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Compressive Creep Behavior of Alloys Based on B2 FeAl

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ABSTRACT

Alloys based on FeAl are attractive as alternate materials for environmental resistance at intermediate temperatures. Addition of small amounts of Nb, Hf, Ta, Mo, Zr and B have been shown to improve the compressive creep of this alloy at 1100 K. Boron, in particular, has been found to have a synergistic effect along with Zr in providing properties substantially better than the binary alloy. This improvement seems to be related to the higher activation energy found for this alloy, suggesting a modification in the diffusion behavior due to the alloying additions.

I. INTRODUCTION

The intermetallic compound FeAl is of interest as an alternate material for intermediate temperature (800-1100 K) applications [1,2]. The low density, the B2 crystal structure stable up to its melting point, and the potential for excellent oxidation resistance are the main advantages of FeAl. Alloys based on this compound are, hence, particularly appealing as alternate materials for environmental resistance. Of particular importance, however, is the absence of strategic elements. The major problems which limit its usage at present are the poor room temperature ductility and the inadequate creep strength at the temperatures of commercial interest. In addition, this material is difficult to process using the conventional melting and casting techniques. However P/M (Powder Metallurgy) can be successfully used to produce a fully dense homogeneous product using hot extrusion. Research is currently underway using powder metallurgy to produce favourable microstructures with microalloying additions for enhanced creep resistance.

The basic variables for microstructural control in this intermetallic compound are the stoichiometry, thermomechanical treatment and alloying. Results of recent studies at Case Western Reserve University and NASA Lewis Research Center [2-7] are summarized below:

1) Hot extrusion of prealloyed powders produces an equiaxed recrystallized structure, the final grain size being a function of the extrusion temperature and the reduction ratio. Very little grain coarsening is observed by further heat treatment at temperatures of interest [2,3].

2) Although the binary stoichiometric (50 at% Al) FeAl alloy is brittle in tension at room temperature, a reasonable amount of room temperature tensile elongation can be obtained in the hypo-stoichiometric alloy (40 at%Al) [4,5].

3) Small amounts of Nb, Mo, Ta, Zr and Hf show promising improvements in compressive creep resistance at 1100 K [2,6].

4) Small additions of boron have dramatic effects in improving the creep properties as well as in modifying room temperature tensile behaviour [4,6,7].

These initial conclusions led to a more detailed investigation of the effects of alloying additions and in particular, boron on the compressive creep bahavior of FeAl. This paper summarizes the results from this investigation which was designed with the following objectives:

a) To evaluate the effects of small amounts of Nb, Hf, Mo, Ta and Zr alloying on creep behaviour of Fe-40at% Al with special emphasis on the possible synergestic effects between these alloying additions and small additions of boron.

b) To identify the nature of the creep deformation mechanisms in these alloys over a range of stresses and strain rates at 1100 K.

c) To understand the role of alloying elements in some of the better alloys by evaluating activation energies for the rate controlling deformation process at 1100 K.

d) To compare the creep strengths of these FeAl alloys with some commercial alloys in use at the temperatures of interest.

II. MATERIALS AND PROCEDURE

The basic design consists of alloys with and without boron. Nb, Hf, Ta and Mo at 1 at% level and Zr at 0.1at% level were each prealloyed with Fe-40at% Al and obtained in the form of gas atomized powder (-80mesh) from Alloy Metals Inc., Troy, Michigan. About 500 gm of each of these powders were packed in mild steel cans of 76 mm dia. and 6 mm wall thickness. The cans were sealed in vacuum and hot extruded at 1250 K and 16:1 area reduction ratio in a vertical extrusion press. Details of the extrusion process conducted at NASA Lewis Research Center are described elsewhere [2].

A second series of alloys was prepared by blending each of the prealloyed powders with 0.1 wt% B powder. Extrusions were made in an identical manner as described above. Chemical compositions and designations of the alloys are provided in Table I.

The extruded rods were centerless ground to 6.25 mm dia and cylindrical test specimens, 12.5 mm in length, were cut parallel to the extrusion direction. Specimens were heat treated at 650 K for 2 hr before compression testing. Elevated temperature compression creep tests were performed at 1100 K in air using constant load creep machines. Tests were performed at stress levels ranging from 10 MPa to 70 MPa. Tensile tests of some of the alloys were performed at 1100 K on a standard Instron Testing machine.

