93 | 78 | 0.052 | <10 | 6 | 25 | -- | 0.034 | --- | --- | --- | --- | --- | --- | --- | --- |

(a) Intentional addition of 35 ppm boron to B ingot of each heat

(b) Intentional addition of 0.045% zirconium to B ingot of each heat

(c) Intentional addition of the amount of lead specified under "Purpose of Heat" column

(d) Re-analyses for lead; these values are preferred over original analyses

LP - Low as possible

M = moderate - 0.1 to 1%

W = weak -0.01 to 0.1%

VW = very weak - 0.001 to 0.01%

T = trace - 0.0001 to 0.001%

ND = not detected

Table 2

## CHEMICAL COMPOSITION OF THE 20Cr-3.8Ti-1.2Al ALLOY HEATS

(B, Pb, and S contents are reported in parts per million, others in weight percent)

| Heat No. | Purpose of Heat | C | Cr | Ti | Al | B | Zr | Pb | Pb | S | Mn | Si | As | Sb | Sn | Bi | Mg | Zn | Cu | Fe |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Aim Composition | | 0.05-0.07 | 20.0 | 3.8- | 1.2 | (a) | (b) | (c) | (d) | LP | LP | LP | LP | LP | LP | LP | LP | LP | LP | LP |
| 1247A | base heat | 0.024 | 20.94 | 3.42 | 1.12 | 17 | <0.01 | ND | --- | 35 | --- | 0.033 | ND | T | ND | ND | VW | ND | VW | --- |
| 1247B | no Pb added | 0.024 | 20.94 | 3.42 | 1.12 | 63 | 0.058 | ND | --- | 28 | --- | 0.023 | ND | T | VW | ND | VW | ND | VW | --- |
| 1290A | base heat | 0.04 | 20.29 | 3.97 | 1.29 | <10 | <0.01 | <10 | --- | 59 | <0.01 | 0.013 | ND | ND | ND | ND | <0.01 | ND | VW | W |
| 1290B | no Pb added | 0.04 | 20.29 | 4.03 | 1.30 | 75 | 0.075 | <10 | --- | 43 | <0.01 | 0.016 | ND | ND | ND | ND | <0.01 | ND | VW | W |
| 1314A | base heat | 0.10 | 19.94 | 3.75 | 0.99 | <10 | <0.01 | --- | 4 | 49 | <0.01 | 0.014 | --- | --- | --- | --- | <0.01 | --- | --- | --- |
| 1314B | no Pb added | 0.10 | 19.85 | 3.70 | 1.01 | 31 | 0.025 | --- | 4 | 89 | <0.01 | 0.014 | --- | --- | --- | --- | <0.01 | --- | --- | --- |
| 1260A | 1 hour vac. dist. | 0.030 | 21.6 | 3.67 | 1.22 | 18 | <0.01 | VW | 3 | 9 | T | 0.040 | ND | T | ND | ND | VW | ND | VW | W-M |
| 1260B | no Pb added | 0.029 | 21.7 | 3.66 | 1.21 | 75 | 0.070 | T | 2 | 17 | T | 0.038 | ND | T | ND | ND | VW | ND | VW | W-M |
| 1300A | 10 ppm Pb added | 0.04 | 19.92 | 4.10 | 1.20 | <10 | <0.01 | 15 | 13 | 47 | <0.01 | 0.01 | --- | --- | --- | --- | <0.01 | --- | --- | --- |
| 1300B | | 0.04 | 19.85 | 4.09 | 1.19 | 42 | 0.080 | 15 | 14 | 49 | <0.01 | 0.01 | --- | --- | --- | --- | <0.01 | --- | --- | --- |
| 1277A | 25 ppm Pb added | 0.05 | 20.25 | 3.83 | 1.17 | 13 | <0.01 | 24 | --- | 34 | --- | 0.040 | --- | --- | --- | --- | --- | --- | --- | --- |
| 1277B | | 0.05 | 20.39 | 3.91 | 1.22 | 66 | 0.046 | 25 | --- | 33 | --- | 0.039 | --- | --- | --- | --- | --- | --- | --- | --- |
| 1278A | 50 ppm Pb added | 0.06 | 20.27 | 3.84 | 1.17 | 12 | <0.01 | 33 | --- | 31 | --- | 0.053 | --- | --- | --- | --- | --- | --- | --- | --- |
| 1278B | | 0.05 | 20.32 | 3.92 | 1.19 | 64 | 0.047 | 34 | --- | 24 | --- | 0.042 | --- | --- | --- | --- | --- | --- | --- | --- |
| 1286A | 60 ppm Pb added | 0.04 | 19.78 | 4.05 | 1.22 | <10 | <0.01 | 40 | --- | 48 | <0.01 | 0.015 | --- | --- | --- | --- | --- | --- | --- | --- |
