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CREEP MECHANISMS IN CsCl (B2) INTERMEDIATE PHASES

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BY QH Kahn Lamb
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PRINCIPAL INVESTIGATOR

R. A. Dodd

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INTRODUCTION

Long-range order is known to strengthen a solid solution during creep^(e.g. 1,2), and so considerable interest attaches to alloys which retain order up to elevated temperatures. Among the B2 alloys, both NiAl and CoAl melt near 1650°C and are ordered up to their melting points. For these reasons, they have been studied quite extensively by other workers⁽³⁻⁷⁾, in addition to the work now reported under AT(11-1)-1489. A few other alloys such as beta brass have also been previously studied in considerable detail⁽¹⁾.

The specific aims of the present investigation were two-fold.

- (1) To attempt to elucidate the creep mechanisms in a wide variety of B2 alloys, and so to arrive at a possibly general model for creep in this compound class.
- (2) To understand the fundamental rules governing the operative slip systems in B2 alloys, and their orientation dependence.

These aims are considered independently in Parts I and II of this report.

PART I Creep in CsCl (B2) Alloy Phases

The following alloys have been studied using both single and polycrystals: NiAl corresponding to stoichiometric, nickel-rich and aluminum-rich (vacancy-defective) compositions; CoAl, FeAl, NiGa, AuZn, AuMg, and CuZn, all near stoichiometry.

The experimental techniques have been considered in several of the attached publications, and so need be outlined only briefly. They include:

1. Growth of single crystals from the melt using induction melting or, more recently, a graphite element high-temperature furnace.
2. Crystal orientation followed by either, electroshaping into tensile creep specimens, or spark machining into compressive creep specimens.
3. Creep testing, followed by creep data analysis and electron microscopy of creep specimens.

A. Creep Properties and Structure of NiAl

On account of its high melting point, and therefore its possible practical use as a base for creep-resistant alloys, NiAl has been studied quite extensively in several earlier publications⁽³⁻⁷⁾, in addition to publications 1, 2, 3 and 5 (see publication list) attached to this report, and which resulted from research under subject contract. Thus, some of the basic features of the creep behavior of NiAl are now well understood, and since they are documented in the attached publications they will not be further reviewed at this time. Instead, the more recent unpublished research contrasting the creep properties and associated structures of stoichiometric and non-stoichiometric NiAl will be discussed.

The wide solidification range of aluminum-rich NiAl made it impossible to grow single crystals of this material, so that the present data refer to chill-cast polycrystalline NiAl kindly supplied by the Pratt and Whitney Aircraft Company. The creep data are shown in Figures 1-6 in the form of log shear stress versus log shear strain rate. A factor of 0.5 was used

to convert the applied stress, F/A , to shear stress, while a factor of 0.75 was used to convert strain rate to shear strain rate. The following essential facts may be noted:

1. The steady state creep rates follow the relationship $\dot{\gamma} = K \tau^n$ for all alloys. The stress exponent, n , is constant throughout the temperature range for the Al-rich alloys, while the stoichiometric and Ni-rich alloys have temperature-dependent slopes at lower temperatures. Figure 7 shows the relationship between n and temperature for the various alloys.
2. At high temperatures, the steady state creep rate is represented by:

$$\dot{\gamma} = A' \tau^n \exp. (-Q_C/RT)$$

the values of n and Q_C for the different alloys being listed below.

<u>Composition, at. % Ni</u>	<u>n</u>	<u>Q_C, K cal mole⁻¹</u>	<u>T, °K</u>
55	3.6	65	1073
51	4.7	73	1123
50	4.6	68	1223
49	4.5	64	973
48	3.7	55	1023
46	3.5	70	1073

The apparent activation energies are close to the self diffusion activation energies.

3. At lower temperatures, the equation of steady state creep for stoichiometric and Ni-rich alloys becomes

$$\dot{\gamma} = \gamma_0 (\tau/\tau_0)^{n(T)}$$

in which n is now temperature-dependent, the values of n being given in Figure 7. The apparent activation energies are now both stress and temperature-dependent, and it appears doubtful that only one mechanism is rate-controlling in these alloys at lower temperatures.

