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UNIVERSITY OF CALIFORNIA
INSTITUTE OF ENGINEERING RESEARCH
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EFFECT OF SOME SOLID SOLUTION ALLOYING ELEMENTS
ON THE CREEP PARAMETERS OF NICKEL

Tenth Technical Report

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T. H. Haslett
V. F. Zackay
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INTRODUCTION

The present research program was undertaken to provide reliable experimental creep data which might be used to provide a foundation for the development of a sound theory of creep of metals. When this program was first conceived, it was believed that the so called "secondary creep rate" could be used as the parameter to compare the effectiveness of various alloy additions. However, as pointed out by Lubahn ⁽¹⁾, under conditions of constant stress loading, the slope of the creep curve is continuously changing and no true constant creep rate is obtained. This concept of a continuously changing creep rate was amplified by Hollomon and Fisher ⁽²⁾ who found that the creep curves for a number of metals, tested under conditions of constant stress, obey a simple relationship between strain and time. Recently, the present authors proposed a more general relationship between strain and time which fits all constant stress creep data investigated to date ⁽³⁾. This relationship may be expressed as

$$\epsilon - \epsilon_0 = A t^b \quad (1)$$

where

ϵ = Total true strain

ϵ_0 = Instantaneous strain at moment of loading

t = Time

A, b = Constants

Thus, the quantity $(\epsilon - \epsilon_0)$ may be considered as being the time dependent strain. This yields a linear relation when $\log (\epsilon - \epsilon_0)$ is plotted against $\log t$.

It has been found that the quantities A , b and ϵ_0 vary with applied

stress, temperature, structure, and alloy content. The present investigation has been concerned with the influence of alloying upon these creep parameters.

MATERIALS AND CONDITIONS

Nickel of 99.95 percent purity was used as the base metal for this investigation. The materials were refined, cast and reduced to a wrought condition in our own laboratory as described previously.⁽⁴⁾ Alloy studies to date have been confined to the four systems having the compositions given in Table I. All alloy additions were well within the solid solubility limits.

TABLE I
COMPOSITION OF NICKEL ALLOYS

<u>At % Fe*</u>	<u>At % Co*</u>	<u>At % Ti**</u>	<u>At % W **</u>
2.55	2.43	0.25	0.5
4.94	4.60	1.00	1.0
9.77	8.69	2.00	

* Determined by chemical analysis.

** Determined by spectrographic analysis.

Elevated temperature creep tests were performed in single specimen creep units similar to those shown in Fig. 1. These units were mounted independently of each other to prevent any possibility of shock loading due to failure of another specimen. The load was applied through a knife edge lever arm system but, since the ultimate goal of the study was to provide data which would be of value for theoretical studies, all tests were performed under conditions of essentially constant stress rather than constant load. Stress variation was held to less than $\frac{1}{2}\%$ by means of a modified lever arm⁽⁵⁾, which consisted of an auxiliary bar attached to the

FIG. 1 SINGLE SPECIMEN CREEP STANDS

back end of the lever and projecting downward from it at an angle of 90°. The levers were counter-balanced and calibrated for several load ranges to correct for possible bending.

Tube type wire wound resistance furnaces with three independent windings were used for heating the test specimens. Each end winding was individually controlled by an auxiliary controller while the temperature of the center winding was determined and controlled by a thermocouple placed at the center shoulder of the specimen. High sensitivity millivolt controllers with an expanded range were used for this purpose. Temperature distribution along the specimens was held within $\pm \frac{1}{2}^{\circ}\text{C}$ and the temperatures of the center of the specimens did not vary more than 1°C during the test.

The test specimens were machined with two reduced sections in tandem - one having a uniform reduced length of one inch and the other three inches. Strain measurements were made by means of telescoping rod and tube extensometers pinned to each shoulder of the specimen and free to move in the direction of the specimen axis, thereby permitting the elongation of each reduced section to be measured. The difference in elongation of the two reduced sections was taken as the elongation in two inches. Strains were measured with precision dial gauges which were modified to permit automatic recording of all data.

Precision Lattice Constants: It has been established that the lattice strain caused by the difference in the atomic radii of the host and foreign atoms is one factor which affects the amount of solid solution hardening.⁽⁶⁾ While no exact method has yet been devised for determining local lattice strains, it may be expected that in binary alloys these are related in some simple manner to the mean lattice strain which can be measured with a high degree of precision.

Lattice parameters of the various alloys were determined using

a symmetrical precision focussing camera with an X-ray beam of nickel-filtered radiation from a copper target source powered with an XRD unit. A photodensitometer trace of the film was then made and the distance between intensity peaks measured on a Bausch & Lomb Profilometer to the precision of 0.0001 inches. Corrections for uniform film shrinkage were made by utilizing calibration lines printed on all films prior to their photographic development. Extrapolation to a Bragg angle of 90° was accomplished by use of the method described by Nelson & Riley⁽⁷⁾. All lattice constants were corrected to a standard temperature of 25°C , using the value of the linear coefficient of expansion for each alloy.

