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A FUNDAMENTAL STUDY OF P/M PROCESSED ELEVATED  
TEMPERATURE ALUMINUM ALLOYS. (U) DREXEL UNIV  
PHILADELPHIA PA DEPT OF MATERIALS ENGINEERING  
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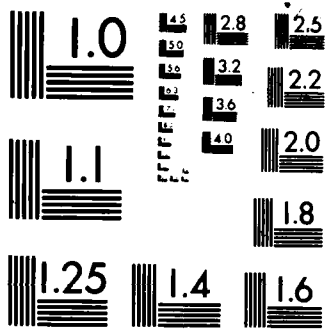
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ANNUAL TECHNICAL REPORT

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

AFOSR Grant #82-0010

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

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March 1984



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## ABSTRACT OF RESULTS

Powder processed Al-Fe-Ni alloys containing  $(\text{FeNi})\text{Al}_9$  dispersoids in the range 0.2-0.3 volume fraction are being examined with respect to processing mode, microstructure, microstructural stability and elevated temperature tensile and creep response. The overall objective is to establish a comprehensive and quantitative fundamental understanding of processing-microstructure relations in this new class of alloys in order to provide design guidelines with respect to service stresses and temperatures. To date, microstructural characterization, dispersoid stability, hot tensile and creep resistance have been characterized in the Al-5.9 w/o Fe-6.1 w/o Ni alloy ( $V_f = 0.32$ ) after consolidation by hot pressing + extrusion. These data and observations confirm the benefits of rapid solidification, promising levels of structural stability, and potential for long-time service at temperatures up to  $250^\circ\text{C}$  ( $482^\circ\text{F}$ ). Using the L-S-W theory, it is possible to predict coarsening kinetics over a wide range of temperatures. Similar studies are in progress on Al-4.2 w/o Fe-3.9 w/o Ni ( $V_f = 0.19$ ) and Al-4.9 w/o Fe-4.8 w/o Ni ( $V_f = 0.25$ ).

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## INTRODUCTION

Aluminum alloys exhibiting high strength and improved creep resistance at elevated temperatures offer the potential for lower weight and reduced costs in aerospace components such as structural members, engine parts, leading edge wing skins and heat exchangers (1-6). A primary goal of the Air Force is to develop aluminum-base alloy compositions for use over the temperature range 230-340°C (450-650°F).

Previous attempts to upgrade the elevated temperature performance of aluminum alloys have been based on composition, processing and heat treatment optimization of ingot metallurgy (I/M) alloys. Prime examples are the precipitation hardening alloys such as 2219 and 2618 (7). Unfortunately, these alloys are limited to service temperatures below 177°C (350°F) since precipitate coarsening and loss of strength occur at higher temperatures.

Utilizing powder metallurgy (P/M) processing science and technology, it is possible to achieve a high volume fraction of stable, fine, uniformly dispersed intermetallic phases in the matrix (4,5,8). These provide dispersion hardening and elevated temperature microstructural stability. The powder processing approach involves atomization followed by hot consolidation (4,5,8). Several P/M processed alloys are being evaluated (4-6) based on transition metal additions to aluminum; these elements exhibit moderate to high liquid solubility, low solid solubility, and a low rate of solid state diffusion in aluminum. Of this multi-element matrix, the Al-Fe-Ce and Al-Fe-Ni alloys are considered prime contenders for scale-up and development.

## THE PRESENT PROGRAM

Notwithstanding the several R and D programs cited above, only a limited understanding exists of the relationships between composition, processing mode,



microstructure, properties and performance in these dispersion-strengthened P/M processed aluminum alloys (2,4,5,9-11). It is the overall objective of the present study to develop such fundamental relationships. This scientific base of knowledge 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 included 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. Previous work on Al-Fe-Ni (5) has given some insight into the influence of processing mode, powder particle morphology, particle size distribution and particle bonding integrity on strength, ductility and toughness.

Procedures, results and observations, and important implications covering the second year of the study are summarized in this report.

