

AD-A174 598

INVESTIGATION AND SYNTHESIS OF HIGH TEMPERATURE AND  
INCREASED STIFFNESS R (U) NORTHWESTERN UNIV EVANSTON  
IL TECHNOLOGICAL INST M E FINE ET AL 31 OCT 86  
AFOSR-TR-86-1095 AFOSR-85-0337

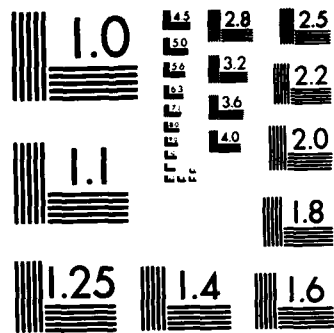
1/1

UNCLASSIFIED

F/G 11/6

NL





MICROCOPY RESOLUTION TEST CHART  
NATIONAL BUREAU OF STANDARDS-1963-A

2

AD-A174 598

AFOSR-TR-86-1095  
DEPARTMENT OF MATERIALS SCIENCE AND ENGINEERING  
THE TECHNOLOGICAL INSTITUTE  
NORTHWESTERN UNIVERSITY  
EVANSTON, IL 60201

Annual Technical Report on

Investigation and Synthesis of High Temperature and  
Increased Stiffness RSP Aluminum Alloys

to

Electronic and Solid State Sciences Division  
Air Force Office of Scientific Research  
Department of the Air Force  
Bolling Air Force Base  
Washington, D. C. 20332

Attn: Dr. Alan H. Rosenstein  
AF Grant No.: AFOSR-85-0337

Approved for public release;  
distribution unlimited.

For the Period

1 October 1985 to 30 September 1986

Principal Investigators:

Morris E. Fine, Walter P. Murphy Professor of Materials Science and  
Engineering

Julia R. Weertman, Professor of Materials Science and Engineering

October 31, 1986

AIR FORCE OFFICE OF SCIENTIFIC RESEARCH (AFOSR)  
NOTICE OF TRANSMITTAL TO DTIC  
This technical report has been reviewed and is  
approved for public release IAW AFR 190-12.  
Distribution is unlimited.  
STEPHEN J. KEEPER  
Chief, Technical Information Division

