A PROPOSED METHOD FOR ESTIMATING RESIDUAL MATRIX CHEMISTRY IN NICKEL-BASE SUPERALLOYS

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
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SUMMARY

A method of estimating the residual matrix (gamma phase) composition of nickel-base superalloys is proposed. This method is based on geometric analysis of multicomponent phase diagrams and, for those elements where no phase diagrams are available, on the currently accepted phase computation methods.

Calculations of residual matrix for IN 713C, IN 713LC, IN 731X, and U 700 are generally in good agreement with those reported in the literature.

INTRODUCTION

Advances in gas turbine technology have required advances in the state of the art of nickel-base superalloys. Of particular concern is the development of highly alloyed materials which possess high temperature strength, corrosion resistance, and stability of mechanical properties for lives in excess of 10,000 hours. A correlation has frequently been observed between the loss of mechanical properties after exposure to elevated temperature for long times and microstructural instabilities exemplified by the formation of intermetallic compounds such as the sigma and mu phases.

Two approaches have been previously used to predict the microstructural stability of alloys; however, they both involve estimating the chemistry of the residual matrix or gamma phase of the alloy by

TM X-52530
making certain assumptions about precipitation of compounds such as carbides, borides, and \textit{gamma} prime. In one, the average electron vacancy concentration \((N_y)\) of the residual matrix may then be used as a stability criterion (refs. 1, 2); in the other, the computed chemistry of the residual matrix may be compared to known phase diagrams (ref. 3). Both approaches represent efforts to reduce a complex alloy system (involving as many as 15 alloy elements) to a system which may be more readily interpreted.

A major problem associated with the procedures mentioned above is that the \textit{gamma} prime chemistry is not well known for most complex superalloys; therefore, the computed residual matrix may have little basis in fact when compared to the actual chemistry. The element which appears to be a major offender is aluminum. The accepted methods for residual matrix computation assume that no aluminum exists in the residual matrix. Recent investigators have indicated that this is not true (refs. 4, 5). The currently used methods also fail to account for any interaction among other elements present, which ternary phase diagrams clearly show exist.

The method described in this paper is offered as a guide designed to eliminate some of the oversimplifications currently being used to compute the \textit{gamma} composition. This is done by a geometric analysis of an existing phase diagram. The specific example cited will be that of nickel-base superalloys using an analysis of a phase diagram for nickel-chromium-aluminum (Ni-Cr-Al) at 750 C. The general geometric principles used here are applicable to any two phase field in a multicomponent system, where the tie lines are known and can be conveniently expressed by a system of equations. It is recognized that the proposed method still relies greatly on empiricism. However, the technique, if extended to its most general case, which would involve geometric analysis of an \(n\) component system, could more fully describe the chemistries of the phases present.
PROPOSED METHOD

Linear solution. - The major deviation of the proposed method from the ones currently used is that the final step involves the direct determination of the residual matrix using a published phase diagram for guidance instead of determining the composition of gamma from the difference between the melt chemistry and assumed precipitated phases. This portion of the calculation will be discussed first, then the entire procedure will be described.

The nickel rich corner of the Ni-Al-Cr phase diagram of Taylor and Floyd (ref. 6) is shown in figure 1. As a simplification the $\gamma$ solvus line is assumed to be linear, and the extensions of the $\gamma - \gamma'$ tie lines are assumed to intersect at 0.27 Al, 0 Cr. The diagram using these two simplifications is shown in figure 2. The composition of interest to the metallurgist (the gamma phase) is simply the intersection of the tie line on which our compositions (in the $\gamma + \gamma'$ field) lie and the solvus line. The assumed point of intersection at 0.27 Al, 0 Cr and the chemistry of the ternary alloy now define a tie line. An idealized solvus line has been defined in figure 2 by a straight line labeled Cr. The problem has now been reduced to finding the intersection of two straight lines. The equation of the solvus line is known. The equation of the tie line is completely defined by the assumed point of intersection of all tie lines and the composition of the alloy.

