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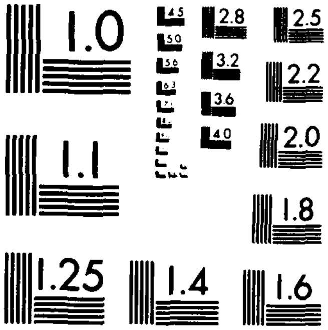

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Elevated temperature tensile and creep properties of powder metallurgy Al-Fe-Ni alloys with $FeNiAl_9$ dispersoid volume fractions of 0.19, 0.25 and 0.32 are being examined with respect to processing mode, microstructure and microstructural stability. The overall objective is to establish a basic understanding of processing-microstructure relations in this new class of alloys in order to provide design guidelines with respect to limits in service stresses and temperature. Tests have been conducted at temperatures up to 400°C.

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Ambient temperature strengthening can be explained by the Orowan dislocation bowing model. Yield strength decreases with increasing temperature and above 300°C, it is independent of dispersoid size and dispersoid volume fraction. Steady state creep rate is independent of the dispersoid volume fraction over the temperature range 250°C - 400°C, and the average stress exponent is 10 with a creep activation energy of 76 Kcal/mole. Elevated temperature deformation is consistent with a cooperative dislocation climb mechanism which is insensitive to dispersoid size and dispersoid volume fraction.

The data and observations confirm the benefits of a dual powder metallurgy/ rapid solidification approach with respect to enhanced microstructural stability at elevated temperatures. Similar studies have been initiated in which a mechanical alloying step is introduced prior to powder consolidation.

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ANNUAL TECHNICAL REPORT

"A FUNDAMENTAL STUDY OF P/M PROCESSED ELEVATED TEMPERATURE ALUMINUM ALLOYS"

AFOSR Grant #82-0010

Principal Investigators: A. Lawley and M.J. Koczak

Department of Materials Engineering
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October 1984

ABSTRACT OF RESULTS

Elevated temperature tensile and creep properties of powder metallurgy Al-Fe-Ni alloys with $FeNiAl_9$ dispersoid volume fractions of 0.19, 0.25 and 0.32 are being examined with respect to processing mode, microstructure and microstructural stability. The overall objective is to establish a basic understanding of processing-microstructure relations in this new class of alloys in order to provide design guidelines with respect to limits in service stresses and temperature. Tests have been conducted at temperatures up to 400°C. Ambient temperature strengthening can be explained by the Orowan dislocation bowing model. Yield strength decreases with increasing temperature and above 300°C, it is independent of dispersoid size and dispersoid volume fraction. Steady state creep rate is independent of the dispersoid volume fraction over the temperature range 250°C - 400°C, and the average stress exponent is 10 with a creep activation energy of 76 Kcal/mole. Elevated temperature deformation is consistent with a cooperative dislocation climb mechanism which is insensitive to dispersoid size and dispersoid volume fraction.

The data and observations confirm the benefits of a dual powder metallurgy/rapid solidification approach with respect to enhanced microstructural stability at elevated temperatures. Similar studies have been initiated in which a mechanical alloying step is introduced prior to powder consolidation.

TABLE OF CONTENTS

INTRODUCTION

Traditionally, aluminum alloys have been used in aircraft structures because of their high strength/weight ratio. In particular, there has been a concerted effort to develop Al alloys for improved performance at elevated temperatures. New and emerging powder metallurgy processing technologies have been particularly beneficial in this regard.

A major goal of the Air Force has been to develop aluminum alloys suitable for long time service in the temperature range 230 - 340°C. A number of powder processed alloys are being evaluated [1-4]; these involve the addition of transition metal elements, e.g. Fe, Ni, Cr, Mo and/or rare earth elements, e.g. Ce. The most promising alloy systems are Al-Fe-Ce, Al-Fe-Co, Al-Fe-Mo and Al-Fe-Ni. Rapid solidification, intrinsic to the atomization process for powder production, results in a high volume fraction of fine, thermodynamically stable dispersoids.

The basic strengthening mechanisms are well understood and have been applied to a broad spectrum of ingot metallurgy Al alloys. In contrast, since powder processed and rapidly solidified Al alloys are relatively new, it is of importance to assess their mechanical behavior in light of existing models of strengthening.

THE PRESENT PROGRAM

Only a limited fundamental understanding exists of the relationships between composition, processing mode, microstructure, properties and performance in this new class of dispersion-strengthened P/M processed aluminum alloys. It is the overall objective of the present study to develop such relationships. This science base is necessary in order to attain optimum properties and reflects a complex interplay of powder characteristics, powder solidification rate, composition, mode(s) of consolidation and subsequent deformation processing.

The present study is on the Al-Fe-Ni system; three compositions are inlcuded namely: Al-4.2 w/o Fe- 3.9 w/o Ni, Al-4.9 w/o Fe-4.8 w/o Ni, and Al-5.9 w/o Fe-6.1 w/o Ni. If all the Fe and Ni is out of solution, the corresponding volume fractions of dispersoid are

0.19, 0.25 and 0.32, respectively; this is based on an intermetallic dispersoid of M_2Al_9 where M is Fe or Ni.