DTIC  
ELECTE  
NOV 26 1986  
S D

DTIC FILE COPY

86 11 25 27

ADA174598

## REPORT DOCUMENTATION PAGE

1a REPORT SECURITY CLASSIFICATION Unclassified		1b RESTRICTIVE MARKINGS --	
2a SECURITY CLASSIFICATION AUTHORITY --		3 DISTRIBUTION / AVAILABILITY OF REPORT  Unlimited	
2b DECLASSIFICATION / DOWNGRADING SCHEDULE --			
4. PERFORMING ORGANIZATION REPORT NUMBER(S) --		5 MONITORING ORGANIZATION REPORT NUMBER(S) <b>AFOSR-TR- 86-1095</b>	
6a. NAME OF PERFORMING ORGANIZATION Northwestern University	6b OFFICE SYMBOL (if applicable)	7a NAME OF MONITORING ORGANIZATION AFOSR/NE	
6c. ADDRESS (City, State, and ZIP Code) Dept. of Materials Science & Engineering Technological Institute, Rm. #2030 Evanston, IL 60201-9990		7b ADDRESS (City, State, and ZIP Code) Building 410 Bolling AFB DC 20332-6448	
8a. NAME OF FUNDING / SPONSORING ORGANIZATION AFOSR	8b OFFICE SYMBOL (if applicable) NE	9 PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER AFOSR-85-0337	
8c. ADDRESS (City, State, and ZIP Code) AFOSR/NE Building 410 Bolling AFB DC 20332-6448		10 SOURCE OF FUNDING NUMBERS	
		PROGRAM ELEMENT NO 61102F	PROJECT NO 2306
		TASK NO. A1	WORK UNIT ACCESSION NO. --
11 TITLE (Include Security Classification) Investigation and Synthesis of High Temperature and Increased Stiffness RSP Aluminum Alloys			
12. PERSONAL AUTHOR(S) Morris E. Fine, Julia R. Weertman			
13a. TYPE OF REPORT Annual Technical Rept.	13b TIME COVERED FROM 10/1/85 TO 9/30/86	14 DATE OF REPORT (Year, Month, Day) 10/31/86	15 PAGE COUNT 10
16 SUPPLEMENTARY NOTATION			
17 COSATI CODES		18 SUBJECT TERMS (Continue on reverse if necessary and identify by block number)	
FIELD	GROUP	SUB-GROUP	
11.02			
		High temperature aluminum alloys alum.num-vanadium-zirconium alloys	
19 ABSTRACT (Continue on reverse) if necessary and identify by block number)			
The objective of this research is to investigate possible high temperature aluminum alloys useful to 425°C (800°F). A metal matrix composite consisting of an aluminum-magnesium alloy matrix reinforced by spinel (magnesium aluminate) particulate and tri-aluminum (zirconium, vanadium) dispersed in aluminum matrix are being studied. A procedure for preparing specimens of the aluminum alloy matrix-spinel composite using mechanical alloying has been worked out. Extrusions containing 5 volume percent tri-aluminum (0.75 vanadium, 0.25 zirconium) were prepared for this research by Lockheed-Palo Alto from rapidly solidified foil. The measured creep rate at 425°C is much lower than in the current aluminum-iron-cerium alloy.			
20. DISTRIBUTION / AVAILABILITY OF ABSTRACT <input checked="" type="checkbox"/> UNCLASSIFIED/UNLIMITED <input type="checkbox"/> SAME AS RPT <input type="checkbox"/> DTIC USERS		21 ABSTRACT SECURITY CLASSIFICATION	
22a. NAME OF RESPONSIBLE INDIVIDUAL Morris E. Fine		22b TELEPHONE (Include Area Code) (312) 491-5579	22c OFFICE SYMBOL NE

PROFESSIONAL PERSONNEL

Professor Morris E. Fine, Principal Investigator

Professor Julia R. Weertman, Principal Investigator

Dr. Mahidhara K. Rao, Postdoctoral Research Associate,  
2/1/86 - 1/31/87

Mr. Yen Cheng (Dan) Chen, Ph.D. student, Graduate  
Research Assistant, 10/1/85 - 10/1/87

Mr. Terry S. Creasy, M.S. student, 10/1/85 - 12/31/86



Accession For	
NTIS CRA&I	<input checked="" type="checkbox"/>
DTIC TAB	<input type="checkbox"/>
Unannounced Justification	<input type="checkbox"/>
By .....	
Distribution / .....	
Availability Codes	
Dist	Avail and/or Special
A-1	

## SUMMARY

→ The objective of this research is to investigate two promising systems as the basis for high temperature aluminum alloys useful to 425° C (800° F). The first is a metal matrix composite consisting of an aluminum-magnesium alloy matrix reinforced by spinel (magnesium aluminate) particulate. The second system is tri-aluminum (zirconium, vanadium) dispersed in aluminum matrix. Here the lattice parameter matches that of the matrix. Research on dilute alloys has shown a low coarsening rate for this intermetallic at 425° C. Study of more concentrated alloys with the necessary volume percent of dispersoid for high temperature creep and fatigue resistance is underway.

A procedure for preparing specimens of the aluminum alloy matrix-spinel composite has been worked out and specimens are being prepared. In the procedure rapidly solidified alloy powder and oxide are mechanically alloyed, cold pressed, hot pressed, pressure forged and annealed.

Extrusions containing 5 volume percent tri-aluminum (0.75 vanadium, 0.25 zirconium) were prepared for this research by Lockheed-Palo Alto from rapidly solidified foil. The measured creep rate at 425° C is much lower than in the current aluminum-iron-cerium alloy.