The idealized solvus line passes through (0.115 Al, 0 Cr, 0.885 Ni) and (0 Al, 0.6 Cr, 0.4 Ni). Its equation is:

$$\frac{Cr_\gamma}{0.6} + \frac{Al_\gamma}{0.115} = 1 \tag{1}$$

The tie lines pass through (0.27 Al, 0 Cr, 0.73 Ni) and (Cr$_a$, Al$_a$, and Ni$_a$) as defined by the alloy. The tie line equation is:

$$(0.27 - Al_a) Cr_\gamma + Cr_a Al_\gamma = 0.27 Cr_a \tag{2}$$
Solving equations (1) and (2) simultaneously for $\text{Cr}_\gamma$ and $\text{Al}_\gamma$

\[
\text{Cr}_\gamma = \frac{0.6 \times 0.27 \text{Cr}_a - 0.6 \times 0.115 \text{Cr}_a}{0.6(0.27 - \text{Al}_a) - 0.115 \text{Cr}_a}
\]  

\[
\text{Al}_\gamma = \frac{0.6 \times 0.115(0.27 - \text{Al}_a) - 0.115 \times 0.27 \text{Cr}_a}{0.6(0.27 - \text{Al}_a) - 0.115 \text{Cr}_a}
\]

where

\(a\) alloy chemistry

\(\gamma\) \(\gamma\) (matrix) chemistry

The behavior of molybdenum and tungsten in nickel-aluminum-chromium alloy systems is not known. However, in the Ni-Mo-Cr-W-Al system, one end of the gamma solvus line must lie in the Ni-Al binary diagram.

For the purpose of this paper, it will be assumed that Mo and W behave similarly to Cr but that the other end of the solvus lines for Mo and W passes through 0 Al, 0.8 Ni, 0.2(Mo, W) instead of 0.4 Ni, 0.6 Cr, as it does for chromium (fig. 2). The tie lines are again assumed to pass through the same point on the Ni-Al diagram giving the following equation for Mo and W:

\[
\text{Mo}_\gamma \text{ or } \text{W}_\gamma = \frac{0.2 \times 0.27 \text{M}_a - 0.2 \times 0.115 \text{M}_a}{0.2(0.27 - \text{Al}_a) - 0.115 \text{M}_a}
\]

where

\(\text{M}_a = \text{Mo}_a \text{ or } \text{W}_a\)

Because Mo and W are usually present in small quantities in the alloys of interest, they are assumed to have the same effect on Al solubility in gamma as Cr. The calculation for aluminum in the matrix is as shown in equation (4) except that the sum of $\text{Cr}_a + \text{Mo}_a + \text{W}_a$ is substituted for $\text{Cr}_a$. The solubilities of Cr, Mo, and W in gamma are
assumed to be independent of each other and the amount of nickel is then determined by difference.

\[ \text{Ni} = 1 - \text{Cr}_\gamma - \text{Mo}_\gamma - \text{W}_\gamma - \text{Al}_\gamma \]  

(6)

Where cobalt and iron are present in the alloy, they are assumed to substitute for nickel in proportion to their relative quantity in the alloy.

**Quadratic solution.** - The chromium and aluminum in the gamma may also be determined by fitting a parabola to the gamma solvus curve (instead of the straight line mentioned earlier). The results of this calculation are also shown in table I.

The calculation is identical to the one mentioned except that the gamma solvus line is assumed to be a parabola passing through (0 Cr, 0.12 Al), (0.2 Cr, 0.06 Al), and (0.3 Cr, 0.07 Al). The equation for the solvus is:

\[ \text{Al}_\gamma = 1.33 \text{Cr}_\gamma^2 - 0.566 \text{Cr}_\gamma + 0.12 \]  

(7)

Solving equation (7) simultaneously with (2) gives:

\[ \text{Cr}_\gamma = \frac{-B + \sqrt{B^2 - 4AC}}{2A} \]  

(8)

\[ \text{Al}_\gamma = 0.27 - \text{Cr}_\gamma \left(\frac{0.27 - \text{Al}_a}{\text{Cr}_a}\right) \]  

(9)

where

\[ A = 1.33 \]
\[ B = -0.566 - \left(\frac{0.27 - \text{Al}_a}{\text{Cr}_a}\right) \]
\[ C = -0.15 \]
The quadratic model gives higher Cr concentrations for the alloys considered and lower Al concentrations except for U 700 than the linear model for the alloys shown in table I. It is not obvious, however, that either is predicting the concentration substantially better than the other.