| 1286B | | 0.04 | 19.92 | 4.06 | 1.19 | 62 | 0.076 | 40 | --- | 67 | <0.01 | 0.017 | --- | --- | --- | --- | --- | --- | --- | --- |
| 1261A | 100 ppm Pb added | 0.04 | 20.5 | 4.10 | 1.29 | 18 | <0.01 | 56 | 60 | 9 | T | 0.037 | ND | T | ND | ND | VW | ND | VW | W-M |
| 1261B | | 0.04 | 20.4 | 4.08 | 1.30 | 65 | 0.062 | 110 | 54 | 11 | T | 0.035 | ND | T | ND | ND | VW | ND | VW | W-M |
| 1321A | 100 ppm Pb added | 0.07 | 19.54 | 3.92 | 1.11 | <10 | <0.01 | 64 | --- | 70 | <0.01 | 0.021 | ND | ND | ND | ND | <0.01 | ND | VW | W |
| 1321C | two carbon levels | 0.11 | 19.58 | 3.88 | 1.13 | <10 | <0.01 | 60 | --- | 70 | <0.01 | 0.021 | ND | ND | ND | ND | <0.01 | ND | VW | W |
| 1302A | 100 ppm Pb added | 0.039 | 19.80 | 4.08 | 1.16 | 12 | <0.01 | 42 | --- | 77 | <0.01 | 0.018 | --- | --- | --- | --- | <0.01 | --- | --- | --- |
| 1302B | 10 min. vac. dist. | 0.039 | 19.98 | 3.96 | 1.18 | 91 | 0.080 | 35 | --- | 67 | <0.01 | 0.017 | --- | --- | --- | --- | <0.01 | --- | --- | --- |
| 1323A | 100 ppm Pb added | 0.07 | 19.62 | 3.88 | 1.12 | <10 | <0.01 | 12 | --- | 50 | <0.01 | 0.020 | ND | ND | ND | ND | <0.01 | ND | VW | W |
| 1323B | 30 min. vac. dist. | 0.09 | 19.60 | 3.80 | 1.11 | 69 | 0.068 | 4 | --- | 50 | <0.01 | 0.023 | ND | ND | ND | ND | <0.01 | ND | VW | W |
| 1280A | 100 ppm Pb added | 0.09 | 20.29 | 4.08 | 1.31 | 14 | <0.01 | <10 | 7 | 42 | --- | 0.037 | --- | --- | --- | --- | --- | --- | --- | --- |
| 1280B | 1 hour vac. dist. | 0.09 | 20.26 | 3.95 | 1.31 | 77 | 0.048 | <10 | 8 | 30 | --- | 0.039 | --- | --- | --- | --- | --- | --- | --- | --- |
| 1304-1* | zero | 0.05 | 19.92 | 4.09 | 1.16 | <10 | <0.01 | 70 | --- | 57 | <0.01 | 0.017 | --- | --- | --- | --- | <0.01 | --- | --- | --- |
| 1304-2 | 1 min. | 0.06 | 19.43 | 4.02 | 1.17 | 12 | <0.01 | 68 | --- | 60 | <0.01 | 0.022 | --- | --- | --- | --- | <0.01 | --- | --- | --- |
| 1304-3 | Sampled at 3 min. | 0.05 | 19.94 | 3.92 | 1.18 | <10 | <0.01 | 56 | --- | 52 | <0.01 | 0.012 | --- | --- | --- | --- | <0.01 | --- | --- | --- |
| 1304-4 | 10 min. | 0.05 | 19.97 | 4.05 | 1.17 | <10 | <0.01 | 37 | --- | 56 | <0.01 | 0.017 | --- | --- | --- | --- | <0.01 | --- | --- | --- |
| 1304-5 | 30 min. | 0.05 | 19.87 | 4.07 | 1.17 | <10 | <0.01 | <10 | --- | 59 | <0.01 | 0.012 | --- | --- | --- | --- | <0.01 | --- | --- | --- |
| 1322-1* | zero | 0.07 | 19.51 | 3.92 | 1.12 | <10 | <0.01 | 55 | --- | 40 | <0.01 | 0.026 | ND | ND | ND | ND | <0.01 | ND | VW | W |
| 1322-2 | 10 min. | 0.07 | 19.56 | 3.88 | 1.13 | <10 | <0.01 | 44 | --- | 30 | <0.01 | 0.026 | ND | ND | ND | ND | <0.01 | ND | VW | W |
| 1322-3 | Sampled at 15 min. | 0.07 | 19.60 | 3.88 | 1.11 | <10 | <0.01 | 19 | --- | 50 | <0.01 | 0.027 | ND | ND | ND | ND | <0.01 | ND | VW | W |
| 1322-4 | 20 min. | 0.07 | 19.56 | 3.87 | 1.12 | <10 | <0.01 | 11 | --- | 50 | <0.01 | 0.021 | ND | ND | ND | ND | <0.01 | ND | VW | W |
| 1322-5 | 30 min. | 0.06 | 19.60 | 3.83 | 1.12 | <10 | <0.01 | 6 | --- | 70 | <0.01 | 0.027 | ND | ND | ND | ND | <0.01 | ND | VW | W |

(a) Intentional addition of 35 ppm boron to B ingot of each heat

(b) Intentional addition of 0.045% zirconium to B ingot of each heat

(c) Intentional addition of the amount of lead specified under the "Purpose of Heat" column