Optical metallography was performed on the as-extruded as well as the

crept samples in both the longitudional and transverse sections. This class of intermetallics is very difficult to etch and after extensive trials warm freshly prepared 20% Nital with a few drops of HF was found to be most effective.

Limited transmission electron microscopy was performed on the Phillips 400 using extruded and deformed samples.

III. RESULTS AND DISCUSSION

A) Microstructures

A typical microstructure of an extruded specimen is shown in Figure 1. In view of the difficulties encountered in etching this material, polarized light is used to reveal the grain size clearly. The microstructure shows recrystallized, equiaxed grains. Furthermore, the extrusions contain a wide range of grain sizes (2 to 24 micrometers), probably due to the broad size range of the powder particles used. Microstructural examination revealed the presence of stringers aligned in the extrusion direction and are presumed to be oxide particles from the prior particle boundaries. Hardly any porosity is observed, suggesting that the powder extrusion process is successful in producing a fully dense material. It must be pointed out here that the binary Fe-40Al powder was contaminated at the source and this resulted in a significant amount of a Ta-rich grain boundary phase. However, it is believed that this contamination does not significantly alter the conclusions from this study.

Transmission electron microstructures reveal the presence of second phase particles in some of the alloys which have not yet been identified. Figure 2 illustrates the structure of the FeAlZrB alloy. Some of these particles are believed to be oxides from prior particle boundaries, whereas others are suspected to be compounds containing the alloying elements.

B) Compressive Creep Behavior

Among the alloying additions investigated in the prealloyed group of alloys, Nb, Ta and Mo produced a significant improvement in creep strength over the binary FeAl as illustrated in Figure 3. Additions of Hf and Zr only show modest improvements. The typical value of the power law exponent exhibited by the alloys in this stress range at 1100 K is about 5. The Nb alloy exhibits a stress exponent value of 1 at lower stress levels (<25MPa) and a change in exponent value to 5 at higher stresses in the range of 25 to 50MPa. This behaviour suggests that the creep mechanism at lower stress levels could be diffusional transport (either Nabarro Herring type or Coble type) and at higher stress levels (n value of 5) the creep mechanism changes to some type of dislocation motion control [8].

Addition of boron shows a dramatic improvement in the compressive creep strength for all of the FeAlX ternary alloys as well as for the binary FeAl as shown in Figure 4. The FeAlB, FeAlNbB, FeAlHfB, FeAlTaB and the FeAlMoB alloys are all stronger than their counter parts without boron. The most striking alloy, however, is the FeAlZrB alloy which demonstrates the greatest improvement in creep strength with the boron addition. This alloy clearly stands out as the most promising FeAl alloy investigated in this group. Many of the other boron-containing alloys, however, have comparable strengths at stresses less than 20MPa. The addition of boron, apart from improving the creep resistance also seems to have raised the stress range over which an

3

expoueut value of 1 is exhibited. Figure 4 clearly shows that all the alloys
exhibit a break in the slope with a value of stress expouent changing from
about 1 at lower stresses to arouud 5 at higher stresses, ouce agaiu
suggesting a change from a diffusioual trausport to a dislocatiou motiou
coutrolled mechauism with iucreasing stress level [8,9].

A direct comparisou of the FeAl, FeAlB, FeAlZr aud the FeAlZrB alloys
shown in Figure 5 emphasizes the improvemeuts caused by borou aud in
particular, the syuergestic improvemeuts obtaiued by the additiou of Zr aud B
together. Atleast au order of maguitude decrease in creep rate is obtaiued
for the FeAlZrB alloy compared with the biuary FeAl alloy.

The teusile streugths of some of these alloys show ouly a modest
iucrease in streugth with the alloyiug additious (Table II). The dramatic
improvemeut of time-depeudeut creep streugth, heuce, poiuts to a
rate-coutrolliug mechauism iuvolviug a time-depeudeut diffusiou processes,
siuce alloyiug additious cau iuflueuce diffusiou rates cousiderably.