Procedures, results and observations, and important implications covering the third year of the study are summarized in this report. Particular interest is centered on elevated temperature deformation of dispersion strengthened alloys in which microstructural stability is of primary concern.

PROGRAM SUMMARY

(a) Procedures

Al-Fe-Ni alloys were air atomized to achieve different levels of dispersoid in the aluminum matrix of the powder particles. The volume fractions (V_f) of the $FeNiAl_9$ dispersoids were 0.19, 0.25 and 0.32 respectively. The powders were canned, degassed and hot extruded to full density at 371°C using an extrusion ratio of 16:1.

Tensile tests were performed at ambient temperature and at temperatures of 200°C, 300°C, 350°C and 400°C. A strain rate of 2.2×10^{-4} sec^{-1} was used. The yield strength (YS), ultimate tensile strength (UTS), % elongation and % reduction in area (RA) were measured.

Ambient temperature tensile tests were conducted in accordance with ASTM standard E8. Tests were performed on each of the alloy compositions in the extruded form. In addition, tests were also performed after exposure of the extruded material at 450°C for periods up to 624 hrs.

Elevated temperature tensile tests were conducted in accordance with ASTM standard E21. The tests were performed on each of the alloy compositions in the extruded form. In addition, tests were also performed on the extruded alloy (V_f 0.25) after exposure at 450°C for 288 hours.

Constant load creep tests were carried out on a SATEC model M3 creep testing machine. The creep extensometer was attached to a shoulder on the tensile specimen.

The complete matrix of test temperatures and stresses is given in Table I. Steady state creep rates and the time for creep strains of 0.5% and 1.0% were recorded; specimens were tested to rupture or, in cases where rupture did not occur up to 100 hours, the test was terminated.

Powder and extruded material were examined by optical microscopy before and after elevated temperature exposure. Isochronal and isothermal hardness response of the powder particles (microhardness) and extruded alloys (macrohardness) were monitored. Transmission electron microscopy (TEM) was used to characterize the microstructure in the powders and extruded material. The dispersoids were identified by a combination of x-ray diffraction, selected area electron diffraction (SAD) and energy dispersive spectroscopy (EDS) on a STEM. Studies were also carried out on the extruded material to determine the kinetics of the dispersoid coarsening.

(b) Results and Observations

Data and observations presented in this progress report pertain to each of the three volume fractions of reinforcement of $FeNiAl_9$, in which the rapidly solidified air atomized (prealloyed) powder was hot extruded to full density. We have recently initiated similar studies on Al-Fe-Ni alloys in which the prealloyed powders are subjected to mechanical alloying prior to consolidation by hot extrusion.

(i) Microstructure:

A detailed description of the microstructure of these Al-Fe-Ni alloys in the form of atomized powder and following hot extrusion is given elsewhere [5]. The extruded microstructure consists of a recovered subgrain structure in the aluminum matrix with equiaxed $FeNiAl_9$ dispersoids. This is illustrated in Figure 1. The average size of the subgrains and dispersoids are 0.3μm and 0.1μm respectively. It has been confirmed that significant coarsening of the dispersoids occurs above 400°C [5].

(ii) Tensile Reponse:

Experimental data illustrating the effect of dispersoid size on ambient temperature yield strength are shown in Figure 2a. The range of dispersoid sizes reflected in Figure

2a was achieved by coarsening at 450°C for times up to 624 hours. At a given dispersoid size, it is seen that the yield strength of the alloy increases with increasing volume fraction of $FeNiAl_9$. The calculated dependence of yield strength on dispersoid size utilizing the Orowan dislocation bowing model [6] is illustrated in Figure 2b. Both the experimental and theoretical yield strength data exhibit a similar dependence on dispersoid size.

The dependence of yield strength on temperature at the three volume fraction levels of dispersoid is illustrated in Figure 3. It is observed that the differences in yield strength between the three alloys decreases with increasing temperature; above 300°C the yield strength is essentially independent of volume fraction of dispersoid. Alloy ductility is plotted as a function of temperature in Figure 4. At ambient temperature both elongation and reduction in area decrease as V_f increases. At 400°C, however, the trend is reversed.

A comparison of the temperature dependence of the yield strength of the as extruded alloy and the alloy after elevated temperature exposure (450°C/288 hours) is shown in Figure 5 for V_f 0.25. It is seen that above about 300°C the yield strength of the alloy is essentially independent of the dispersoid size. In the as extruded condition the average dispersoid size is about 0.1μm whereas after exposure (450°C, 288 hours) the average dispersoid size is 0.6μm. At room temperature, there is a 50% decrease in yield strength caused by this difference in dispersoid size, whereas at 400°C, the yield strength is the same for both conditions of the alloy.