(d) Re-analyses for lead; these values are preferred over original analyses

LP - Low as Possible

* - 100 ppm lead added at zero time

M = moderate - 0.1 to 1 %

W = weak - 0.01 to 0.1%

VW = very weak - 0.001 to 0.01%

T = trace - 0.001 to 0.0001%

ND = not detected

Table 3

## RUPTURE TEST DATA FOR THE 20Cr-2.5Ti-0.8Al ALLOY

| No Boron or Zirconium Added | | | | | | | 0.0035% Boron and 0.045% Zirconium Added | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Heat | Lead Content (ppm) | Temp. (°F) | Stress (psi) | Rupture Time (hours) | Elongation (% in 1 inch) | Reduction of Area (%) | Heat | Lead Content (ppm) | Temp. (°F) | Stress (psi) | Rupture Time (hours) | Elongation (% in 1 inch) | Reduction of Area (%) |
| Base Alloy | | | | | | | | | | | | | |
| 1249A | 3 | 1200 | 87,000 | 25.4 | 7 | 11 | 1249B | 4 | 1200 | 87,000 | 70.9 | 15 | 19 |
| | | | 80,000 | 79.0 | 4 | 4 | | | | 82 000 | 115.7 | 14 | 16 |
| | | | 74,000 | 217.7 | 5 | 8 | | | | | | | |
| | | 1500 | 25,000 | 45.6 | -- | 10 | | | 1500 | 25,000 | 75.5 | 22 | 38 |
| | | | 22,000 | 107.0 | 13 | 14 | | | | 22 500 | 188.1 | 28 | 45 |
| | | | 20 000 | 148.7 | 14 | 13 | | | | | | | |
| | | | 18,000 | 225.8 | 15 | 14 | | | | | | | |
| Base Alloy Plus 60 Minutes Vacuum Distillation | | | | | | | | | | | | | |
| 1259A | 4 | 1200 | 80,000 | 11.4 | 6 | 14 | 1259B | <10 | 1200 | 80,000 | 64.8 | 6 | 10 |
| | | | 76,000 | 107.3 | 3 | 11 | | | | 79,000 | 126.8 | 6 | 13 |
| | | | 74,000 | 188.6 | 4 | 9 | | | | 76,000 | 213.1 | 4 | 12 |
| | | | 70,000 | 232 | 3 | 11 | | | | | | | |
| | | 1500 | 25,000 | 81.5 | 5 | 11 | | | 1500 | 30,000 | 77.3 | 19 | 21 |
| | | | 23,000 | 136.0 | 10 | 14 | | | | 26,000 | 154.8 | 19 | 25 |
| | | | 22,000 | 221.9 | 8 | 9 | | | | 22,000 | 336.3 | 19 | 24 |
| 10 ppm Lead Addition | | | | | | | | | | | | | |
| 1298A | <10 | 1200 | 90,000 | 10.7 | 7 | 8 | 1298B | <10 | 1200 | 90,000 | 24.7 | 8 | 9 |
| | | | 80,000 | 68.3 | 2 | 7 | | | | 85,000 | 71.9 | 2 | 8 |
| | | | 70,000 | 282.1 | 3 | 9 | | | | 80,000 | 148.2 | 3 | 12 |
| | | 1500 | 30,000 | 16.3 | 8 | 10 | | | 1500 | 40,000 | 5.4 | 15 | 19 |
| | | | 25,000 | 46.6 | 6 | 10 | | | | 30,000 | 27.0 | 18 | 21 |
| | | | 22,000 | 92.7 | 6 | 6 | | | | 25,000 | 77.3 | 7 | 13 |
| | | | 18,000 | 187.8 | 3 | 5 | | | | 20,000 | 245.7 | 11 | 16 |
| 25 ppm Lead Addition | | | | | | | | | | | | | |
| 1275A | 21 | 1200 | 78,000 | 59.6 | 4 | 9 | 1275B | 20 | 1200 | 100,000 | 7.0 | 9 | 12 |
| | | | 75,000 | 71.4 | 3 | 8 | | | | 90,000 | 24.4 | 5 | 10 |
| | | | 70,000 | 135.5 | 3 | 7 | | | | 80,000 | 301.3 | 2 | 6 |
| | | 1500 | 25,000 | 46.6 | -- | 4 | | | 1500 | 30,000 | 48.1 | 6 | 9 |