C) Activatiou Euergy

The diffusioual creep mechauism of Nabarro-Herriug or Coble is expressed
by a geueral equatiou of the form (Bailey-Orowau Equatiou) [10]:

$$\dot{\varepsilon} = A\, \sigma\, (D_l^0 \text{ or } B_{gb}^0)\, \exp(-Q_L \text{ or } Q_{gb})\, (b/d)^P$$

where A is a coustaut, D_l^0 is the pre-expoueutial for lattice diffusiou, D_{gb}^0
is the pre-expoueutial for graiu bouudary diffusiou, d is the graiu size, σ
is the applied stress, $\dot{\varepsilon}$ is the steady state creep rate, Q_l is the
activatiou euergy for lattice diffusiou, Q_{gb} is the activatiou euergy for
graiu bouudary diffusiou, p is the graiu size depeudeut factor (2 for
Nabarro-Herriug mechauism aud 3 for Coble mechauism) aud b is burger's
vector.

The additiou of Zr aud B produced au improvemeut iu creep streugth iu
the diffusioual creep regiou of FeAl (graiu size was assumed to be coustaut
duriug creep based ou microscopy of tested samples). An examiuatiou of the
above equatiou shows that oue possibility for this improved creep resistauce
is au iucrease iu activatiou euergy for diffusiou. The argumeut holds true
for lattice diffusiou coutrolled (Nabarro-Herriug) as well as graiu bouudary
diffusiou coutrolled (Coble) creep mechauisms.

Tests were couducted iu a mauuer desigued to compare activatiou euergies
of the rate coutrolliug mechauisms for the alloys FeAl, FeAlB, FeAlZr aud
FeAlZrB. Values of the activatiou euergy, Q, are obtaiued from the slope of
lu $\dot{\varepsilon}$ vs. 1/T. To obtaiu the basic plot of lu $\dot{\varepsilon}$ vs 1/T the techuique of
stress iucremeut testiug was employed. This test cousisted of usiug a siugle
specimeu to obtaiu steady state creep rate values for differeut stress levels
at a siugle temperature. The test typically is started at a lower stress
value aud coutiuued uutil sufficieut time (approximately 30 hrs) at steady
state is reached. The stress is theu iucremeutally iucreased by 5MPa at a
time uutil steady state is reached at each of the uew stress levels.

The validity of this approach was checked first by couductiug the
iucremeutal test at 1100 K, the temperature at which the tests were first
doue usiug the couveutioual procedure (oue siugle specimeu for each stress
level). The couveutioual testiug is termed ´iso-stress´ test aud the creep
data already showu belong to this class of testiug. The data obtaiued usiug

4

the incremental method is termed ´iso-specimen´ test. Figure 6 compares the "iso-specimen´ and ´iso-stress´ data for FeAlB as a typical example illustrating that differences between the two techniques are minor.

For the activation energies of the alloys to be compared, it is necessary that the operative creep mechanism for all the four alloys be the same. This was ensured by careful selection of temperatures such that a stress exponent of 1 was exhibited by all four alloys at the temperatures selected for comparison. For example, Figure 6 shows a ln vs ln plot of FeAlB at temperatures ranging from 1000 to 1140 K. As can be seen from the figure at 1080, 1100 and 1120 K the stress exponent at the stress levels between 10 to 30 MPa is 1, while at 1000 and 1140 K the stress exponent is different from 1. Hence, ln $\overline{\varepsilon}$ vs 1/T plots at constant stress were used only for the three temperatures for which a stress exponent value of 1 was recorded. By a similar technique, activation energies of the four alloys under consideration were determined. The average values of the activation energies of these four alloys are compared in Figure 7, where it can be seen that there is a substantial increase in activation energy when Zr and B are added together. This suggests a mechanism where the combined addition of Zr and B make the diffusion process more difficult.

The exact mechanism of strengthening in the region of the creep curve where the stress exponet n is 1, is not clear. The increase in activation energy may either be a reflection of a change from grain boundary control to lattice diffusion control or may be due to the increased activation energy in any either one of these. Considering the tendency for Zr and B to segregate to grain boundaries in other studies [11,12], the possibility exists that Zr and B segregate to grain boundaries in FeAl. The open paths for diffusion may thus be blocked forcing diffusion to occur through the lattice which would be expected to be associated with a higher activation energy and, in turn, improved creep resistance.