APPLICATION OF PROPOSED METHOD

The equations shown above require that the alloys be reduced to one containing only Ni, Cr, Al, W, Mo, Co, and Fe. To do this, one must first take into account the behavior of the more reactive metals (Ti, Hf, and Zr), carbon, boron, and other refractory elements (Ta, Nb) in the alloy. Because specific multicomponent phase diagrams are also lacking for these elements, they are handled in a method similar to the currently used phase computation procedures (ref. 2).

Specifically, it is assumed first that all boron forms an $M_3B_2$ boride having the composition $(Mo_{0.5}, Ti_{0.15}, Cr_{0.25}, Ni_{0.1})_3B_2$. It is then assumed that 50 percent of the carbon in the alloy forms monocarbides (MC) in the sequence TaC, NbC, TiC, ZrC, VC (i.e. TaC forms first). The carbides, however, are assumed in this paper to be nonstoichiometric in that it is assumed that the formula is given as:

$$MC = \left[ M^* \left(1-0.04(Mo_a+W_a)\right)^{(Mo + W)}0.04(Mo_a+W_a) \right]C$$

where $M^*$ is Ta, Nb, etc. and where $(Mo_a+W_a)$ are in at. % in the melt. That is, the Mo and W concentrations in the M fraction of MC are four times the Mo and W concentrations of the original melt. The remaining 50 percent of the carbon is assumed to form either $M_{23}C_6$ or $M_6C$. The $M_{23}C_6$ is assumed to have the composition $(Cr_{21}Mo_2)C_6$ and the $M_6C$ to have the composition $[2.5 Ni, 1 Co, 0.5 Cr, 2(Mo + W)]C$. 
The selection of \( M_{23}C_6 \) or \( M_6C \) is based on the correlation proposed earlier by the author (ref. 7). Specifically, \( M_{23}C_6 \) forms if:

\[
Cr > 3.5(Mo + 0.4W)
\]

Otherwise, \( M_6C \) will form.

The carbon and boron have now been accounted for. However, to further reduce the alloy to one containing only Ni, Al, Cr, Mo, W, Co, and Fe, an additional assumption is required concerning the Ti, Nb, Ta, Zr, and V not accounted for by boride and carbide formation. It will be assumed that all of the Ti, Nb, Ta, Zr, and one-half of V remaining will enter the gamma prime phase. This gamma prime will then be:

\[
(Ni, Co, Fe)_{3.3} (Ti, Nb, Ta, Zr, 1/2 V)
\]

It is recognized that this assumption, together with the geometric analysis implies the formation of two gamma primes. However, the use of the \( Ni_{3.3} \gamma \) gamma prime is strictly an artificial mathematical construction and is the final step in reducing the alloy to the Ni-Cr-Al-Mo-W-Fe-Co system. It is moreover consistent with the approach used in current methods for handling these elements. The form \( Ni_{3.3} \gamma \) is used in place of the more common \( Ni_3 \gamma \) because the lower limit of the gamma solvus in the (Nb and Ta)-Ni diagram is approximately 23 percent (ref. 8).

The final step in the computation is the application of equations (3), (4), (5), and (6) or (7), (8), (9), and (6).

A Fortran IV computer program for the method as detailed above is shown in the appendix. It should be noted that the program is based on the 750\(^{\circ}\) C phase diagram with the tie line intersection at 0.27 Al, 0 Cr, 0 Ni.

The results of this method for alloys IN 713C, IN 713LC, and IN 731X are compared in table I to results determined by Mihalisin (ref. 5), who determined the amount and composition of carbides and
gamma prime experimentally and computed the composition of gamma by maintaining mass balance. The U-700 results shown in table I were computed by the author based on the data of Kriege (ref. 4); however, carbides were ignored. It can be seen from table I that the agreement between the experimentally determined chemistry and the calculated chemistries for IN 713 is excellent; however, for IN 731X and U-700 it is poorer, particularly with respect to Co and Al. It is not known if the lack of agreement is due to the influence of Co or Ti on the element distributions in IN731X and U-700.

The reported melt chemistries on which the calculations are based are shown in table II.

CONCLUDING REMARKS

A method of estimating the residual matrix composition of nickel-base superalloys has been presented. This method, which is based on a combination of empiricism and geometric-analysis of a phase diagram appears to show promise for better estimation of residual matrix chemistry than methods currently being used. It is believed that the residual matrix compositions estimated by the method can offer useful guidance in alloy development from a stability and perhaps a corrosion resistance point of view. It offers refinements in methods of estimating the residual matrix composition which are intended to reduce the need for melting of alloys and running tedious and time consuming extractions and chemical analyses.