The substructural features observed in stoichiometric NiAl after steady-state creep can be summarized as follows:

- (a) Below 1123°K (850°C) sub-boundaries are rarely observed. Instead, there exists an evenly distributed high density of jogged dislocations, dipoles and loops (Figure 8a).
- (b) Sub-boundaries are frequently observed at 850°C, although the lower temperature features are still apparent (Figure 8b).
- (c) Sub-boundaries become dominant when the alloy is deformed above 1173°K (900°C), as in Figure 9a.
- (d) At $\geq$ 1318°K (1045°C) the sub-boundaries take the form of regular large-mesh networks, while large loops are often observed simultaneously (Figure 9b).
- (e) Geometrically shaped voids are observed in specimens deformed at and below 1123°K (850°C) but never in specimens deformed above 1173°K (900°C). Figure 10 shows typical examples.
- (f) The creep behavior of stoichiometric NiAl is highly erratic below $\sim$ 800°C, the creep rate often being abnormally rapid compared with specimens deformed at higher temperatures under the same stress. This effect will be discussed later.

Structurally, the 51 at. % Ni alloy behaves similarly to stoichiometric NiAl except that voids are now observed at all temperatures, and the density of voids is considerably higher. In the case of NiAl containing 55 at. % Ni, no sub-boundaries were ever observed at any testing temperature, and the void density was always low.

In many respects, Al-rich NiAl, even though vacancy-defective, behaves in a simpler and more consistent manner than do the stoichiometric and Ni-rich alloys. Voids are now observed only in the 51 at. % Al alloy, and they have a low density compared with the stoichiometric and Ni-rich alloys. Dislocation loops, dipoles and jogs are commonly observed in the deformed 51 at. % Al alloy (Figure 11a), while sub-boundary networks are a usual feature of the 52 at. % Al alloy.

Transmission electron microscopy of the Al-rich alloy containing 54 at. % Al has revealed the following features:

1. Stacking faults are observed in most specimens.
2. The higher the creep temperature the more uniformly the faults are distributed.
3. The density of faults decreases as the creep rate increases, and faults are rarely observed in the samples deformed at shear strains exceeding 10^{-5} sec^{-1} .
4. The faulted dislocations are frequently arranged in tilt boundaries in annealed uncrept specimens.

Contrast analysis showed that the fault vector, $\bar{R}$, is normal to the cube plane, and that the Burgers vectors of the terminal dislocations are $\langle 100 \rangle$ and are identical to the displacement vectors of the fault. These observations are identical to Ball's findings⁽⁸⁾, and when considered along with features 1-3 above support the concept of a clustered form of structural defects in Al-rich NiAl. That is, the faults observed do not result from the dissociation of dislocations during plastic deformation. Finally, the stable form of the faults in an annealed specimen is such that the faults are concentrated into tilt boundaries as in Figure 11b. In terms of Ball's model of the defect structure, this implies that the excess aluminum is also concentrated at the sub-boundaries, and this may well cause local embrittlement leading to the "pest" phenomenon. Westbrook⁽⁹⁾ has discussed pest in NiAl in terms of grain boundary embrittlement by oxygen and/or nitrogen, but the main objection to this suggestion is that the pest occurs even in single crystals. Furthermore, Westbrook⁽⁹⁾ observed that pest in NiAl is more prevalent in the aluminum-rich alloys.

B. Point Defects and Abnormal Properties of Stoichiometric NiAl

In the temperature range of $\sim 750-850^\circ\text{C}$ the steady state creep rate of stoichiometric NiAl is frequently abnormally rapid. As already noted, voids form in this temperature range but it seems unlikely that they are the cause of the rapid creep rate since dislocations pinned by voids are frequently seen in crept samples. That is, the likely effect of voids is a strengthening effect.