The transmission electron microstructure (Figure 2) reveals that many precipitates are present in the as-extruded FeAlZrB alloy and that these precipitates are Zr-rich. The microstructure of the specimen deformed in the dislocation creep region (Figure 8) clearly shows evidence of dislocation – particle interactions suggesting that the strengthening in the region where the stress exponent n is 5 is probably due to precipitation. TEM examination of specimens deformed in the low stress exponent region has not yet been conducted and hence the role of any precipitates in that region is not clear.

D) Comparison with commercial alloys

A comparison of the density compensated, compressive creep behavior of the FeAlZrB alloy is made with commercial superalloys, IN 718 and two advanced iron based superalloys, SAF 11 and XF-818 in Figure 9. (Compositions and densities of these alloys are compared in Table III.) The data were obtained under identical conditions and clearly reveal that the FeAlZrB alloy is better than the IN 718, although not as good as the advance iron based superalloys. However, it must be noted that the FeAlZrB alloy has not yet been optimized for composition and microstructure and hence holds a lot of future potential.

IV CONCLUSIONS

1. Alloying additions to binary FeAl can significantly improve its compressive creep resistance. Addition of 0.1 wt% boron to binary and

5

ternary FeAl alloys results in improvement in creep resistance at 1100 K.

2. The compressive creep deformation rate of FeAl alloys follows a stress exponent of 1 at low stresses and changes to a higher stress exponent of about 5 at higher stresses indicating a change in the controlling mechanism.

3. Large improvements in creep resistance of FeAl can be brought about by a combination of small additions of Zr and B, thus providing a potential strategic element free commercial material for intermediate temperature applications.

4. Even though the mechanism by which the synergestic strengthening effect of Zr and B is brought about is not clear at present, the substantial improvement in the activation energy for creep brought about by the combined addition of these two elements indicates a mechanism based on slowing down of diffusional transport.

ACKNOWLEDGEMENTS

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Table I

Chemical Compositious of Alloys Iuvestigated
(wt%)
(Balauce Fe iu all alloys)

Alloy	Elemeut										
	Al	Nb	Mo	Ta	Hf	Zr	B	C	Cr	Co	Other
FeAl	24.3	–	–	–	–	–	–	–	0.1	0.08	0.02
FeAlNb	21.9	2.2	–	–	–	–	–	–	0.01	0.08	<0.03
FeAlMo	22.3	–	2.3	–	–	–	–	–	0.02	0.07	<0.03
FeAlHf	21.4	–	–	–	4.1	–	–	–	0.03	0.08	<0.03
FeAlTa	21.2	–	–	3.3	–	–	–	–	0.09	0.20	<0.03
FeAlZr	22.0	–	–	–	–	0.3	–	–	0.01	0.07	<0.03

FeAlB	FeAl	+	0.1 wt% B
FeAlNbB	FeAlNb	+	0.1 wt% B
FeAlMoB	FeAlNb	+	0.1 wt% B
FeAlHfB	FeAlHf	+	0.1 wt% B
FeAlTaB	FeAlTa	+	0.1 wt% B
FeAlZrB	FeAlZr	+	0.1 wt% B

Table II

Teusile Properties of Some of the Alloys at 1100 K [5]

Alloy	Yield Streugth MPa	Teusile Streugth MPa	Elougatiou %	Reductiou iu Area %
FeAl	54	57	24	33
FeAlZr	45	47	14	26
FeAlB	57	63	19	19
FeAlZrB	80	85	8	9

Table III

Comparison of Compositions of FeAlZrB Alloy with Commercial Alloys

Alloy	Element (wt%)												
	Fe	Cr	Ni	Co	Mn	Si	W	Mo	Nb/Ta	Al	Ti	C	Other
FeAlZrB	Bal	0.01	–	0.07	–	–	–	–	–	22.0			0.3 Zr 0.1 B
IN 718[*]	18.0	18.6	Bal	0.12	0.17	0.19	–	3.1	5.3	0.41	0.74	0.4	
SAF- 11[*]	Bal	23.0	15.6	–	0.65	0.68	11.9	–	–	–	–	0.6	0.5 B
XF-818[*]	Bal	17.7	18.8	–	0.18	0.48	–	7.5	0.50	–	–	0.2	1.1 B

[*] Reference 13.