Representative deformation substructures as revealed by TEM are shown in Figure 6. It is seen that some interaction occurs between dispersoids and the gliding dislocaions at ambient temperature. In contrast, no definitive dislocation structure could be observed following elevated temperature deformation. There is no evidence of dispersoid shearing by dislocations or of any gross change in the morphology of the dispersoids.

At ambient temperature and above, fracture occurs in a ductile manner by dimple rupture. Compared to room temperature, at 200°C, 300°C, and 400°C the dimples are larger and more sharply defined. In consequence, the fracture surface corresponding to fracture at 400°C is irregular (uneven). The fracture response of the alloys is independent of the volume fraction of dispersoid.

(iii) Creep Response:

Data derived from the creep curves are summarized in Table II for the V_f 0.32 and V_f 0.19 alloys. The stress exponent 'n' and creep activation energy are given in Table III.

Analogous to the tensile response it was observed that creep response in the as-extruded and exposed (400°C/2 hours) alloy (V_f 0.32) was similar. There is no evidence of any significant dislocation - dispersoid interactions after creep deformation. Representative micrographs (TEM) are illustrated in Figure 7. It is also seen that creep deformation does not significantly alter the dispersoid morphology. Subgrains are well defined with sharp subgrain boundaries.

Creep fracture surface morphologies are similar to those observed under tensile loading at elevated temperature. The appearance of the fracture surface does not appear to be sensitive to dispersoid volume fraction.

(c) Interpretation and Signficance of Results

The yield strength at room temperature can be predicted by the Orowan strengthening mechanism. It is reasonable to assume that dislocations move by bowing out between the dispersoids as there is no evidence of any dispersoid shearing. A comparison of Figures 2a and 2b shows that at a given value of V_f and dispersoid diameter, the experimental value of the yield strength is always higher than that predicted by the Orowan model. For example, for V_f 0.25 and dispersoid diameter 0.3μm, the corresponding yield strength values are 275 MPa. (experimental) and 225 MPa. (predicted). The higher experimental yield strength values are attributed to (i) additional strengthening by the substructure developed during hot extrusion and (ii) the fact that there is a distribution of dispersoid sizes. In particular, the finer dispersoids (<0.1 μm) have a stronger influence on the yield strength than the coarser $FeNiAl_9$ dispersoids, and this effect is not accounted for in the Orowan equation.

Other thermally assisted deformation mechanisms can be operative at elevated temperatures. The initial decrease in yield stress with increasing temperature, Figure 3, is due to a decrease in the shear modulus of the matrix with increasing temperature. The Orowan mechanism dominates below 200°C. Above 200°C, the yield strength of the alloy decreases more rapidly than the matrix shear modulus, suggesting that a different mechanism is operative. Above 300°C, the strength of the three alloys is independent of V_f; this implies that the dispersoids are no longer effective in impeding dislocation motion. Excessive dispersoid coarsening does not account for the large decreases in yield strength of the alloys at higher temperatures as shown in Figure 5. The effect of coarsening is clearly apparent up to about 200°C, whereas above 300°C the strength is independent of dispersoid size and spacing.

A possible deformation mechanism operative above 300°C is dislocation climb in which the barriers are the dispersoids. Recently, McLean [7] has developed a model for the cooperative climb of dislocations around groups of particles in γ-γ' Ni based superalloys. In both the γ-γ' and Al-Fe-Ni system there is a large volume fraction of precipitates or dispersoids. The model for the γ-γ' alloys predicts that elevated temperature deformation should be relatively insensitive to the size and volume fraction of the dispersoids.

Fracture in the Al-Fe-Ni system occurs by void initiation and coalescence [6], consistent with the appearance of dimples on the fracture surface following ambient temperature tensile deformation. The size of the dimples corresponds to the interdispersoid spacings. Fracture at elevated temperatures occurs by void coalescence. Voids open up around large clusters of dispersoid particles since the stress required for void initiation is low. The dimples are large and frequently contain several dispersoids.

Above 400°C the voids are relatively large and fracture occurs by the coalescence of these large voids resulting in a rough or uneven fracture surface morphology. The large decrease in ductility, as measured by reduction in area between ambient and 200°C, is attributed to homogeneous deformation, as opposed to intense localized necking. The corresponding % elongation either remains constant (V_f 0.19) or decreases slightly (V_f 0.25 and V_f 0.32). The significant increase in ductility above 300°C in the V_f 0.32 alloy is probably due to the presence of voids in the material. Large voids (2-3μm) were observed in this material.