## STATEMENT OF WORK

By analogy with Ni-base superalloys which are useful to  $0.75 T_m$  ( $T_m$  is the absolute melting temperature of Ni), one may anticipate development of an Al base alloy useful to approximately  $425^\circ\text{C}$  or  $800^\circ\text{F}$ . In order to develop a successful Al alloy for use at such a temperature, many basic principles must be considered. It is proposed that such an alloy will owe its strength at high as well as room temperature to a uniform dispersion of second phase particles which are coherent or semi-coherent with the matrix. To test this theory, two systems are under study:

1. Al-Mg alloy matrix -  $\text{MgAl}_2\text{O}_4$  composite
2. Al- $\text{Al}_3(\text{V},\text{Zr})$  dispersions

Both of these systems were selected on the basis of lattice matching between an Al alloy matrix and the dispersed phase. Preliminary work was done on Al- $\text{Al}_3\text{Zr}$  and Al- $\text{Al}_3(\text{Zr},\text{V})$  under AFOSR Grant No. 82-0005 where the results on dilute alloys were very promising.

## PROGRESS AND RESULTS

1. Al-Mg alloy matrix- $\text{MgAl}_2\text{O}_4$  spinel composite

The cubic spinel, nominal composition  $\text{MgAl}_2\text{O}_4$ , appears to be a better candidate oxide for the dispersion strengthening of Al than the hexagonal oxide  $\alpha\text{-Al}_2\text{O}_3$ . The lattice parameter of stoichiometric  $\text{MgAl}_2\text{O}_4$  is  $8.083\text{\AA}$  which is almost twice that of Al,  $4.0496\text{\AA}$ . Thus a semicoherent interface may be anticipated. Furthermore, at least one spinel-structured oxide exhibits some ductility at room temperature.<sup>2</sup> If the dispersed spinel particles in the Al alloy matrix have even very limited ductility, this should result in a much better fracture toughness than with particles like  $\alpha\text{-Al}_2\text{O}_3$  or SiC which behave in a very brittle fashion. Since the elastic

constants of spinel are considerably larger than those of Al, improved stiffness as well as improved high temperature strength is expected from dispersing spinel in an Al alloy matrix.

To date a procedure for preparing creep and elevated temperature fatigue specimens of Al-Mg alloy matrix-spinel ( $\text{MgAl}_2\text{O}_4$ ) or corundum ( $\alpha\text{-Al}_2\text{O}_3$ ) dispersions has been developed. Al-3% Mg rapidly solidified powder for the matrix was kindly provided for this research by the Alcoa Technical Center. Commercially available spinel and  $\alpha\text{-Al}_2\text{O}_3$  powders were provided by Baikowski. Composites containing 5 vol.% of either oxide were prepared by mechanical alloying with 1 wt.% oleic acid as a "grinding" aid using alumina pellets as the "grinding" medium. The mechanically alloyed powders were cold pressed to 0.75 in diameter disks with a dual action die at 350 MPa pressure. The cold pressed pellets were heated to 600°C for 5 minutes and then pressure forged in a platen press heated to 300°C to 1 inch diameter. The pressure forged disks were then annealed for 10 hrs at 450°C. The final densities were in the range 99 to 100% of the theoretical. The specimens are cut using electro-discharge machining. Creep and tensile testing of these specimens and microstructural studies are about to begin. Spinel and corundum as dispersoids will be compared.

## 2. Al- $\text{Al}_3$ (V,Zr) dispersions

In previous research completed under AFOSR Grant No. 82-0005C<sup>1</sup> precipitation of the metastable cubic  $\text{Ll}_2$  and tetragonal  $\text{DO}_{23}$   $\text{Al}_3$ (Zr,V) phases from dilute supersaturated solid solutions of Zr and V in Al were investigated. The alloys were prepared and solution treated by arc melting. By this technique it was possible to prepare alloys containing only up to 1 vol.% dispersoid. Addition of V was found to stabilize the cubic  $\text{Ll}_2$  phase (which

is the crystal structure of  $\gamma'$  in the Ni-base superalloys) so that it becomes a candidate dispersed phase for high temperature Al alloys. Both the cubic phase (spherical particles) and the tetragonal phase (platelets) coarsened slowly on isothermal aging at 425°C but the coarsening rate for the cubic phase was a factor of 10 slower in keeping with its lower interfacial energy.