Interesting evidence has been obtained from transmission electron microscopy of specimens annealed in the range 700-900°C. Etch pits repeatedly appeared on the polished thin foil surface, examples being shown in Figure 12. Proof of the existence of pits was obtained using optical microscopy at high magnification. In fact, the pits originate from small loops and helices (Figure 12), with the density of loops being $\sim 2 \times 10^9 \text{ cm}^{-2}$. On the other hand, the dislocation density of NiAl annealed at 1300°C is only $\sim 10^5 \text{ cm}^{-2}$.

The appearance of this large number of loops is believed to be due to the condensation of thermal vacancies on to impurity atoms, and subsequent collapse of vacancy aggregates into loops. The accelerated creep is evidently due to the fact that the loops actually operate as dislocation sources during plastic deformation. To further investigate this hypothesis, two groups of creep specimens were annealed at different temperatures; one group was annealed at 1300°C and the other at 900°C. The annealing time was 48 hr. for both groups and both were furnace cooled. Figure 13 shows the results of creep tests at 800°C, and it is seen that specimens annealed at 900°C have faster creep rates. Also, Figure 14 shows that the specimens annealed at 900°C have a much higher dislocation density despite the lower creep stress.

Another two groups of samples annealed at 1300°C and 900°C respectively were compression tested at room temperature, the results being shown in Figure 15. Specimens annealed at 900°C are markedly more ductile than

those annealed at 1300°C, and do not have an abrupt change between elastic and plastic deformation. The evidence seems overwhelming in favor of the hypothesis that dislocation loops form in the range 700-900°C, and act as dislocation sources in subsequent plastic deformation.

C. Creep Properties and Structure in Stoichiometric NiGa

A substantial amount of information has been obtained on the creep properties and associated substructures of NiGa, and the similarity to NiAl is quite marked. The essential conclusions are:

1. Slip in NiGa occurs on $\{100\}$ and $\{110\}$ with $\bar{b} = \langle 100 \rangle$; cross slip between $\{100\}$ and $\{110\}$ results in dislocation dipoles and elongated loops.
2. For "hard" orientations near $\langle 001 \rangle$, $\{110\} \langle 110 \rangle$ slip systems have been observed to operate, but $\langle 111 \rangle$ Burgers vectors have not been observed.
3. The dislocation substructures corresponding to both low-temperature and high-temperature creep substantially resemble those observed in stoichiometric NiAl. An exception here is that neither voids nor small loops appear to form in NiGa. However, at this time it is not known whether this is an intrinsic property difference or related to impurity effects. Certainly there is no basis for assuming that the impurity species and amounts are similar in the two alloys.

4. The high temperature primary and steady state creep behavior of NiGa single crystals can be described by a universal creep curve. In the steady state region the stress exponent, n , is equal to 3, independent of crystal orientation.
5. On a tentative basis the rate controlling mechanism for steady state creep of NiGa single crystals may be the climb of edge dislocations and edge components of screw dislocations, together with the formation of tilt boundaries and their movement.

D. Creep Properties and Structure of FeAl, AuZn, and AuMg

Although creep data has been obtained for these three compounds, the structural studies have been more superficial than in the case of NiAl and NiGa. However, in the case of FeAl enough has been accomplished to say that this compound behaves similarly to NiAl and NiGa, viz. the primary Burgers vector is $\langle 100 \rangle$, and creep at lower temperatures produces dipoles and elongated loops. At higher temperatures, the sub-boundaries seem analogous to those in NiAl, but they have not been subject to detailed analysis.

A most interesting feature of the creep behavior of AuZn is that its steady-state creep rate at $\sim 300^\circ\text{C}$ exceeds the rate at $\sim 400^\circ\text{C}$. Presumably, an explanation of this behavior must be sought along the lines of that given for similar behavior in NiAl. That is, at $\sim 300^\circ\text{C}$ it is very likely that vacancies condense on to impurity atoms as a preliminary to forming dislocation loops; the loops then act as sources producing softening. However, electron microscopy has not yet been carried out on this alloy so that the necessary proof of the theory is lacking.