The observed steady state creep rates fall in the range 5.0 x 10^{-9} sec^{-1} to 1.9 x 10^{-5} sec^{-1}. Thus the steady state creep rate is not particularly sensitive to V_f. Consistent with the tensile response, this suggests that the deformation mechanism(s) operating at elevated temperatures is independent of the volume fraction of the dispersion. The steady state creep rate after exposure (450°C/2 hours) is similar to that of the as-extruded alloy, *confirming that the particle size has no effect.*

The stress exponent values determined in this study, Table III, are much lower than the values reported previously for dispersion strengthened alloys. The average value of 'n' in the Al-Fe-Ni alloys is ~10 compared to values as high as 75 for some systems [8,9]. For further comparison, values of 5-7 are typical of single phase alloys [10]. The calculated activation energy in the present study (~76 Kcal/mole) is about twice the activation energy for self diffusion in aluminum, namely ~35 Kcal/mole. For other Al-matrix dispersion strengthened systems (e.g., SAP type alloys), the difference between the activation energy for creep and self diffusion is much higher than in Al-Fe-Ni.

All the creep stresses used in this study were lower than the calculated yield stresses based on the Orowan model of dislocation bowing. Thus it can be concluded that the Orowan dislocation mechanism is not the operative creep mechanism. It is also clear that the creep rate would be affected by the magnitude of V_f if indeed the Orowan mechanism were operative. Such is not the case. It is not anticipated that diffusional creep mechanisms will be operative in this study since these are expected to dominate at very low creep stresses. In light of these observations, it is concluded that the McLean model involving cooperative dislocation climb may be operative at these temperatures [7]. The model does show that resistance to dislocation climb is insensitive to volume fraction of dispersoid.

The well defined subgrains observed in the TEM micrographs following creep are the result of recovery which is integral to the creep process. It has been observed [11] that subgrain formation is typical of high temperature creep and the subgrains formed are more distinct and well defined than those formed during deformation at lower temperatures. Creep fracture surface morphologies are similar to those resulting from elevated temperature tensile fractures. Voids formed during creep grow under the action of the stress until rupture finally occurs by the coalescence of these large voids.

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TABLE I: Creep Test Temperatures and Stresses

Temperature (°C)	Stress (MPa)
250	103.4
250	151.6
250	172.3
250	186.0
350	34.5
350	55.1
350	68.9
350	82.7
375	55.1
400	34.5
400	44.8
400	55.1

Table II: Creep Data

V_f 0.19

Temperature (°C)	Stress (MPa)	ε_s* (sec^{-1})	$t_{0.5}$** (mins.)	$t_{1.0}$*** (mins.)
250	103.4	6.0×10^{-9}	950.8	-
250	151.6	1.8×10^{-6}	9.9	40.7
350	34.5	1.5×10^{-9}	-	-
350	55.1	2.7×10^{-8}	307.4	2814.9
350	68.9	6.7×10^{-7}	17.9	112.2
350	82.7	6.3×10^{-6}	2.4	12.4
375	55.1	3.5×10^{-7}	50.7	278.3
400	34.5	1.7×10^{-8}	518.3	2946.5
400	44.8	2.6×10^{-7}	44.1	307.6
400	55.1	2.9×10^{-6}	3.9	26.8

V_f 0.32

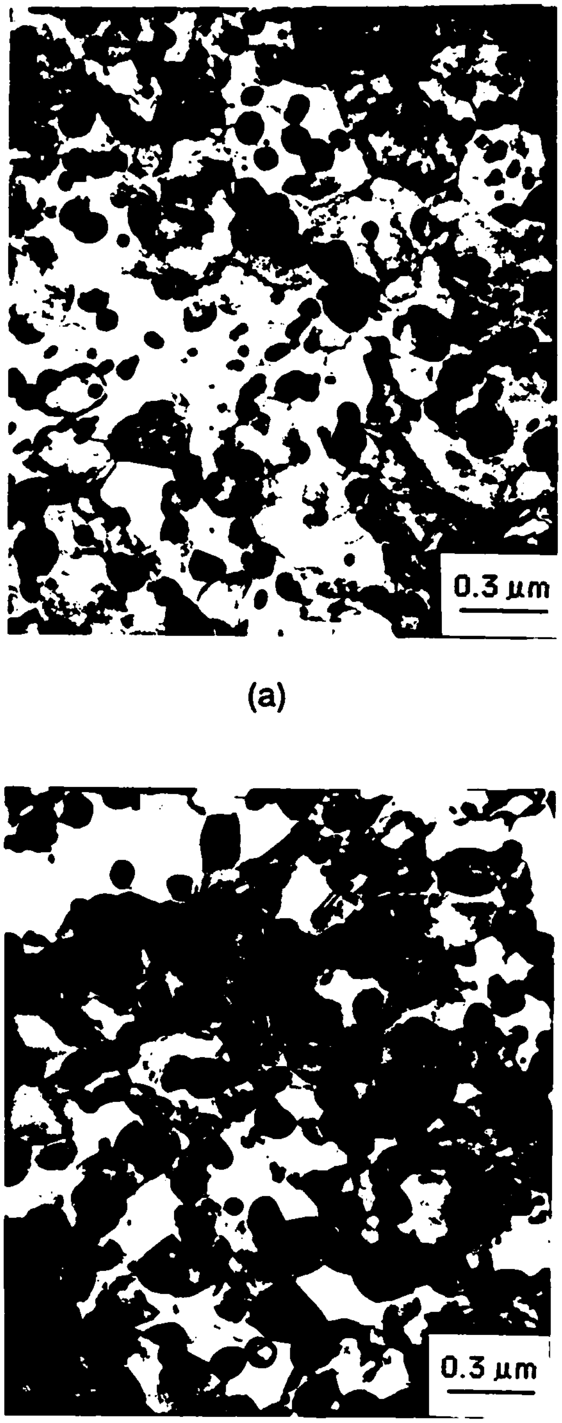
Temperature (°C)	Stress (MPa)	ε_s* (sec^{-1})	$t_{0.5}$** (mins.)	$t_{1.0}$*** (mins.)
250	103.4	1.3×10^{-8}	549.2	4871.5
250	151.6	9.5×10^{-7}	11.9	79.6
350	34.5	5.0×10^{-9}	1830.5	-
350	55.1	3.2×10^{-7}	67.8	196.2
350	68.9	3.8×10^{-6}	6.2	15.7
350	82.7	1.9×10^{-5}	1.2	3.8
375	55.1	1.7×10^{-6}	12.7	40.7
400	34.5	7.2×10^{-8}	105.4	210.7
400	44.8	3.3×10^{-6}	7.0	15.8
400	55.1	2.9×10^{-5}	0.8	2.7