EXPERIMENTAL RESULTS

Lattice parameters for each of the alloy systems are shown in Fig. 2. It may be noted in all cases, the lattice parameter a_0 varies linearly with alloy content.

An indication of the reproducibility of creep test results obtainable on the pure nickel base material prepared from four different ingots is illustrated in Fig. 3. It may be readily seen that the scatter band is quite small. This reproducibility is further illustrated in the log-log plot of the same data in Fig. 4 and by the values of A , b and E_0 listed in Table II. The creep rates of these four specimens did not deviate more than approximately 10% from the average, a figure which is generally considered excellent for such tests.

Conventional rectangular coordinate creep curves of each of the four solid solution alloy systems are shown in Figs. 5 to 8, and the logarithmic plots are given in Figs. 9 to 12. It may be seen that all

FIG. 2 EFFECT OF ALLOYING ELEMENTS ON THE LATTICE CONSTANT OF NICKEL AT 25°C

FIG. 3 REPRODUCIBILITY OF NICKEL CREEP CURVES:
TEMPERATURE 700 °C, STRESS 5750 PSI.

FIG. 4 REPRODUCIBILITY OF NICKEL CREEP CURVES:
TEMPERATURE 700 °C, STRESS 5750 PSI.

FIG. 5 CREEP CURVES OF NICKEL-COBALT ALLOYS:
TEMPERATURE 700°C, STRESS 5750 PSI.

FIG. 6 CREEP CURVES OF NICKEL - IRON ALLOYS:
TEMPERATURE 700 °C, STRESS 5750 PSI.

FIG. 7 CREEP CURVES OF NICKEL-TITANIUM ALLOYS:
TEMPERATURE 700°C, STRESS 5750 PSI.

FIG. 8 CREEP CURVES OF NICKEL - TUNGSTEN ALLOYS:
TEMPERATURE 700 °C, STRESS 5750 PSI.

FIG. 9 CREEP CURVES OF NICKEL-COBALT ALLOYS:
TEMPERATURE 700 °C, STRESS 5750 PSI.

FIG. 10 CREEP CURVES OF NICKEL - IRON ALLOYS:
TEMPERATURE 700 °C, STRESS 5750 PSI.

FIG. II TIME DEPENDENT STRAIN CREEP CURVES FOR NICKEL-TITANIUM ALLOYS: TEMPERATURE 700°C, STRESS 5750 PSI

FIG. 12 CREEP CURVES OF NICKEL-TUNGSTEN ALLOYS:
TEMPERATURE 700 °C, STRESS 5750 PSI.

the curves on the logarithmic plots are essentially linear when time dependent strain is plotted against time.

TABLE II
CREEP PARAMETERS FOR PURE NICKEL
Stress = 5750PSI Temp. = 700°C

Ingot	<u>A</u>	<u>b</u>	<u>ϵ_o</u>
a	.017	.36	.0098
b	.018	.35	.0075
c	.016	.36	.0109
d	.020	.35	.0085

The addition of cobalt up to 8.7 atomic percent has no detectable effect on the creep strength of pure nickel as shown by the data in Figs. 5 and 9; the alloy curves cluster about the pure nickel line in a random manner.

On the other hand, the nickel-iron alloys, shown in Figs. 6 and 10, reveal a significant increase in creep resistance as the alloy content is increased. This is reflected in Fig. 10 by the increasing values of ϵ_o and b, while A decreases with increasing alloy content. Additions of tungsten (Figs. 8 and 12) and titanium (Figs. 7 and 11) increase the creep resistance of pure nickel even more, and the parameters ϵ_o , b and A vary with alloy content in the same manner, but with different magnitudes, than for the nickel-iron alloys.

DISCUSSION OF RESULTS

Examination of Fig. 5 reveals considerable deviation of the nickel-cobalt data from that for pure nickel. Moreover, as may be noted from both Figs. 5 and 9, this deviation is not consistent with alloy content, and is a great deal larger than the normal scatter obtained with duplicate pure nickel ingots as shown in Figs. 3 and 4. Although the nickel-cobalt alloys were somewhat higher in trace impurities than the other systems tested, the impurities increase with increasing cobalt content so that the observed scatter in the creep data does not appear to be due to uncontrolled variations in composition. This is further verified by the self consistent lattice parameters shown in Fig. 2. Also, the grain sizes are very nearly the same for all compositions. It may be seen from Figs. 5 and 9, as well as subsequent correlation plots, that the data from the 2.4 atomic percent cobalt alloy is inconsistent with the other results. Consequently, the data from this ingot will be disregarded in the ensuing discussion. It may be concluded, however, from the data obtained on the other two compositions that the addition of cobalt to nickel has but slight effect on the creep strength of the base material.