The gamma compositions computed by the proposed method compare well with those reported in the literature for the low titanium, cobalt free alloys IN 713C and IN 713LC. The computed concentrations of chromium and the sum of nickel, cobalt, and iron in high titanium alloys containing cobalt (U-700 and IN 731X) also compare well to those reported in the literature; however, the distribution among nickel, cobalt, and iron appears to be incorrect. The calculated aluminum for IN 731X and the calculated molybdenum for U-700 are in poorer agreement with the reported values than for the other alloys.
The limitations of the proposed method are not fully known because of a lack of reported gamma compositions for commercial alloys. Since no reliable data are available for tungsten containing alloys, the appropriateness of the method to alloys containing tungsten is unknown. It was shown that the method begins to become inadequate if titanium and cobalt are present at high levels in the alloy system, as in IN 731X and U-700. Inspection of figures 1 and 2 shows that if the chromium content at the last computation steps exceeds approximately 25 atomic percent, the method will fail because the tie line, assumed-solvus line intersection in the idealized diagram falls outside the $\gamma + \gamma'$ region of the real phase diagram.

It is hoped that as the chemistry of the phases in more alloys is determined and more detailed phase diagrams become available, the method can be improved and extended to other compositions. It should be emphasized that this is a preliminary form of calculation. Emphasis should be placed on development and geometric analysis in the nickel-rich regions of appropriate phase diagrams. Since in a two-phase region of a multi-component phase diagram, a tie line is always a straight line and a solvus surface can always be expressed mathematically, the potential of the type of analysis used on the Ni-Cr-Al system appears to offer considerable promise. It should be noted that the same technique can also be applied to determination of the gamma prime chemistry; however, the author has not explicitly done so in this writing.
```
C
C
C
PHASE EQUILIBR
C
C
NUMERICAL ILE
C
C
TI 1
C
AL 2
C
CR 4
C
CF 6
C
MC 1
C
V 8
C
w =
C
TA 10
C
CF 11
C
FF 12
C
B 13
C
ZK 14
C
RF 15
C
TT 16
C
INPUT IS IN
C
Af = ATOMIC
C
APCL = ATOMIC
C
AFCT = ATOMIC
C
MCDE 1 LINE
C
MCDE 2 LCLH
C
ITCH IS INTG
C
OTHERWISE PRINT
C
REAL CON(16)
C
NAMESI/MT1/1
C
DATA AB1/47.
C
1.55, S2, 51.55
C
WRITE(4,2)
C
FORMAT(111)
C
ICH=0
C
KCOUNT=0
C
REAL(5.1C1)
C
Determine CC
C
S = 0.
C
GO TO 11
C
IF(CON(5))=1CC,
11
CCN(5)=1CC
C
331 FCCJ=-APCTR(14)/QM
C
APCTR(14)=C.
C
Z KC
C
340 IF(APCTR(14).EQ.0.) GC TO 350
C
APCTR(14)=APCTR(14)-CM*HOLD1
C
341 FCCJ=-APCTR(14)/QM
C
APCTR(14)=C.
C
VC
C
350 IF(APCTR(8).EQ.0.) GC TO 391
C
APCTR(8)=APCTR(8)-QM*HOLD1
C
351 FCCJ=-APCTR(8)/QM
C
APCTR(8)=C.
C
CL TO 391
C
390 CONTINUE
C
391 ATEMPR=ATEMPR-07*(HCLUD-HCLC1)
C
392 FCCJ=ATEMPR
C
393 IF(APCTR(10).EQ.0.) GC TO 394
C
APCTR(10)=APCTR(10)-HCLUD1
C
394 IF(APCTR(11).EQ.0.) GC TO 396
C
APCTR(11)=APCTR(11)-HCLUD1
C
395 FCCJ=APCTR(11)
C
396 IF(APCTR(11).EQ.0.) GC TO 398
C
APCTR(11)=APCTR(11)-HCLUD1
C
397 IF(APCTR(12).EQ.0.) GC TO 399
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C
399 FCCJ=APCTR(14)
C
400 IF(APCTR(14).EQ.0.) GC TO 405
C
APCTR(14)=APCTR(14)-HCLUD1
C
401 FCCJ=APCTR(14)
C
405 IF(APCTR(14).EQ.0.) GC TO 406
C
APCTR(14)=APCTR(14)-HCLUD1
C
406 CONTINUE
C
407 IF(HCLUD.GT.C.) HCLUD=HCLUD+HCLUD1
C
408 IF(ITCH.EQ.0.) GO TO 431
C
409 CONTINUE
C
410 IF(APCTR(1).LE.3.48*(APCTR(1)+4*APCTR(9)) ITCH=1
C
411 IF(APCTR(4).GT.3.48*(APCTR(7)+4*APCTR(9)) ITCH=2
```
IF(ITCH.EQ.1) GO TO 841
IF(ITCH.EQ.2) GO TO 836