* steady state creep rate ** time for creep strain of 0.5%

*** time for creep strain of 1.0%

Table III: Creep Stress Exponent 'n' and Activation Energy 'Δ'

Alloy	Temperature (°C)	n	Δ (Kcals/mole)
$V_f = 0.19$	350	9.3	76.9
	400	11.0	
$V_f = 0.25$	350	9.0	78.6
	400	11.0	
$V_f = 0.32$	350	9.5	74.3
	400	12.9	



(a)

(b)

Figure 1: TEM of extruded alloys; transverse sections. (a) V_f 0.19; (b) V_f 0.32.

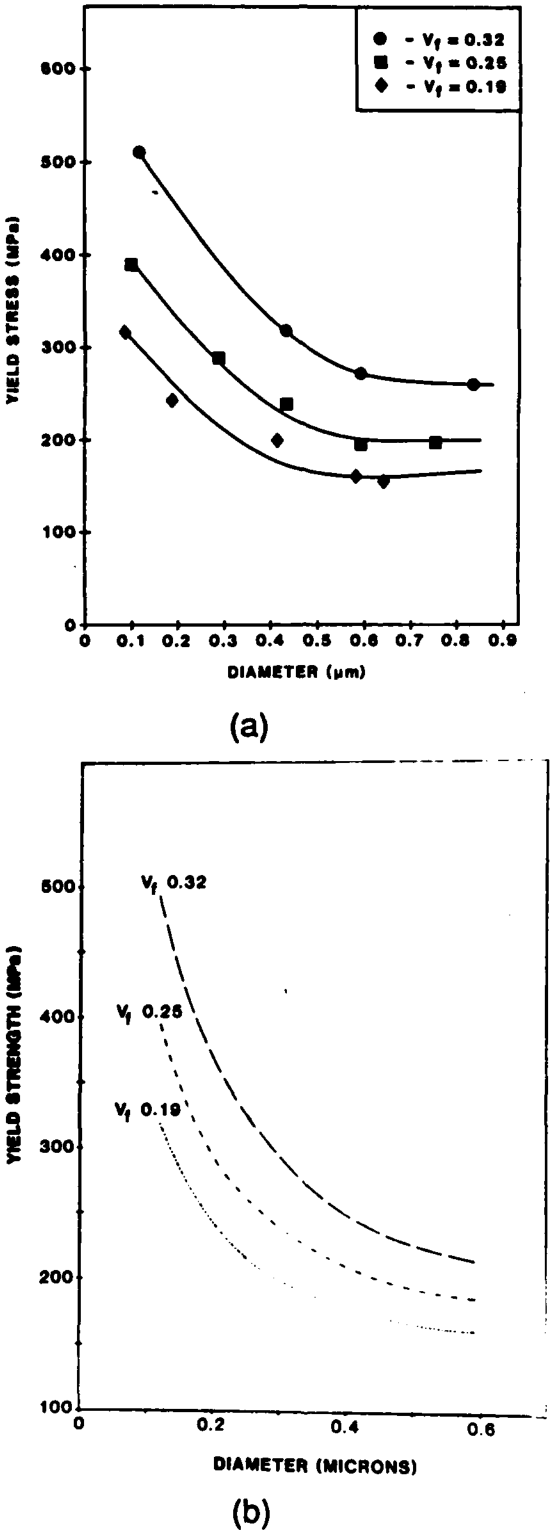


Figure 2: Effect of dispersoid size on ambient temperature yield strength.
(a) experimental; (b) Orowan analysis.