| | | | 22,000 | 79.6 | 3 | 5 | | | | 25,000 | 99.5 | 6 | 9 |
| | | | 20,000 | 121.4 | 3 | 2 | | | | 23,000 | 136.0 | 4 | 7 |
| 50 ppm Lead Addition | | | | | | | | | | | | | |
| 1276A | 27 | 1200 | 70,000 | 29.4 | 4 | 10 | 1276B | 24 | 1200 | 90,000 | 9.0 | 6 | 11 |
| | | | 63,000 | 134.6 | 2 | 7 | | | | 87,500 | 30.4 | 4 | 11 |
| | | | 60,000 | 184.0 | 3 | 4 | | | | 85,000 | 127.2 | 4 | 6 |
| | | 1500 | 25,000 | 46.4 | -- | 4 | | | 1500 | 30,000 | 39.3 | 8 | 8 |
| | | | 22,000 | 97.2 | 3 | 3 | | | | 26,000 | 70.7 | 6 | 9 |
| | | | 20,000 | 128.7 | 2 | 4 | | | | 22,000 | 177.8 | 3 | 7 |
| 60 ppm Lead Addition | | | | | | | | | | | | | |
| 1301A | 36 | 1200 | 80,000 | 6.5 | 3 | 9 | 1301B | 34 | 1200 | 90,000 | 19.2 | 6 | 10 |
| | | | 70,000 | 72.1 | 2 | 6 | | | | 85,000 | 53.2 | 3 | 8 |
| | | | 60,000 | 306.5 | 2 | 6 | | | | 80,000 | 148.8 | 3 | 6 |
| | | 1500 | 25,000 | 29.9 | 4 | 3 | | | 1500 | 30,000 | 25.4 | 12 | 14 |
| | | | 20,000 | 72.5 | 3 | 5 | | | | 25,000 | 65.6 | 10 | 12 |
| | | | 15,000 | 103.6 | 2 | <1 | | | | 22,000 | 103.7 | 10 | 11 |
| | | | 15,000 | 133±11 | 2 | 2 | | | | 20,000 | 188.3 | 9 | 9 |
| 100 ppm Lead Addition | | | | | | | | | | | | | |
| 1257A | 110 | 1200 | 78,500 | 8.6 | 2 | 4 | 1257B | 95 | 1200 | 83,500 | 27.4 | 4 | 7 |
| | | | 65,000 | 51.8 | 3 | 5 | | | | 74,000 | 214.8 | 2 | 3 |
| | | | 60,000 | 107.4 | 2 | 4 | | | | | | | |
| | | 1500 | 22,000 | 32.8 | 2 | 2 | | | 1500 | 24,000 | 65.3 | 5 | 2 |
| | | | 18,000 | 73.4 | 4 | <1 | | | | 22,000 | 105.7 | 5 | 2 |
| | | | 16,500 | 92.3 | 4 | 1 | | | | 20,000 | 103.4 | 5 | 2 |
| 100 ppm Lead Addition Plus 10 Minutes Vacuum Distillation | | | | | | | | | | | | | |
| 1303A | 34 | 1200 | 80,000 | 5.1 | 3 | 12 | 1303B | 45 | 1200 | 90,000 | 28.6 | 5 | 11 |
| | | | 70,000 | 90.7 | 1 | 5 | | | | 82 000 | 157.7 | 4 | 7 |
| | | | 65,000 | 185.8 | 2 | 8 | | | | 80,000 | 177.4 | 3 | 8 |
| | | 1500 | 30,000 | 15.4 | 7 | 9 | | | 1500 | 30,000 | 26.1 | 13 | 16 |
| | | | 25,000 | 30.9 | 6 | 8 | | | | 25,000 | 65.7 | 12 | 13 |
| | | | 20,000 | 91.1 | 7 | 2 | | | | 20,000 | 199.1 | 5 | 11 |
| | | | 15,000 | 152.7 | 2 | 2 | | | | | | | |
| 00 ppm Lead Addition Plus 60 Minutes Vacuum Distillation | | | | | | | | | | | | | |
| 1279A | 7 | 1200 | 84,000 | 52.6 | 7 | 11 | 1279B | 6 | 1200 | 100,000 | 32.6 | 13 | 14 |
| | | | 80,000 | 86.2 | 8 | 11 | | | | 90 000 | 82.5 | 10 | 12 |
| | | | 76,000 | 131.3 | 7 | 9 | | | | 80 000 | 287.1 | 8 | 10 |
| | | 1500 | 22,000 | 59.4 | 1 | 13 | | | 1500 | 27,000 | 77.6 | 27 | 33 |
| | | | 20,000 | 77.8 | .6 | 15 | | | | 25,000 | 105.2 | 25 | 33 |
| | | | 18,000 | 97.3 | 15 | 16 | | | | 23,000 | 143.5 | 29 | 34 |
| | | | 16,000 | 136.4 | 15 | 13 | | | | | | | |