In the present research, 5 vol.% dispersoid alloys obtained from Lockheed, Palo Alto are being investigated. Initially, splats were studied. In the more concentrated alloys cellular precipitation of the cubic phase was more extensive but this mode was suppressed by adding sufficient V. In an alloy designed to give 5 vol.%  $\text{Al}_3(\text{V}_{.875}\text{Zr}_{.125})$  only spherical cubic precipitates formed. These coarsened even more slowly than the 1 vol.% alloy which is contrary to the usual effect of increasing the volume fraction on the coarsening rate. Figure 1 compares the volumetric coarsening rate for the 1%  $\text{Al}_3(\text{V}_{.875}\text{Zr}_{.125})$  arc cast and the 5 vol.% splat. Using the modified Lifshitz-Slyozov-Wagner theory of volume diffusion coarsening due to Brailsford and Wynblatt<sup>3</sup> for 5 vol.% splat cooled samples,

$$\bar{r}_t^3 - \bar{r}_0^3 = \frac{12.8\sigma DV_m^2 C_\infty}{9RT} t = Kt \quad , \quad (1)$$

an effective interfacial energy 0.13 Joules/m<sup>2</sup> was computed from the measured coarsening rate and the diffusivity of Zr in Al reported in the literature. This is an order of magnitude smaller than computed for the 1% alloy. However,  $\text{Al}_{10}\text{V}$ , a stable phase in this alloy, precipitated at grain boundaries during the aging. Its role required further study. Subsequently, Lockheed, Palo Alto prepared rapidly solidified foils and then made bar-shaped extrusions from the foils. Three compositions were prepared, all designed to have 5 vol.% (a)  $\text{Al}_3(\text{V}_{.75}\text{Zr}_{.25})$ , (b)  $\text{Al}_3(\text{V}_{.5}\text{Zr}_{.5})$

and (c)  $Al_3(V_{.25}Zr_{.75})$ .

Transmission electron microscope study of the 5 vol.% melt spun ribbon with  $(V/Zr) = (3/1)$  showed precipitation of mostly spherical cubic  $Ll_2$  particles on aging at  $425^\circ C$  plus a small amount of cellular precipitation of the cubic phase. The coarsening rate of the cubic spherical particles was lower than in the splats, as shown in Fig. 1. The interfacial energy computed from eq.(1) was  $0.029 J/m^2$ , the expected value for a coherent interface between the  $Ll_2$  precipitate and the matrix. It is suggested that defects in the splats lead to accelerated aging. It is also suggested that the coarsening of the 1 vol.% chill cast arc melted alloys was enhanced by dislocations and subgrains.  $Al_{10}V$  precipitated at the grain boundaries preventing grain growth although an  $Ll_2$  precipitate free zone formed. Again, long time aging at  $425^\circ C$  (400 hrs) produced a small amount of the tetragonal dispersed phase on dislocations. Decreasing the  $V/Zr$  ratio increased the tendency toward cellular precipitation and also increased the amount of precipitation during solidification. This may be related to the fact that  $V$  reduces the liquidus temperature of  $Al-Zr$  alloys.

The 5 vol.% extruded bars were examined by TEM in the as-extruded condition. Precipitation of the cubic phase occurred during processing. No cellular precipitation was observed in the  $(V/Zr) = (3/1)$  alloy as well as no  $Al_{10}V$ . There was an occasional tetragonal particle. Processing of the lower  $V/Zr$  ratio alloys resulted in more cubic cellular and tetragonal precipitation.

In tests by Lockheed, Palo Alto<sup>4</sup> all extruded bars had very satisfactory elongations to fracture at room temperature being in the range 22 to 32%. The tensile strengths were approximately 42 Ksi. A higher tensile strength would, of course, be achieved with a higher volume fraction of dispersed

phase. Exposure of 100 hrs at 425°C had little effect on the elongations to fracture but the ultimate tensile strengths were reduced to 31 Ksi.