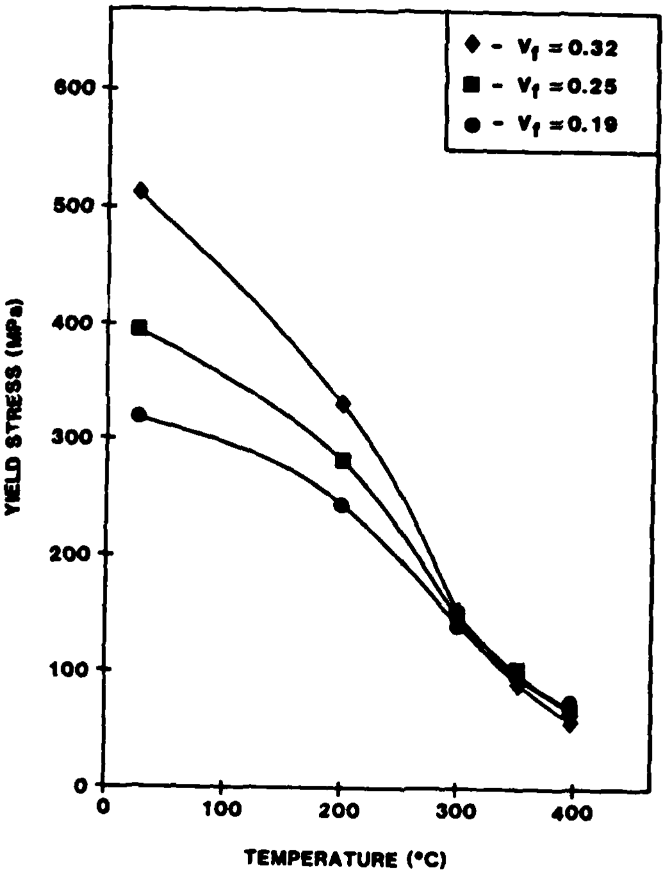
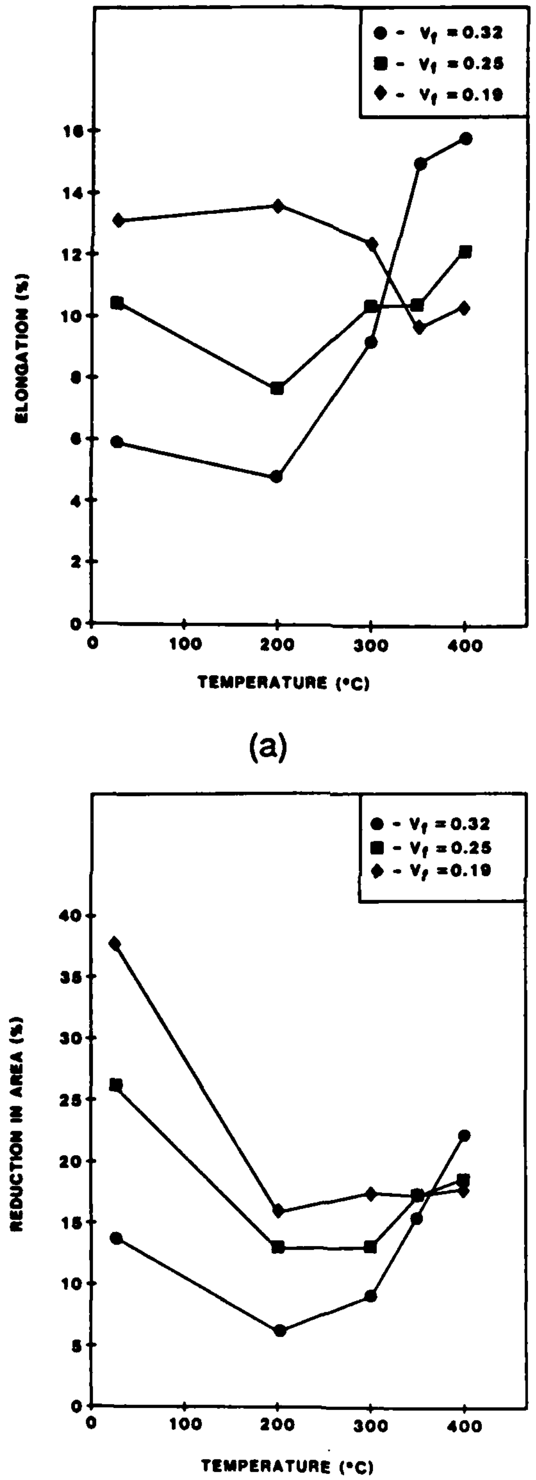


Figure 3: Effect of temperature on the yield strength of as-extruded alloys.



(a)

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Figure 4: Elevated temperature ductility. (a) % elongation; (b) % reduction in area.