## Table 4

## INFLUENCE OF LEAD CONTENT ON THE 100-HOUR RUPTURE STRENGTH AND RUPTURE DUCTILITY OF THE 20Cr-2.5Ti-0.8Al ALLOY AT 1200°F AND 1500°F

| Heat | Analyzed Pb Content (ppm) | Pb Added (ppm) | Holding Time in Vacuum (min.) | 100-Hour Rupture Strength (psi) 1200°F | 100-Hour Rupture Strength (psi) 1500°F | Average Reduction of Area (%) 1200°F | Average Reduction of Area (%) 1500°F |
|---|---|---|---|---|---|---|---|
| | | | | No Boron or Zirconium Added | | | |
| 1249A | 3 | none | zero | 78,000 | 22,000 | 8 | 13 |
| 1259A | 4 | none | 60 | 73,000 | 24,200 | 11 | 11 |
| 1298A | <10 | 10 | zero | 78,000 | 21,000 | 8 | 8 |
| 1275A | 21 | 25 | zero | 73,000 | 21,000 | 8 | 4 |
| 1276A | 27 | 50 | zero | 63,500 | 22,000 | 8 | 4 |
| 1301A | 36 | 60 | zero | 66,000 | 16,500 | 7 | 3 |
| 1257A | 110 | 100 | zero | 60,000 | 15,500 | 4 | 1 |
| 1303A | 34 | 100 | 10 | 66,000 | 18,200 | 8 | 5 |
| 1279A | 7 | 100 | 60 | 78,000 | 18,200 | 10 | 14 |
| | | | | 0.0035% Boron and 0.045% Zirconium Added | | | |
| 1249B | 4 | none | zero | 84,500 | 25,000 | 18 | 42 |
| 1259B | <10 | none | 60 | 78,500 | 28,300 | 12 | 23 |
| 1298B | <10 | 10 | zero | 81,500 | 23,600 | 10 | 15 |
| 1275B | 20 | 20 | zero | 84,500 | 25,000 | 9 | 8 |
| 1276B | 24 | 50 | zero | 81,500 | 25,000 | 9 | 8 |
| 1301B | 34 | 60 | zero | 81,500 | 22,700 | 8 | 12 |
| 1257B | 95 | 100 | zero | 76,000 | 21,500 | 5 | 2 |
| 1303B | 45 | 100 | 10 | 81,500 | 22,700 | 9 | 13 |
| 1279B | 6 | 100 | 60 | 89,000 | 25,000 | 12 | 33 |