Part 2. Slip Systems in B2 Alloys

The fundamental rules governing the deformation behavior of the B2 alloys cannot yet be described with certainty. Following the initial work of Rachinger and Cottrell⁽¹⁰⁾, attempts have been made to predict slip systems on the basis of hard-sphere models⁽⁵⁾, anisotropic elasticity theory⁽⁷⁾, and the latter combined with calculations of ordering energies⁽¹¹⁾. Table 1 compares the slip systems actually observed in various B2 alloys with those predicted by the several models. It will be noted that the data for CoAl, FeAl and NiGa refers exclusively to work under subject contract.

To a certain extent, the agreement between observation and prediction is good. For instance, CuZn and FeCo have size factors favoring a $\langle 111 \rangle$ Burgers vector⁽⁵⁾, the energy of the antiphase boundaries separating the superlattice dislocations is low^(11, 14), and the dissociated superdislocation energy is low in the case of CuZn at least⁽¹¹⁾. Also, CuZn and FeCo are typical metallic phases with only a slight covalent tendency; they undergo order-disorder transformations at temperatures well below the melting points, and there is no bond directionality. Therefore, on all counts a $\langle 111 \rangle$ slip direction is expected and is always observed.

NiAl, CoAl, NiGa and FeAl have a strong covalent component in the bonding which results in some bond directionality. The first two have high heats of formation and high congruent melting points, while all remain ordered up to the melting point. The antiphase boundary energy in NiAl is high⁽¹¹⁾, the $a\langle 100 \rangle$ dislocations in NiAl have the lowest energy, and all four

compounds have size factors which are favorable for a $\langle 100 \rangle$ Burgers vector. In fact, this is the Burgers vector always observed except that $\langle 110 \rangle$ slip is possible when the orientation is unsuitable for cube and dodecahedral slip along $\langle 100 \rangle$ (Bevk, Dodd and Strutt, attached paper).

The situation is less clear in the case of the remaining alloys, AgMg, AuZn, and AuCd. All these alloys have congruent melting points ranging from 627°C for AuCd to 820°C for AgMg; they exist over similar composition ranges, are ordered to the melting point, and have quite similar heats of formation. There is no evidence for a strong covalent, i.e., directional, bond component, so that it may be tentatively concluded that the observed slip systems should be determined by the relative atom sizes. However, the latter would suggest the likelihood of a $\langle 100 \rangle$ Burgers vector in AgMg (and possibly AuCd) and a $\langle 111 \rangle$ Burgers vector in AuZn - precisely the opposite of what is actually observed. Potter⁽¹¹⁾ concluded that the possible dislocations in AuZn have similar energies, and that predictions are difficult to make. Similarly, Lawley⁽¹⁸⁾ suggested that AuZn and AuCd probably have ordering forces just large enough to cause $\langle 100 \rangle$ slip, while AgMg is a borderline case.

Evidently, accurate predictions of slip systems in ordered alloys are not yet possible, although it is obvious that the hard sphere model utilizing the concept of relative atomic sizes is an unreliable guide. Other factors such as antiphase boundary energies could not be thoroughly studied with the various alloys used in the research under AT(11-1)-1489 so that further work on other alloy systems is necessary to permit reliable predictions.

Table I

Comparison of Actual and Theoretical Slip Systems for Some B2 Intermediate Phases