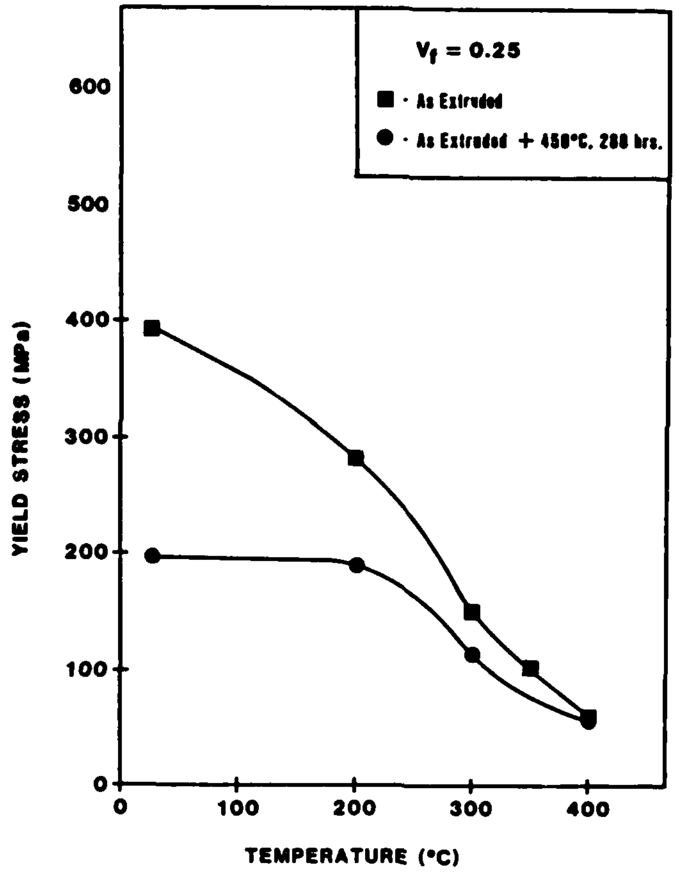
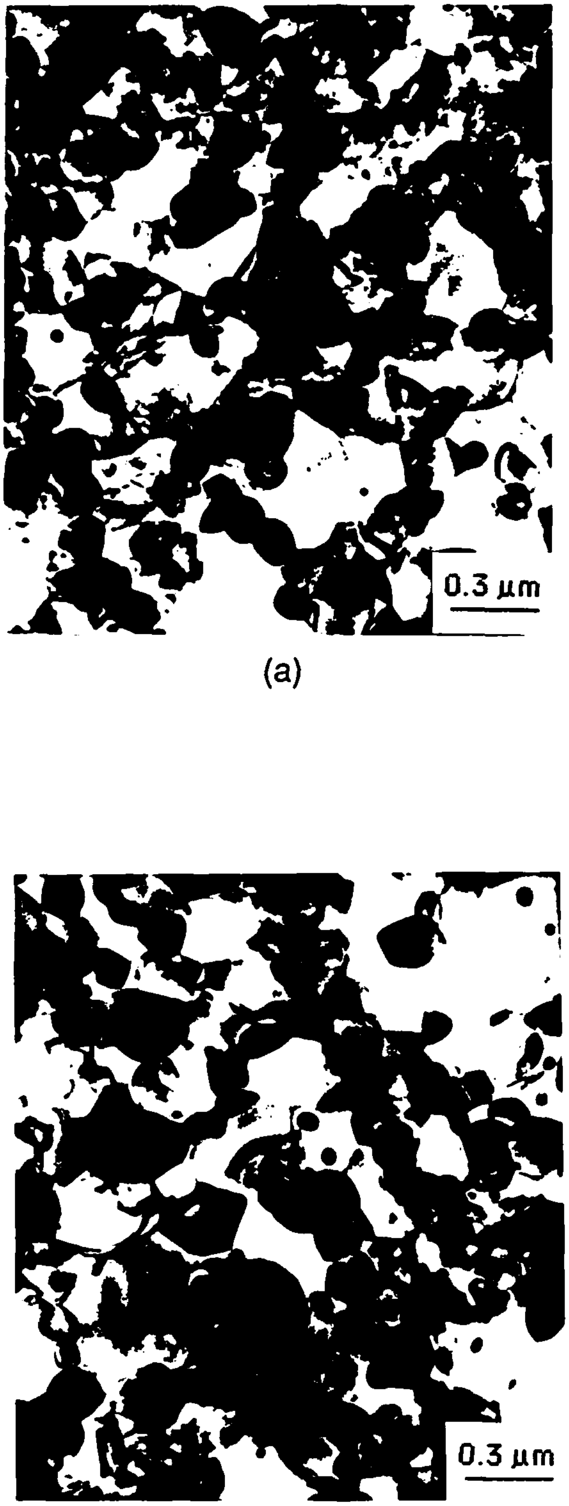