Table 5

## RUPTURE TEST DATA FOR THE 20Cr-3.8Ti-1.2Al ALLOY

| No Boron or Zirconium Added | | | | | | | 0.0035% Boron and 0.045% Zirconium Added | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Heat | Lead Content (ppm) | Temp. (°F) | Stress (psi) | Rupture Time (hours) | Elongation (% in 1 inch) | Reduction of Area (%) | Heat | Lead Content (ppm) | Temp. (°F) | Stress (psi) | Rupture Time (hours) | Elongation (% in 1 inch) | Reduction of Area (%) |
| Base Alloy | | | | | | | | | | | | | |
| 1247A | <10 | 1200 | 87,000 | 15.2 | 3 | 4 | 1247B | <10 | 1200 | 100,000 | 12.4 | 3 | 9 |
| | | | 87,000 | 19.2* | -- | -- | | | | 90.000 | 374.5 | 2 | 5 |
| | | | 87,000 | 12.8 | 4 | 7 | | | | 87.000 | 196.7 | 3 | 9 |
| | | | 78,000 | 65.0 | 2 | 3 | | | | | | | |
| | | | 70,000 | 62.4 | 3 | 7 | | | | | | | |
| | | 1500 | 30,000 | 43.0 | 2 | <1 | | | 1500 | 40.000 | 45.7 | 6 | 10 |
| | | | 26,000 | 110.8 | 2 | 3 | | | | 34.000 | 112.7 | 9 | 12 |
| | | | | | | | | | | 26,000 | 407.4 | 10 | 12 |
| Base Alloy | | | | | | | | | | | | | |
| 1290A | <10 | 1200 | 90,000 | 9.7 | 2 | 6 | 1290B | <10 | 1200 | 110.000 | 40.3 | 2 | 8 |
| | | | 80,000 | 91.0 | 2 | 5 | | | | 100,000 | 14.9 | <1 | 6 |
| | | | 70,000 | 57.6 | 2 | 6 | | | | 95,000 | 344.4 | 2 | 4 |
| | | | 70,000 | 177.1 | 1 | 4 | | | | 90,000 | 412.7 | 3 | 5 |
| | | | 60,000 | 562.6 | 1 | 3 | | | | | | | |
| | | 1500 | 30,000 | 35.2 | <1 | 1 | | | 1500 | 50.000 | 17.6 | 6 | 11 |
| | | | 25,000 | 125.3 | 2 | 2 | | | | 40,000 | 65.4 | 7 | 6 |
| | | | 20,000 | 187.1 | 3 | 2 | | | | 35,000 | 153.0 | 6 | 8 |
| | | | 20,000 | 180.5 | 5 | 4 | | | | 30,000 | 316.0 | 8 | 9 |
| | | | 15,000 | 1348.3 | -- | 4 | | | | 25,000 | 797.4 | 11 | 12 |
| Base Alloy | | | | | | | | | | | | | |
| 1314A | 4 | 1200 | 70,000 | 302.0 | 2 | 2 | 1314B | 4 | 1200 | 100.000 | 182.5 | 8 | 10 |
| | | | | | | | | | | 90,000 | 644.3 | 5 | 7 |
| | | 1500 | 40.000 | 9.6 | 3 | 3 | | | 1500 | 35,000 | 97.3 | 13 | 20 |
| | | | 25,000 | 105.7 | -- | 7 | | | | 25.000 | 487.5 | 19 | 26 |
| | | | 15.000 | 758.9 | 21 | 21 | | | | | | | |
| Base Alloy Plus 60 Minutes Vacuum Distillation | | | | | | | | | | | | | |
| 1260A | 3 | 1200 | 86,000 | 65 | <1 | 3 | 1260B | 2 | 1200 | 100.000 | 166.3 | 7 | 10 |
| | | | 80,000 | 89.2 | 2 | 3 | | | | 95.000 | 243.2 | 6 | 9 |
| | | | 76,000 | 134.0 | 3 | 3 | | | | 90.000 | 443.8 | 4 | 7 |
| | | 1500 | 28,000 | 69.0 | 3 | 2 | | | 1500 | 34,000 | 59.4 | 16 | 20 |
| | | | 26,000 | 94.9 | 3 | 2 | | | | 34.000 | 52.5** | -- | -- |
| | | | 24,000 | 70.4 | 3 | 2 | | | | 32.000 | 225.3 | 14 | 17 |
| | | | 24,000 | 70.2 | 3 | 3 | | | | 31,000 | 139.8 | 18 | 18 |
| 10 ppm Lead Addition | | | | | | | | | | | | | |
| 1300A | 13 | 1200 | 90,000 | 24.7 | 2 | 7 | 1300B | 14 | 1200 | 110.000 | 40.9 | 3 | 7 |
| | | | 80,000 | 79.9 | 1 | 2 | | | | 100.000 | 159.9 | 4 | 6 |
| | | | 70,000 | 137.3 | 2 | 2 | | | | 90.000 | 472.1 | 2 | 4 |
| | | 1500 | 30,000 | 41.0 | <1 | <1 | | | 1500 | 50.000 | 17.0 | 6 | 10 |
| | | | 25,000 | 64.0 | <1 | 1 | | | | 40.000 | 62.4 | 4 | 6 |
| | | | 22,500 | 116.8 | 2 | <1 | | | | 35 000 | 116.6 | 3 | 4 |
| | | | 20,000 | 74.0 | 3 | <1 | | | | 30.000 | 194.5 | 5 | 5 |
| | | | 15,000 | 454.1 | 3 | <1 | | | | 25.000 | 336.8 | 4 | 3 |
| 25 ppm Lead Addition | | | | | | | | | | | | | |
| 1277A | 24 | 1200 | 75,000 | 34.2 | 1 | 2 | 1277B | 25 | 1200 | 105.000 | 50.6 | 3 | 5 |
| | | | 70,000 | 101.4 | 6 | 4 | | | | 100.000 | 77.9 | 3 | 6 |
| | | | 66,000 | 117.2 | 1 | 2 | | | | 95.000 | 186.7 | 2 | 5 |
| | | | | | | | | | | 80 000 | 1094.1 | 3 | 2 |
| | | 1500 | 25,000 | 35.5 | 2 | <1 | | | 1500 | 40.000 | 49.7 | 4 | 5 |
| | | | 20,000 | 65.1 | 1 | <1 | | | | 30.000 | 135.3 | 3 | 3 |
| | | | 18,000 | 69.1 | 1 | <1 | | | | 27.000 | 195.2 | 2 | <1 |
| | | | 17,000 | 80.3 | 2 | <1 | | | | 23.000 | 364.5 | <1 | 4 |
| | | | 16,000 | 139.9 | 2 | <1 | | | | | | | |
| 50 ppm Lead Addition | | | | | | | | | | | | | |
| 1278A | 33 | 1200 | 60,000 | 20.8 | <1 | 3 | 1278B | 34 | 1200 | 100.000 | 109.6 | 1 | 5 |
| | | | 55,000 | 444.9 | 1 | 2 | | | | 90.000 | 248.4 | 2 | 3 |
| | | | 55,000 | 253.3 | <1 | <1 | | | | 80 000 | 690.0 | 1 | 2 |
| | | | 50,000 | 263.9 | <1 | 2 | | | | | | | |
| | | 1500 | 20,000 | 53.3 | 2 | <1 | | | 1500 | 50.000 | 9.6 | 4 | 6 |
| | | | 17,000 | 48.5 | 3 | <1 | | | | 40,000 | 33.2 | 2 | 3 |
| | | | 16,000 | 95.0 | <1 | <1 | | | | 32,000 | 65.4 | 3 | 1 |
| | | | 14,000 | 124.7 | 1 | <1 | | | | 26,000 | 117.5 | 2 | <1 |
| | | | | | | | | | | 23,000 | 174.5 | 1 | <1 |
| 60 ppm Lead Addition | | | | | | | | | | | | | |
| 1286A | 40 | 1200 | 70,000 | 0.1 | <1 | 2 | 1286B | 40 | 1200 | 100.000 | 18.2 | 2 | 7 |
| | | | 60,000 | 2.2 | 2 | 2 | | | | 94.000 | 76.1 | 1 | 6 |
| | | | 53 000 | 8.1 | 1 | 3 | | | | 90 000 | 141.4 | 2 | 4 |
| | | | 50,000 | 352.1 | 1 | <1 | | | | | | | |
| | | 1500 | 30,000 | 24.1 | <1 | <1 | | | 1500 | 50.000 | 15.3 | 3 | 4 |
| | | | 25,000 | 51.8 | <1 | <1 | | | | 40.000 | 40.9 | 2 | 2 |
| | | | 20,000 | 49.9 | <1 | <1 | | | | 30.000 | 82.0 | <1 | <1 |
| | | | 15,000 | 126.0 | <1 | <1 | | | | 20.000 | 291.0 | 2 | <1 |