Alloy	Atom Size Ratios R_A/R_B	Observed Slip System(s) and References	Hard Sphere Prediction (Lautenschlager et al, Ref. 5)	Anisotropic Elasticity Prediction (Ball and Smallman, Ref. 15)	Anisotropic Elasticity and Ordering Energy Prediction (Potter, Ref. 16)	Relevant Alloy Properties (ΔH_f in cal/mole.)
CuZn	0.911	$\langle 111 \rangle \{110\} \{112\} \{123\}$ Refs. 10, 12, 13	$\langle 111 \rangle \{110\}$	$\langle 111 \rangle \{110\}$	$\langle 111 \rangle \{110\}$	M.pt 875°C. Order-disorder transformation at 468°C. $\Delta H_f = -2135$
FeCo	0.989	$\langle 111 \rangle \{110\}$ Refs. 14 and 15	$\langle 111 \rangle \{110\}$	--	--	M.pt 1480°C. Order-disorder transformation at $\sim 730^\circ\text{C}$. $\Delta H_f = -2270$ at 870°C
AgMg	0.844	$\langle 111 \rangle \{110\} \{112\} \{123\}$ Refs. 10 and 16	$\langle 100 \rangle \{100\}$ or $\langle 100 \rangle \{110\}$ or $\langle 111 \rangle \{110\}$	--	--	Ordered up to the M.pt of 820°C. This phase has a very wide composition range of stability. $\Delta H_f = -4600$ at 500°C.
AuZn and AuCd	AuZn 0.903 AuCd 0.379	$\langle 001 \rangle \{110\}$ Ref. 10	$\langle 100 \rangle \{100\}$ or $\langle 100 \rangle \{110\}$ or $\langle 111 \rangle \{110\}$	--	$\langle 100 \rangle \{110\}$	AuZn and AuCd melt at 725°C and 627°C respectively, and both are ordered up to the M.pt. ΔH_f (AuZn) = -5500 at 20°C. ΔH_f (AuCd) = -4650 at 25°C.
NiAl	0.855	$\langle 001 \rangle \{001\}$ $\langle 001 \rangle \{011\}$ $\langle 011 \rangle \{011\}$ Refs. 3, 5, 17 and this report.	$\langle 100 \rangle \{100\}$ or $\langle 100 \rangle \{110\}$	$\langle 100 \rangle \{110\}$	$\langle 100 \rangle \{110\}$	Ordered up to the M. pt of 1638°C. $\Delta H_f = -14050$ at 25°C.
CoAl	0.852	$\langle 001 \rangle \{001\}$ $\langle 001 \rangle \{011\}$ This report.	$\langle 100 \rangle \{100\}$ or $\langle 100 \rangle \{110\}$	--	--	Ordered up to the M. pt of 1645°C. $\Delta H_f = -13200$ at 25°C.
FeAl and NiGa	FeAl 0.847 NiGa 0.882	$\langle 001 \rangle \{001\}$ This report.	$\langle 100 \rangle \{100\}$ or $\langle 100 \rangle \{110\}$	--	--	M. pt of FeAl 1350°C M. pt of NiGa 1175°C No order-disorder trans ΔH_f (FeAl) = -6070 at 25°C.

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Publications Resulting From Research Performed
Under AT(11-1)-1489.

1. "Nature of Slip Line and Substructure Formation During Creep in Stoichiometric NiAl at Temperatures Between 475 and 775°C," W. R. Kanne, P. R. Strutt and R. A. Dodd, Trans. AIME, 1969, vol. 245, p. 1259.
2. "Creep in Ordered Alloys," Chapter in: "Ordered Alloys: Structural Applications and Physical Metallurgy," P. R. Strutt and R. A. Dodd, Claitor's Publishing Division, 1970, p. 475.
3. "Comparison of Steady-State Compression Creep Behavior in Stoichiometric CoAl and NiAl Single Crystals Between 850 and 1050°C ($0.69 > T/T_m > 0.58$)," L. Hocking, P. R. Strutt and R. A. Dodd, Jnl. Inst. Metals, 1971, vol. 99, p. 98.
4. "Structure of Beta Brass Single Crystals After Steady-State Creep," A. Lazaridis and R. A. Dodd, Met. Trans., 1970, vol. 1, p. 1081.
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Publications in Preparation Resulting from Research Performed
under AT(11-1)-1489.

1. "Orientation Dependence of Deformation Mode and Structure in Stoichiometric NiAl Single Crystals Deformed in High Temperature Steady State Creep," J. Bevk, R. A. Dodd and P. R. Strutt.
Note: This is submitted as a prepublication in the present report.
2. "Creep Properties, Mechanisms, and Associated Microstructures in Stoichiometric and Non-Stoichiometric NiAl," W. Yang and R. A. Dodd.
3. A general review of creep and slip systems in the several CsCl phases investigated. These are: NiAl, CoAl, FeAl, NiGa, AuZn and AuMg. The last four alloys have not so far been the subject of discussion of any paper resulting from research under AT(11-1)-1489. However, it appears that they will be most expeditiously treated in a single general review paper.