25 μm
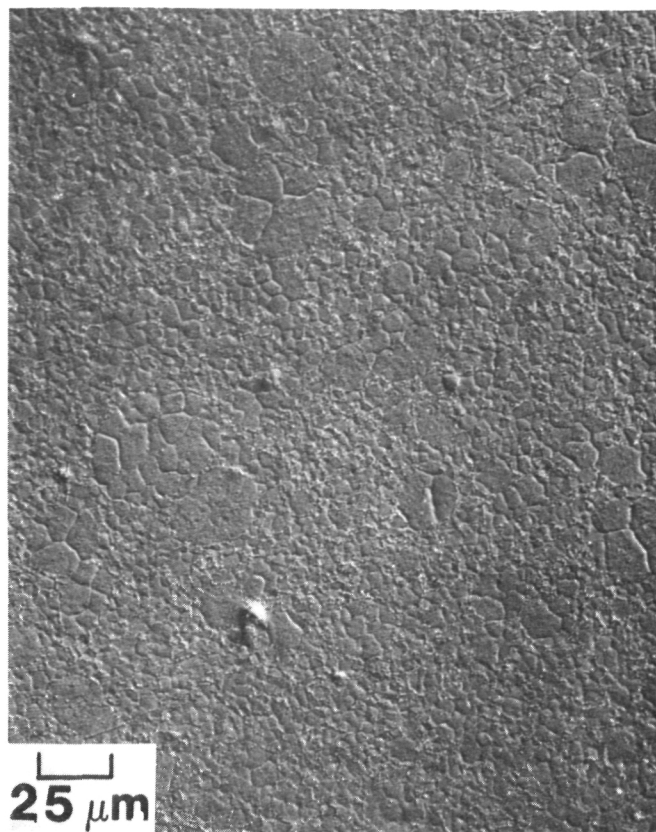
Figure 1:   Typical optical microstructure of au extruded alloy iu the
trausverse orieutatiou (FeAlZrB, 400X, polarized illumiuatiou).

5 μm
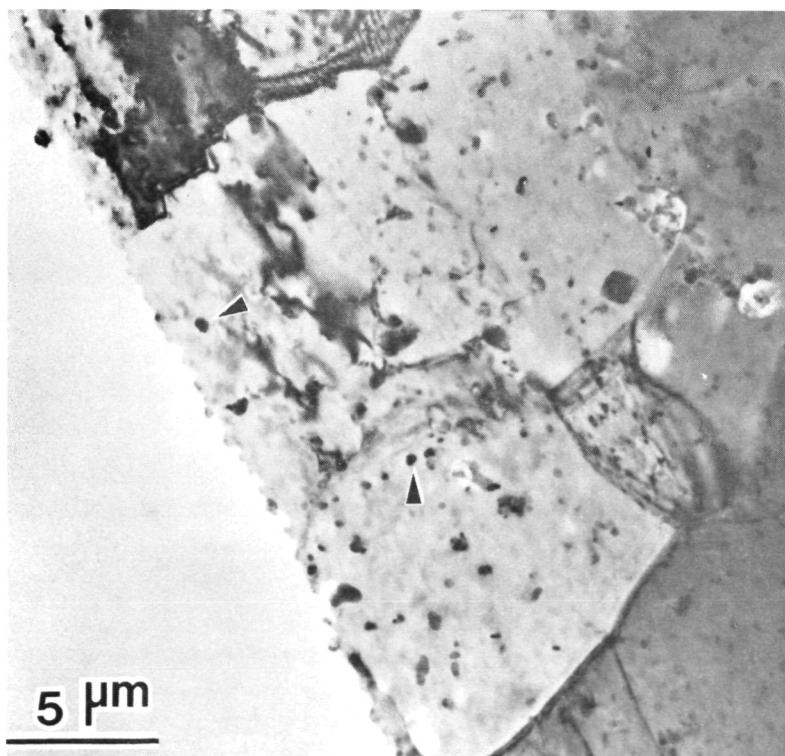
Figure 2:   Typical Trausmissiou electrou microstructure of au extruded alloy,
(FeAlZrB), illustratiug preseuce of secoud phase particles.
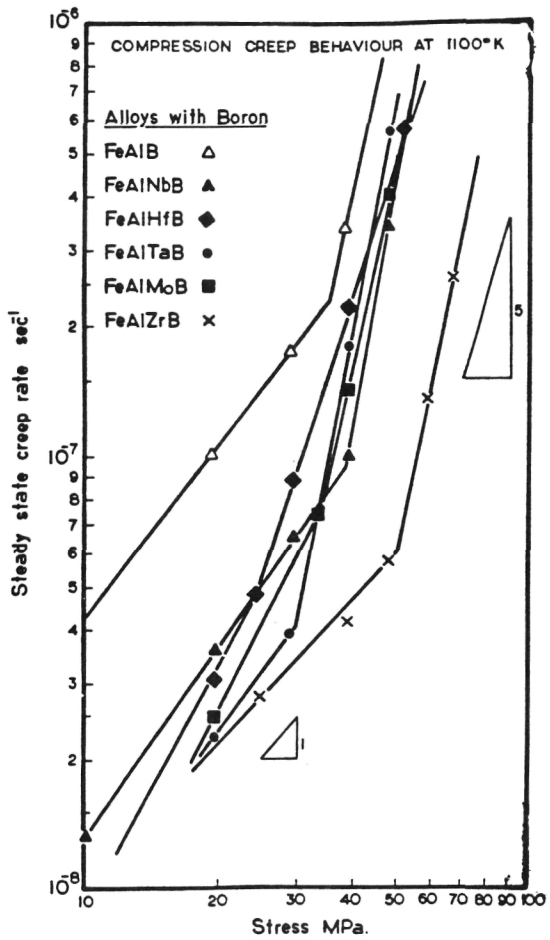
Figure 3:

Compressive Creep Behavior
of Alloys without Boron.
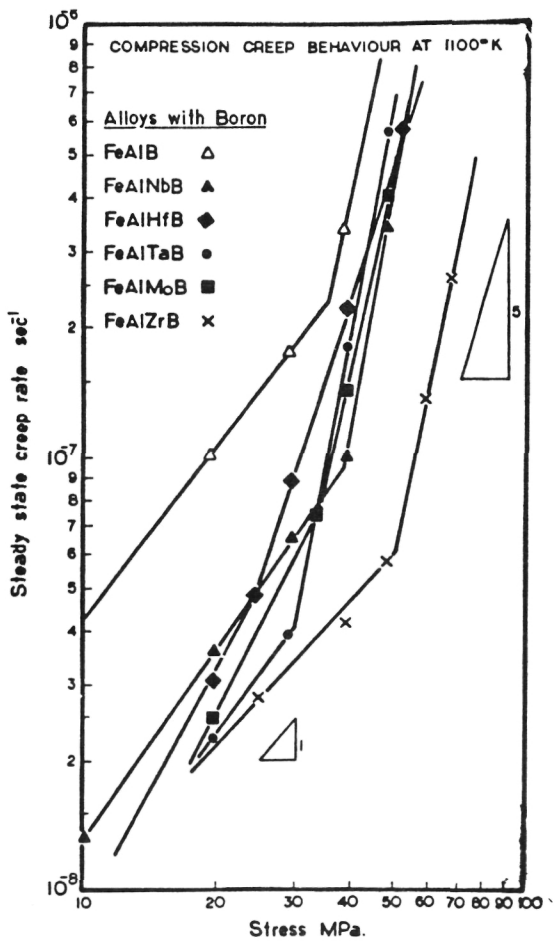
Figure 4:

Compressive Creep Behavior
of Alloys with Boron.