* Fractured in threads

**Test stopped when controller malfunctioned

Table 5 (continued)

| No Boron or Zirconium Added | | | | | | | 0.0035% Boron and 0.045% Zirconium Added | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Heat | Lead Content (ppm) | Temp. (°F) | Stress (psi) | Rupture Time (hours) | Elongation (% in 1 inch) | Reduction of Area (%) | Heat | Lead Content (ppm) | Temp. (°F) | Stress (psi) | Rupture Time (hours) | Elongation (% in 1 inch) | Reduction of Area (%) |
| 100 ppm Lead Addition | | | | | | | | | | | | | |
| 1261A | 60 | 1200 | 80,000 | <1* | <1 | <1 | 1261B | 54 | 1200 | 80.000 | 40,1 | 1 | 2 |
| | | | 60,000 | 0,5 | 1 | 2 | | | | 75,000 | 47.6 | 2 | 3 |
| | | | 40,000 | 156,3 | 1 | <1 | | | | 70,000 | 129,3 | 2 | 2 |
| | | | | | | | | | | 60.000 | 379.8 | <1 | <1 |
| | | 1500 | 26,000 | 5,8 | 2 | <1 | | | 1500 | 50,000 | 4.6 | 4 | 4 |
| | | | 17,000 | 14,1 | <1 | <1 | | | | 40.000 | 14.8 | 1 | 2 |
| | | | 10,000 | 79,4 | 2 | <1 | | | | 26 000 | 27.5 | 4 | 2 |
| | | | | | | | | | | 21,000 | 50,9 | 3 | <1 |
| | | | | | | | | | | 15.000 | 146,5 | -- | 1 |
| 100 ppm Lead Addition - Two Carbon Levels | | | | | | | | | | | | | |
| 1321A | 64 | 1200 | 50,000 | 1,5 | <1 | 2 | | | | | | | |
| | | | 45,000 | 301,1 | 1 | <1 | | | | | | | |
| | | | 40,000 | 220,0 | 1 | <1 | | | | | | | |
| | | 1500 | 25,000 | 10,1 | 1 | <1 | | | | | | | |
| | | | 15,000 | 45,3 | -- | <1 | | | | | | | |
| | | | 10,000 | 130,7 | -- | <1 | | | | | | | |
| 1321C | 60 | 1200 | 70,000 | 20,6 | 1 | 4 | | | | | | | |
| | | | 60,000 | 46,9 | <1 | 3 | | | | | | | |
| | | | 50,000 | 161,8 | <1 | 1 | | | | | | | |
| | | 1500 | 25,000 | 5,5 | <1 | <1 | | | | | | | |
| | | | 15,000 | 29,2 | 2 | <1 | | | | | | | |
| | | | 10,000 | 129,4 | 4 | 2 | | | | | | | |
| 100 ppm Lead Addition Plus 10 Minutes Vacuum Distillation | | | | | | | | | | | | | |
| 1302A | 42 | 1200 | 70,000 | 0,7 | <1 | <1 | 1302B | 35 | 1200 | 100,000 | 17,1 | 2 | 6 |
| | | | 60,000 | 1,1 | 1 | 4 | | | | 94.000 | 117,8 | 1 | 4 |
| | | | 50,000 | 90,9** | 2 | <1 | | | | 90.000 | 150,8 | 2 | 4 |
| | | 1500 | 25,000 | 22,2 | <1 | <1 | | | 1500 | 50 000 | 15,3 | 3 | 4 |
| | | | 20,000 | 73,4 | <1 | <1 | | | | 40,000 | 38,0 | <1 | 2 |
| | | | 15,000 | 61,0 | <1 | <1 | | | | 30,000 | 102,9 | 2 | <1 |
| | | | 15,000 | 83,7 | <1 | <1 | | | | 25.000 | 146,0 | 2 | <1 |
| 100 ppm Lead Addition Plus 30 Minutes Vacuum Distillation | | | | | | | | | | | | | |
| 1322A | 6 | 1200 | 90,000 | 11,6 | 1 | 5 | | | | | | | |
| | | | 70,000 | 63,9 | 2 | 4 | | | | | | | |
| | | 1500 | 30,000 | 26,7 | 2 | 2 | | | | | | | |
| | | | 20,000 | 97,0 | 2 | <1 | | | | | | | |
| 1323A | 12 | 1200 | 90.000 | 1,5 | 1 | 9 | 1323B | 4 | 1200 | 110.000 | 28,7 | 3 | 8 |
| | | | 70,000 | 77,8 | <1 | 2 | | | | 100 000 | 105,6 | 2 | 5 |
| | | 1500 | 30,000 | 21,7 | 1 | 1 | | | 1500 | 40.000 | 40,8 | 5 | 8 |
| | | | 20,000 | 62,4 | 2 | <1 | | | | 30 000 | 102,0 | 3 | 4 |
| | | | | | | | | | | 20 000 | 310,0 | 4 | 2 |
| 100 ppm Lead Addition Plus 60 Minutes Vacuum Distillation | | | | | | | | | | | | | |
| 1280A | 7 | 1200 | 87,000 | 37,5 | 3 | 4 | 1280B | 8 | 1200 | 105.000 | 65,8 | 8 | 9 |
| | | | 80,000 | 65,0 | 2 | 3 | | | | 100 000 | 143,2 | 7 | 8 |
| | | | 76,000 | 83,1 | 2 | 3 | | | | 95.000 | 196,9 | 7 | 11 |
| | | | 70,000 | 113,5 | 2 | 3 | | | | 90 000 | 555,3 | 5 | 7 |
| | | 1500 | 40,000 | 6,0 | 4 | 4 | | | 1500 | 35 000 | 73,1 | 14 | 22 |
| | | | 25,000 | 32,7 | 7 | 9 | | | | 32.500 | 103,4 | 16 | 23 |
| | | | 20,000 | 74,3 | 12 | 11 | | | | 30 000 | 141,9 | 15 | 19 |
| | | | 18,000 | 132,7 | 21 | 14 | | | | | | | |