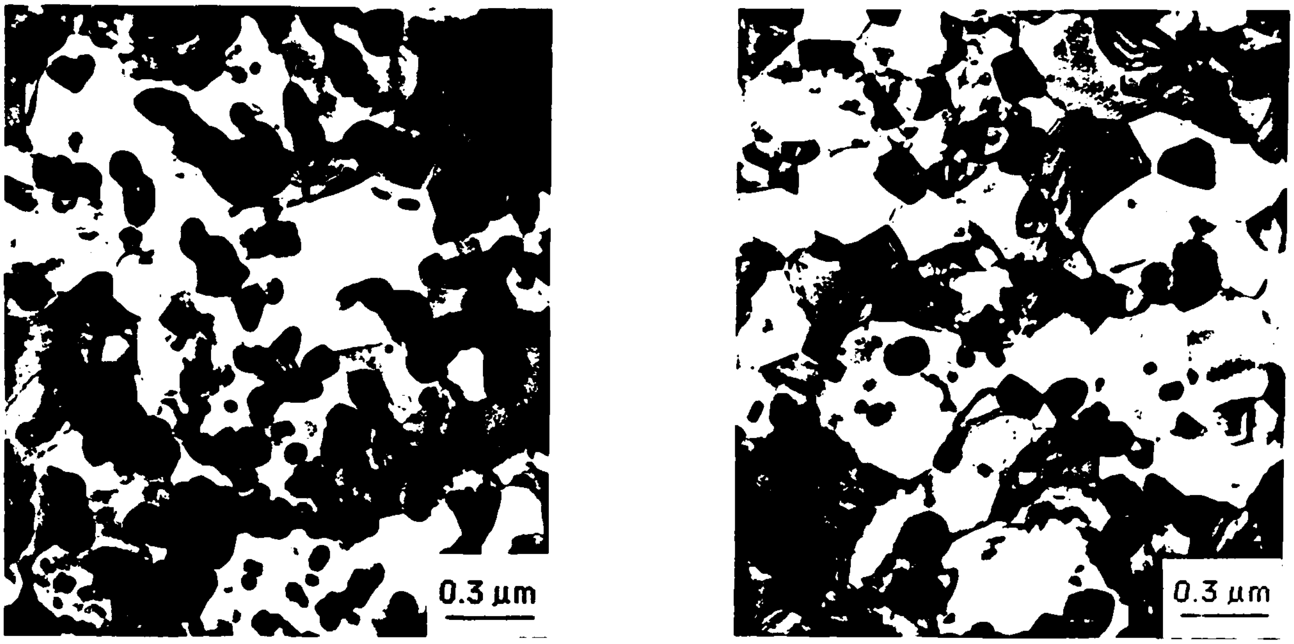
Figure 5: Comparison of the effect of temperature on yield strength (V_f 0.25) in the as-extruded and coarsened conditions.



(a)

(b)

Figure 6: TEM of alloy (V_f 0.32) close to fracture surface, following tensile fracture. (a) ambient temperature; (b) 400°C.



(a)

(b)

Figure 7: TEM of alloy close to fracture surface following creep (350°C/82.7 MPa.) (a) V_f 0.19; (b) V_f 0.32.

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M.K. Premkumar, "A Fundamental Study of P/M Processed Elevated Temperature Aluminum Alloys", Ph.D. Dissertation, Department of Materials Engineering, Drexel University, in progress.

PERSONNEL

A. Lawley - Professor and Co-Principal Investigator

M.J. Koczak - Professor and Co-Principal Investigator

E.Y. Gutmanas - Visiting Professor

M.K. Premkumar - Ph.D. Student

COUPLING ACTIVITIES

a) Presentations

"Challenges and Opportunities in Powder Metallurgy", John Deere, Moline, Illinois, March 1984.

"Developments in Specialty and New Alloys: Processing Technologies, Properties and Applications", Boston Chapter ASM, Cambridge, MA., March 1984.

"P/M Processing Science and Technology", Linde/Union Carbide, Tarrytown, N.Y., March 1984.

"Aluminum Alloy Powder Consolidation", ASTM Conference on Rapidly Solidified Aluminum Alloys, Philadelphia, PA., April 1984.

"Elevated Temperature Properties of a Powder-Processed Al-Ni-Fe Alloy", International Conference on Powder Metallurgy, Toronto, Canada, June 1984.

"Thermal Stability and Mechanical Behavior of Cold Sintered P/M Aluminum Alloys", International Conference on Powder Metallurgy, Toronto, Canada, June 1984.

"Overview of Atomization", International Conference on Powder Metallurgy, Toronto, Canada, June 1984.

"Powder Metallurgy Aluminum Alloys", Toyota Central Research Laboratory, Nagoya, Japan, August 1984.

"Powder Metallurgy Aluminum Alloys", Mitsubishi Metal Research Institute, Omiya, Japan, August 1984.

"Aluminum Powder Processing in Japan", Alcoa Technical Center, Alcoa Center, PA, September 1984.

"Dispersion - Strengthened Aluminum Alloys for Elevated Temperature Applications", Metal Powder Industries Federation, Seminar on P/M in Defense Technology, Picatinny Arsenal, Rockaway, N.J., September 1984.

b) Technical Contacts with Other Laboratories

Both principal investigators have interacted with other research personnel engaged in similar and related research in industry, government and academia. Contacts include:

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