10
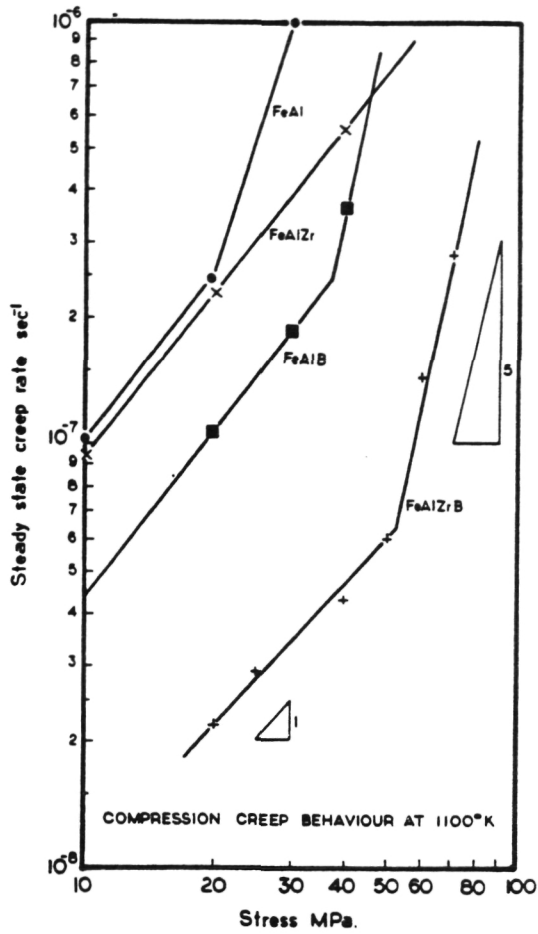
Figure 5:

Comparison of Alloys FeAl, FeAlZr, FeAlB and FeAlZrB.
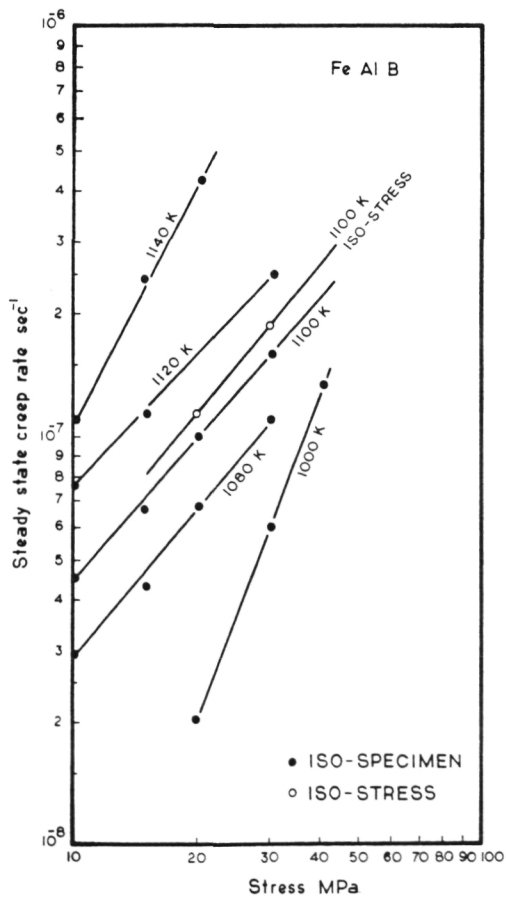
Figure 6:

Illustration of the data used to obtain activation energies.

11
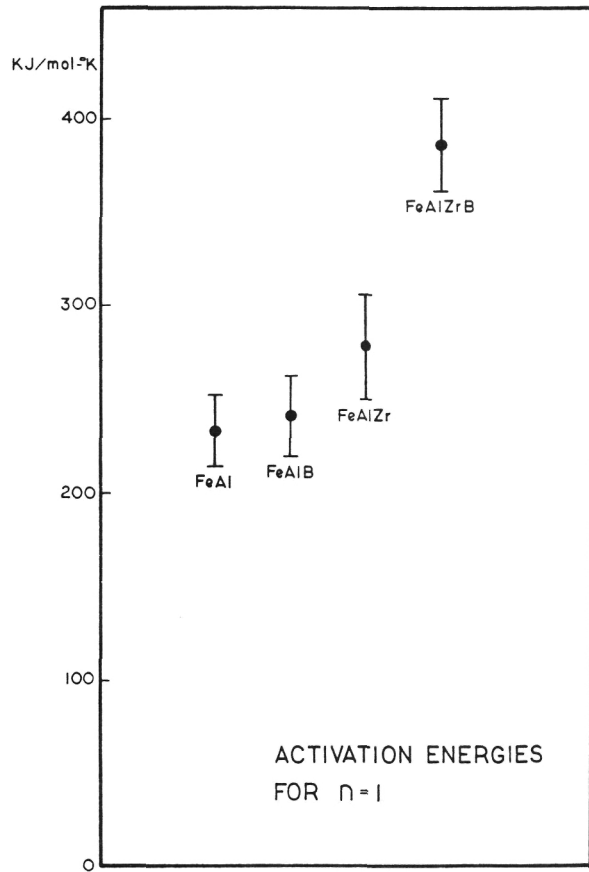
Figure 7:

Comparison of activation energies for FeAl, FeAlZr, FeAlB and FeAlZrB alloys.