C M?3CE
E36 APCTR(4)=APCTR(4)-21.*HCLD/6.
IF(APCTR(4).LE.0.) GO TO 837,838

E37 APCTR(4)=0.
E38 ATEMPK=ATEMPK-2.*HCLD/6.
IF(ATEMPK).LE.E35,E35,E46
E39 APCTR(4)=APCTR(4)+ATEMPK
ATEMPK=0.
E40 GO TO 846

C
C CONTINUE
E41 APCTR(5)=APCTR(5)-2.5*HCLD
IF(APCTR(5).GT.0.) GO TO 841
APCTR(5)=APCTR(5)-HCLD

E41 APCTR(6)=APCTR(6)-HCLD
APCTR(4)=APCTR(4)-.5*HCLD
ATEMPK=ATEMPK-2.*HCLD
IF(APCTR(5).LT.0.) APCTR(5)=0.
IF(APCTR(6).LT.0.) APCTR(6)=0.
IF(APCTR(4).LT.0.) APCTR(4)=0.
IF(ATEMPK).LT.0.) ATEMPK=0.
E46 CONTINUE

C
C CONTINUE
E47 APCTR(7)=ATEMPK*F7
APCTR(9)=ATEMPK*F9

C GAMMA CALCULATION
S1=APCTR(1)+APCTR(2)+APCTR(4)+APCTR(5)+APCTR(6)+APCTR(7)+APCTR(8)+
1APCTR(9)+APCTR(10)+APCTR(11)+APCTR(12)+APCTR(14)+APCTR(15)+
2APCTR(16)
1=APCTR(5)+APCTR(6)+APCTR(12)
K5=APCTR(15)/T
K6=APCTR(16)/T
R12=APCTR(12)/T

X=3.3
Z=APCTR(1)+APCTR(10)+APCTR(11)+APCTR(14)+APCTR(16)+.5*APCTR(8)
APCTR(8)=.5*APCTR(8)
APCTR(5)=APCTR(5)-K5*X*Z
APCTR(6)=APCTR(6)-R6*X*Z
APCTR(12)=APCTR(12)-K12*X*Z
S2=APCTR(2)+APCTR(5)+APCTR(6)+APCTR(7)+APCTR(9)+APCTR(12)+APCTR(4)
C U0=W IN GAMMA
C W=W IN GAMMA
C A0=A IN GAMMA
C Y=W IN GAMMA
Y=APCTR(4)/(S2)/(.4*(.27-APCTR(2)/S2)-.115*APCTR(4)/S2)
W=.2*APCTR(9)/S2/(1.2*(.27-APCTR(2)/S2)-.115*APCTR(9)/S2)
UL=.2*(.155*APCTR(7)/S2)/(.2*(.27-APCTR(2)/S2)-.115*APCTR(7)/S2)
GO TO (140C,150C),MODE
140C CONTINUE

C LINEAR SOLUTION
AL=.115*(.6*(.27-APCTR(12)/S2)-.27*(APCTR(4)+APCTR(7)+APCTR(9))/S2)
Continued from previous page:

14

0

REFERENCES


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<td>23.4</td>
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\(^{a}\)Calculated from ref. 4 but not corrected for carbides and borides.
TABLE II. - MELT CHEMISTRY

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<th>V</th>
<th>Nb + Ta&lt;sup&gt;c&lt;/sup&gt;</th>
<th>C</th>
<th>B</th>
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<sup>a</sup>Ref. 5.

<sup>b</sup>Ref. 4.

<sup>c</sup>For calculations Nb + Ta is assumed to be Nb.
Figure 1
Nickel rich corner of the Ni-Al-Cr system at 750°C
After Taylor and Floyd Ref. 6