#### PROGRAM SUMMARY

##### (a) Procedures

Powders were air atomized at Alcoa; for each of the three compositions included, the size distribution was approximately 95% by weight less than 44  $\mu\text{m}$ . Powder surfaces and internal structure were characterized by means of SEM and optical microscopy. Powders were then exposed 1 hour at temperatures up to 600°C (1112°F) to assess microstructural stability and hardness retention. Dispersoid morphology and the distribution of Fe and Ni in the as-atomized and annealed powder were characterized by means of TEM/STEM. Electron

transparent foils were prepared from the powder utilizing the technique of cold sintering (12). Dispersoid coarsening and the precipitation of Fe and Ni from the matrix were followed by means of differential scanning calorimetry (DSC). Phase identification and monitoring of the precipitation process were accomplished by means of x-ray diffraction.

Powders were consolidated to full density by one of two routes, namely vacuum hot pressing followed by extrusion, or extrusion of the canned powder; details are given in Figure 1. The consolidated material was then exposed for 1 hour periods up to 600°C (1112°F). Characterization of the consolidated powder material is in progress, utilizing quantitative optical microscopy, TEM/STEM and DSC.

To assess microstructural stability, elevated temperature tensile response and creep behavior are being studied up to 400°C (752°F). Micro and macrohardness are also measured at ambient following elevated temperature exposure.

#### (b) Results and Observations

Data and observations presented in this progress report are for the Al-5.9 w/o Fe-6.1 w/o Ni composition in the form of powder, after hot pressing, and following extrusion. Work is in progress on each of the three compositions after canning + extrusion.

##### (1) Powders

A portion of the x-ray diffraction patterns from as-atomized powder, and from powder after 1 hour at 500°C (932°F) are shown in Figure 2. The diffraction lines shown are from the dispersoid only. As a result of elevated temperature exposure the lines become much sharper and stronger, with an increase in area under each peak. The d-spacings of the dispersoid, calculated from the diffraction pattern, are summarized in Table I.

Corresponding d-spacings for  $\text{Co}_2\text{Al}_9$  (ASTM Index Card #6-0699) are included in Table I; this intermetallic is of the type  $\text{M}_2\text{Al}_9$ .

It has previously been shown that the as-atomized powder exhibits a duplex microstructure of fine and coarse  $(\text{FeNi})\text{Al}_9$  dispersoids in the aluminum matrix (13,14). The morphology of the fine structure is illustrated in Figure 3; a representative energy dispersive spectrum (EDS) from the dispersoid is included in Figure 3. Approximately equal weight percentages of Fe and Ni are present. The EDS from the matrix of the as-atomized powder is given in Figure 4(a), and from the matrix of annealed powder in Figure 4(b). Significant amounts of Fe and Ni are present in the matrix of the as-atomized powder. Some Fe and Ni is retained in the matrix after annealing at  $400^\circ\text{C}$  ( $752^\circ\text{F}$ ), Figure 4(b).

A representative DSC trace of the as-atomized powder is shown in Figure 5(a). After heating to  $600^\circ\text{C}$  ( $1112^\circ\text{F}$ ) the sample was cooled to ambient and then re-heated. The corresponding DSC trace is designated as the 'base-line' since it approximates equilibrium conditions. The broad exothermic peak around  $450^\circ\text{C}$  ( $842^\circ\text{F}$ ) suggests that precipitation is occurring.

#### (ii) Consolidated Material

DSC traces of the hot pressed and hot pressed + extruded material are given in Figures 5(b) and 5(c). After hot pressing the DSC trace still exhibits a peak at approximately  $480^\circ\text{C}$  ( $896^\circ\text{F}$ ). In the extruded condition, there is no peak, but only a deviation from the base-line trace.

Results of quantitative metallography on the hot-pressed + extruded material are given in Figure 6. These show the dependence of dispersoid size distribution on temperature and time. The average measured volume fractions of dispersoid for all the times examined were 0.313 and 0.325 after exposure at  $550^\circ\text{C}$  ( $1022^\circ\text{F}$ ) and  $600^\circ\text{C}$  ( $1112^\circ\text{F}$ ) respectively. Similarly,

quantitative metallography of each of the three alloy compositions in the canned + extruded material is in progress.