Creep tests of the three alloy extrusions were done at 425°C on a dead load creep machine. Creep curves with an initial creep stress  $P_i = 17$  MPa are shown in Fig. 2. For comparison, the creep curve for Al-8.8Fe-3.7Ce alloy, tested by Dr. Lynn Angers, is also given in the figure. The much slower steady state creep rates for alloys 2 and 4 shows that the approach we have adopted as the basis for high temperature alloys is very promising. However, alloy 3 showed poor creep resistance. It entered stage 3 of the creep curve almost immediately after loading indicating a much lower yield stress at 425°C for this alloy. The fractographs show that alloy 3 failed by ductile fracture while alloy 2 and 4 failure by intergranular fracture, the typical fracture mode for creep. TEM examination of foils prepared from crept specimens showed a lower density of  $Ll_2$  phase in alloy 3 ( $V/Zr = 1$ ) than in alloys 2 ( $V/Zr = .33$ ) and 4 ( $V/Zr = 3$ ). Presumably, the  $Ll_2$  phase was transformed to the  $DO_{23} Al_3 (V_{.5} Zr_{.5})$  and the  $Al_{10} V$  phases.

It is an exciting result that alloys 2 and 4 which contain only 5 vol.% of dispersoid show even better creep resistance at 425°C with  $P_i = 17$  MPa than Al-Fe-Ce alloy which contains about 22 vol.% of its dispersoids. It is due to the greater resistance of the  $Ll_2 Al_3 (Zr, V)$  to the creep deformation at that temperature than the incoherent  $Al_{13} Fe_4$  and  $Al_{10} Fe_2 Ce$  present in the Al-Fe-Ce alloys. Why the  $Ll_2 Al_3 (Zr, V)$  phase in alloy 3 decomposes faster than that in the alloys 2 or 4 is not known at this time and requires further study.

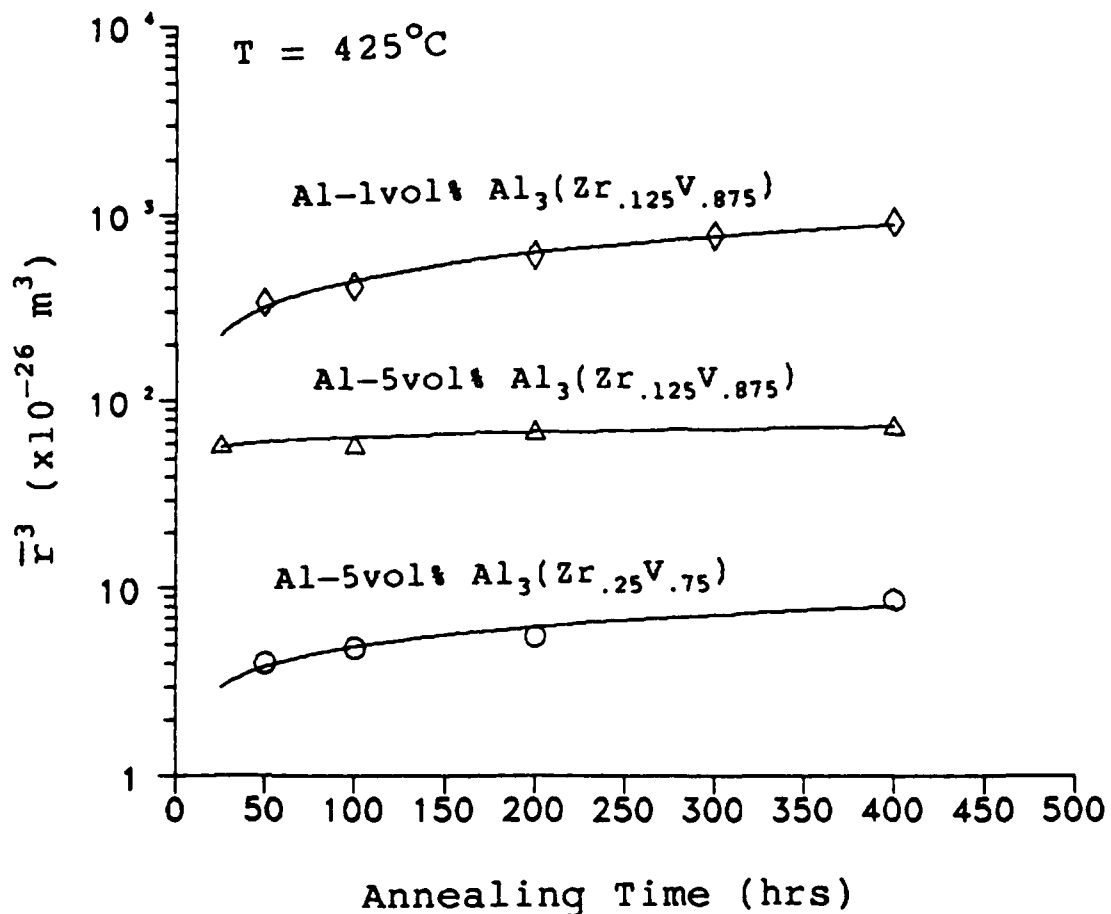
Future research will be on more concentrated Al base Zr, V alloys being prepared by Lockheed. Richard E. Lewis at Lockheed is a collaborator on the current Al-V-Zr alloy research.