* Broke on loading

** Fractured in threads at 7,6 hours and restarted

Table 6

## INFLUENCE OF LEAD CONTENT ON THE 100-HOUR RUPTURE STRENGTH AND RUPTURE DUCTILITY OF THE 20Cr-3.8Ti-1.2Al ALLOY AT 1200°F AND 1500°F

| Heat | Analyzed Pb Content (ppm) | Pb Added (ppm) | Holding Time in Vacuum (min.) | 100-Hour Rupture Strength (psi) | | Average Reduction of Area (%) | |
|---|---|---|---|---|---|---|---|
| | | | | 1200°F | 1500°F | 1200°F | 1500°F |
| | | | No Boron or Zirconium Added | | | | |
| 1247A | <10 | none | zero | 71,000 | 24,600 | 5 | 2 |
| 1290A | <10 | none | zero | 71,000 | 24,600 | 5 | 3 |
| 1314A | 4 | none | zero | 79,000 | 24,600 | 2 | 10 |
| 1260A | 3 | none | 60 | 80,000 | 24,500 | 3 | 2 |
| 1300A | 13 | 10 | zero | 76,000 | 22,800 | 4 | <1 |
| 1277A | 24 | 25 | zero | 68,000 | 17,000 | 3 | <1 |
| 1278A | 33 | 50 | zero | 58,000 | 15,000 | 2 | <1 |
| 1286A | 40 | 60 | zero | 47,000 | 16,000 | 2 | <1 |
| 1261A | 60 | 100 | zero | 41,000 | 8,900 | <1 | <1 |
| 1321A * | 64 | 100 | zero | 41,000 | 11,100 | 1 | <1 |
| 1321C * | 60 | 100 | zero | 53,900 | 10,600 | 3 | 1 |
| 1302A | 42 | 100 | 10 | 49,700 | 15,000 | 2 | <1 |
| 1322A | 6 | 100 | 30 | 68,000 | 20,000 | 5 | 1 |
| 1323A | 12 | 100 | 30 | 68,000 | 18,200 | 6 | <1 |
| 1280A | 7 | 100 | 60 | 76,000 | 18,900 | 3 | 10 |
| | | | 0.0035% Boron and 0.045% Zirconium Added | | | | |
| 1247B | <10 | none | zero | 97,500 | 34,400 | 8 | 11 |
| 1290B | <10 | none | zero | 97,500 | 37,000 | 6 | 9 |
| 1314B | 4 | none | zero | 105,000 | 34,400 | 9 | 23 |
| 1260B | 2 | none | 60 | 100,000 | 33,000 | 9 | 18 |
| 1300B | 14 | 10 | zero | 102,000 | 35,500 | 6 | 6 |
| 1277B | 25 | 25 | zero | 98,000 | 33,000 | 5 | 3 |
| 1278B | 34 | 50 | zero | 97,000 | 27,400 | 3 | 2 |
| 1286B | 40 | 60 | zero | 91,000 | 29,000 | 6 | 2 |
| 1261B | 54 | 100 | zero | 70,500 | 15,200 | 2 | 2 |
| 1302B | 35 | 100 | 10 | 92,000 | 29,000 | 5 | 2 |
| 1323B | 4 | 100 | 30 | 101,000 | 29,600 | 7 | 5 |
| 1280B | 8 | 100 | 60 | 100,000 | 33,000 | 9 | 21 |

* Intentionally high carbon heat