Using the formulation $\epsilon - \epsilon_0 = At^b$ to describe the creep curves, the values of the three parameters involved are given in Table III. For comparative purposes, the creep rate for time = 10 hours is also listed. The latter were computed from the creep parameters using the following expression:

$$\dot{\epsilon} = \frac{d\epsilon}{dt} = bAt^{b-1} = \frac{b(\epsilon - \epsilon_0)}{t} \quad (2)$$

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TABLE III
CREEP PARAMETERS AND CREEP RATES FOR
SEVERAL NICKEL ALLOYS

Alloy (Atomic Percent)	b	Creep Parameters A	ϵ_0	Creep Rate $\dot{\epsilon} = 10$ hrs (in/in/1000 hrs)
Pure Ni	0.362	0.0168	0.0098	1.58
2.43% Co	0.428	0.0187	0.0114	2.07
4.60% Co	0.371	0.0155	0.0092	1.33
8.69% Co	0.399	0.0146	0.0101	1.45
2.55% Fe	0.449	0.0123	0.0084	1.56
4.94% Fe	0.499	0.0089	0.0077	1.38
9.77% Fe	0.614	0.0044	0.0052	1.14
0.25% Ti	0.418	0.0093	0.0090	1.06
1.00% Ti	0.668	0.0018	0.0053	0.56
2.00% Ti	0.870	0.00025	0.00205	0.16
0.50% W	0.422	0.0063	0.0087	0.71
1.00% W	0.466	0.0024	0.0065	0.36

The self consistency of the parameters b, A and ϵ_0 is illustrated in Figs. 13, 14 and 15. Figure 13 reveals that the slope b increases directly while Fig. 14 shows that A decreases exponentially with alloy additions. The values of ϵ_0 also decrease in a regular manner as the solid solution alloy content is increased. These relationships may be expressed mathematically as

$$A = A_0 e^{-a\gamma} \quad (3)$$

$$b = b_0 + c\gamma \quad (4)$$

$$\epsilon_0 = (\epsilon_0)_0 - f\gamma \quad (5)$$

where

γ = atomic percent solid solution alloy addition

a, c, f = constants characteristic of the alloying element

$A_0, b_0, (\epsilon_0)_0$ = values of the parameters for pure nickel

Examination of Equation (2) reveals that the creep rate at any given time is a function of both parameters, A and b. For a constant value of A, the creep rate decreases as b decreases. The same is true for decreasing A if b is constant. However, these two parameters change at quite different rates as indicated by Eqs. (3) and (4) so that casual inspection of the individual values of these parameters does not necessarily reveal the true situation as to creep resistance. A better, but still not precise, index of creep resistance is the product (A x b).

Although several types of cross-plots such as the values of a, c and/or b (from Eq. 3, 4, and 5) vs. the melting point of the alloying addition can be made, the limited number of elements from any one long row

FIG. 13 VARIATION OF SLOPE OF CREEP CURVE WITH
SOLID SOLUTION ALLOY CONTENT

FIG. 14 EFFECT OF SOLID SOLUTION ALLOY CONTENT ON PARAMETER "A"

FIG. 15 VARIATION OF ϵ_0 WITH SOLID SOLUTION ALLOY CONTENT.

of the periodic table render the results obtained from this procedure open to question. However, the creep resistance of nickel does appear to increase as elements are added which have higher melting points. It is also impossible at the present time to relate the creep parameters to the lattice constants in any simple manner.

The results obtained to date indicate potential correlations between creep data and other material constants of alloys. However, many additional tests, as well as an extension of the number of alloy systems, will be required to clarify the situation. Studies of the recovery characteristics, room temperature tensile tests, and additional creep tests at various temperatures and stresses will be made on the alloys to further elucidate the role of solid solution alloying on the plastic properties of transition elements.

CONCLUSIONS

1. The formulation $\epsilon - \epsilon_0 = A t^b$ accurately describes the constant stress creep curves for Ni-Co, Ni-Fe, Ni-W, and Ni-Ti solid solution alloys as well as those for the metallurgically stable metals previously reported.

2. The three creep parameters A, b and ϵ_0 vary in a regular manner with solid solution alloy addition as follows:

- (a) The parameter A decreases exponentially as the alloy content increases.
- (b) The parameter b increases directly as the alloy content increases.
- (c) The parameter ϵ_0 decreases directly as the alloy content increases.

3. The data given indicate the possibility of correlations between creep parameters and other material constants. However, such correlations must await the gathering of data on additional alloys as well as an extension of the work on current materials.

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