## REFERENCES

1. M. S. Zedalis, Ph.D. thesis research - "Development of an Elevated Temperature Aluminum Alloy Containing Al<sub>3</sub>X-Type Dispersed Phases", Northwestern University, Evanston, Illinois, June 1985; M. E. Fine and J. R. Weertman, "Synthesis and Properties of Elevated Temperature P/M Aluminum Alloys", AFOSR Final Technical Report, 10/1/81 - 9/30/85, Grant No. AFOSR-82-0005.
2. G. W. Groves and M. E. Fine, "Solid Solution and Precipitation Hardening in Mg-Fe-O Alloys", J. Appl. Phys. 35 (1964) 3587.
3. A. D. Brailsford and P. Wynblatt, "The Dependence of Ostwald Ripening Kinetics on Particle Volume Fraction", Acta Metall. 27 (1979) 489.
4. Richard E. Lewis, Lockheed Research Laboratory, Palo Alto, CA. Private communication.

## LIST OF PUBLICATIONS -- AF SUPPORTED

1. M. E. Fine, Y. Chen, J. Conley and J. Caputi, "Interface Adsorption and Ostwald Ripening", Scripta Metall. 20 (1986) 743.
2. L. Angers, Y. Chen, M. E. Fine, J. R. Weertman and M. S. Zedalis, "Rational Design of High Temperature Aluminum Alloys" in Aluminum Alloys - Physical and Mechanical Properties (Conference Proceedings) Vol. I, E. A. Starke, Jr. and T. H. Sanders, Jr. (eds.) EMAS Publishers, U.K., pp. 321-337.
3. Y. C. Chen, M. E. Fine, J. R. Weertman and R. E. Lewis, "A Promising Strengthening Dispersoid for the High Temperature Al Alloys", to be presented at The Metallurgical Society Annual Meeting, 1987.
4. M. S. Zedalis and M. E. Fine, "Precipitation and Ostwald Ripening in Dilute Al Base-Zr-V Alloys", Metallurgical Transactions A, in press.
5. L. Angers, M. E. Fine and J. R. Weertman, "Effect of Plastic Deformation on the Coarsening of Dispersoids in a Rapidly Solidified Al-Fe-Ce Alloy", Metallurgical Transactions A, in press.
6. J. B. Cohen and M. E. Fine, "Solid State Phase Transformations in Al-Alloys" in Aluminum Alloys - Their Physical and Mechanical Properties (Conference Proceedings) Vol. III, EMAS Publishers, U.K., 1986.