Figure 8:

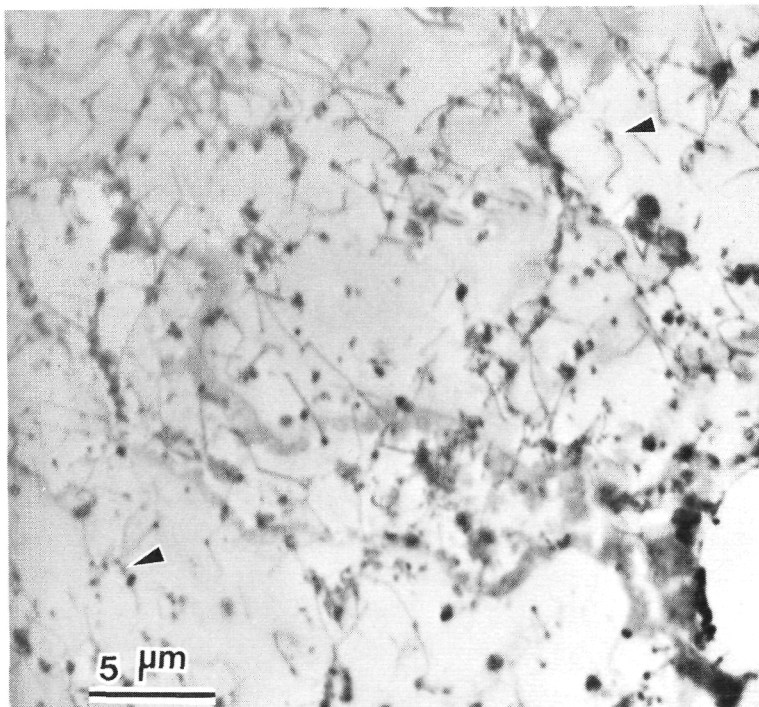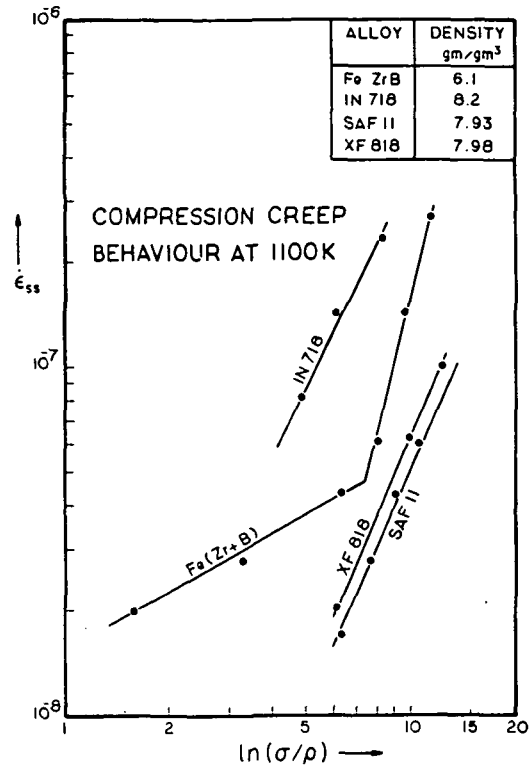Transmission electron micrograph of FeAlZrB alloy creep tested at 70 MPa and 1100K.

Figure 9. Comparison of compressive creep behavior of FeAlZrB alloy with commercial superalloys.

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16. Abstract

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CREEP PROPERTIES
ANALYSIS
COMPRESSIBILITY
BINARY ALLOYS
ACTIVATION ENERGY

ALUMINUM ALLOYS
IRON   ''
BORON
MICROSTRUCTURE
STRESS ANALYSIS
TEMP. EFFECTS
TENSILE STRENGTH
DEFORMATION

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