FIGURE 1 - Log Shear Strain Rate vs Log Shear Stress for Polycrystalline Stoichiometric NiAl Deformed in Steady State Creep.

FIGURE 2 - Log Shear Strain Rate vs Log Shear Stress for 55 at.% Ni - 45 at.% Al Deformed in Steady State Creep.

FIGURE 3 - Log Shear Strain Rate vs Log Shear Stress for 51 at.% Ni - 49 at.% Al Deformed in Steady State Creep.

FIGURE 4 - Log Shear Strain Rate vs Log Shear Stress for 49 at.% Ni - 51 at.% Al Deformed in Steady State Creep.

FIGURE 5 - Log Shear Strain Rate vs Log Shear Stress for 48 at.% Ni - 52 at.% Al Deformed in Steady State Creep.

FIGURE 6 - Log Shear Strain Rate vs Log Shear Stress for 46 at.% Ni - 54 at.% Al Deformed in Steady State Creep.

FIGURE 7 - Stress Exponent, n , versus Temperature for all NiAl Compositions.

FIGURE 8

Structure of Stoichiometric NiAl After Creep at (a) 790°C and (b) 850°C.

(a)

X 10,800

T = 790°C

$\tau = 3.8 \text{ kg. mm}^{-2}$

$\dot{\gamma} = 3.53 \times 10^{-7} \text{ sec}^{-1}$

$\epsilon = 3.43\%$

(b)

X 13,500

T = 850°C

$\tau = 4.22 \text{ kg. mm}^{-2}$

$\dot{\gamma} = 1.65 \times 10^{-5} \text{ sec}^{-1}$

$\epsilon = 4.06\%$

FIGURE 9

Structure of Stoichiometric NiAl After Steady-State Creep at $T \geq 900^\circ\text{C}$.

(a)

X 10,800

$T = 900^\circ\text{C}$

$\tau = 2.39 \text{ kg. mm}^{-2}$

$\dot{\gamma} = 1.55 \times 10^{-6} \text{ sec}^{-1}$

$\epsilon = 4\%$

(b)

X 12,000

$T = 1045^\circ\text{C}$

$\tau = 1.96 \text{ kg. mm}^{-2}$

$\dot{\gamma} = 3.0 \times 10^{-5} \text{ sec}^{-1}$

$\epsilon = 5.5\%$

FIGURE 10

Voids in Stoichiometric NiAl Crept at 800°C X 15,000

FIGURE 11

(a)

51 at.% Al 49 at.% Ni

$T = 800^{\circ}\text{C}$

$\tau = 3.51 \text{ kg. mm}^{-2}$

$\dot{\gamma} = 7.49 \times 10^{-6} \text{ sec}^{-1}$

$\epsilon = 2.9\%$

$\times 10,800$

(b)

54 at.% Al 46 at.% Ni

Concentration of Stacking Faults
into Sub-boundaries as a result
of annealing at 900°C .

$\times 48,000$

FIGURE 12

Structure of Stoichiometric NiAl After Quenching from 870°C X 8,000.

(a)

Surface pits.

(b)

Pits, loops and helices.

Fig. 13

FIGURE 14

Dislocation Configurations and Densities in Stoichiometric NiAl Pre-annealed at Different Temperatures X 14,000.

(a)

Pre-annealed at 1300°C for 48 hr.,
then crept at 810°C.

$$\tau = 3.8 \text{ kg. mm}^{-2}$$

$$\dot{\gamma} = 7.03 \times 10^{-8} \text{ sec}^{-1}$$

$$\epsilon = 3.51\%$$

(b)

Pre-annealed at 950°C for 48 hrs.,
then crept at 800°C.

$$\tau = 2.46 \text{ kg. mm}^{-2}$$

$$\dot{\gamma} = 4.22 \times 10^{-7} \text{ sec}^{-1}$$

$$\epsilon = 2.0\%$$

Fig. 15

The stress-strain curve of stoichiometric NiAl compressed at room temperature. S051 & S052 are pre-annealed at 1573°K for 72 hours. S053 & S054 are pre-annealed at 1178°K for 72 hours.