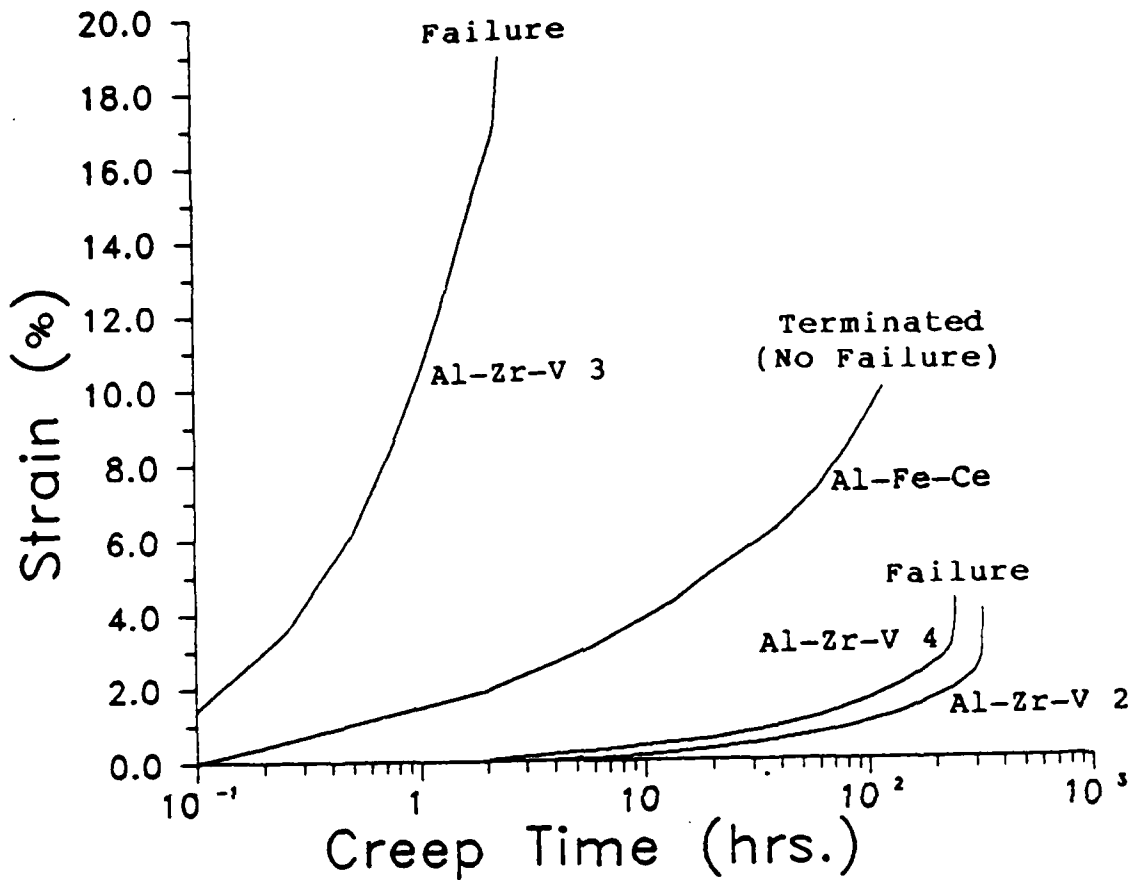


Alloy	Rate Constant $K$ ( $\text{m}^3/\text{hr}$ ) - slope of $\bar{r}^3$ vs $t$
Al-1vol% $\text{Al}_3(\text{Zr}_{.125}\text{V}_{.875})$	$1.63 \times 10^{-26}$
Al-5vol% $\text{Al}_3(\text{Zr}_{.125}\text{V}_{.875})$	$4.7 \times 10^{-28}$
Al-5vol% $\text{Al}_3(\text{Zr}_{.25}\text{V}_{.75})$	$1.03 \times 10^{-28}$

Fig. 1 Coarsening kinetics of three Al-Zr-V alloys ( $\bar{r}^3$  vs  $t$ ) and their rate constants,  $K$ .

T = 425°C

P<sub>i</sub> = 17MPa



Alloy	"Steady state creep rate (1/sec)"
Al-Fe-Ce	1.3x10 <sup>-7</sup>
Al-Zr-V 2	1.7x10 <sup>-8</sup>
Al-Zr-V 3	1.7x10 <sup>-5</sup>
Al-Zr-V 4	3.2x10 <sup>-8</sup>

Fig. 2 Creep curves of Al-Zr-V and Al-Fe-Ce alloys and their steady state creep rates.

END

1-87

DTIC