Hot tensile data on the hot-pressed + extruded Al-5.9 w/o Fe-6.1 w/o Ni were summarized in a previous report (13). A detailed evaluation of the elevated temperature tensile response of the three alloy compositions in the canned + extruded condition is in progress. This includes a characterization of deformation microstructures (TEM/STEM) and associated fracture morphologies.

To date, constant load creep tests have been conducted on the Al-5.9 w/o Fe-6.1 w/o Ni alloy after hot pressing + extrusion. The creep tests were run at 250°C (482°F), 350°C (662°F), 375°C (707°F) and 400°C (752°F); stress levels were in the range 5-30 ksi (35-207 MPa). Data for the highest and lowest creep test temperatures are summarized in Table II. A comparison of the creep curves, as a function of stress level and temperature, is given in Figure 7. Similar tests are in progress on each of the three alloy compositions in the canned + extruded condition. A concurrent in-depth characterization of creep response in terms of microstructures (optical microscopy, TEM/SEM) and fracture mode/mechanisms (SEM) is in progress.

(c) Interpretation and Significance of Results

From the EDS and x-ray diffraction studies, it is concluded that the dispersoid in both powder and consolidated forms is the intermetallic (FeNi)Al<sub>9</sub>. The observed d-spacings compare closely with those of Co<sub>2</sub>Al<sub>9</sub> since Fe, Ni and Co are interchangeable transition elements of similar atomic size.

Rapid solidification of the powder particles during atomization results in increased matrix solid solubility for Fe and Ni. A combination of DSC and EDS confirms that the solubility levels of these elements decrease on exposure above ambient.

The fine-scale microstructure achieved in the powder particles can be explained in terms of undercooling and the associated nucleation and growth kinetics (15). For purposes of comparison, a piece of the extruded alloy was re-melted and cooled slowly. The resulting microstructure was extremely coarse and consisted of large needles of  $\text{FeNiAl}_9$ ; in addition,  $\text{FeAl}_3$  and a coarse eutectic structure ( $\text{Al}$ ,  $\text{NiAl}_3$  and  $\text{FeNiAl}_9$ ) were present. Thus, it is only possible to achieve the fine-scale dispersoid by rapid solidification.

The data obtained by quantitative metallography are being analyzed in light of the L-S-W theory (16,17) of dispersoid coarsening kinetics. While the theory has several important constraints, it does provide a basis for predicting an upper bound for service temperature.

Creep data are currently being analyzed to determine activation energy for the creep mechanism, stress exponent, and the dependence of rupture time on stress level. The concurrent characterization of creep deformation substructure allows for an understanding of operative creep mechanisms. A preliminary comparison of the present creep data for extruded material with that of an Al-Fe-Ni alloy containing 0.246 volume fraction of dispersoid consolidated by hot-pressing + forging (5), reveals similar steady-state creep rates and overall creep response. Similarly the hot tensile response following both modes of consolidation is comparable.

In total, this on-going study is providing a comprehensive and quantitative scientific base with which to understand and utilize the interplay of composition, powder processing, microstructure and mechanical properties in the Al-Fe-Ni system. This will provide design guidelines in terms of processing and service temperatures. Because of the fundamental nature of the information gained in this study, it should be of general utility with respect to aluminum-base high temperature alloys.

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Table I: Dispersoid d-Spacings in As-Atomized Powder

<u>Observed d (Å)</u>	<u>Co<sub>2</sub>Al<sub>9</sub></u> <sup>*</sup>
5.082	5.07
4.428	4.41
4.278	4.27
4.034	4.019
3.836	3.829
3.678	3.654
3.543	3.525
3.384	3.379
3.105	3.095
2.961	2.948
2.710	2.693
2.589	2.584
2.471	2.458
2.425	2.513
2.394	2.385
2.305	2.303
2.253	2.251
2.174	2.168
2.142	2.131

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\* ASTM Index Card #6-0699.

Table II: Selected Creep Data

<u>Temperature</u>	<u>Stress</u>		$\dot{\epsilon}_s^*$	$t_r^{**}$
	ksi	(MPa)	$\text{sec}^{-1}$	min.
250°C (482°F)	15	(103)	$8.94 \times 10^{-9}$	(1)
250°C (482°F)	22	(152)	$8.03 \times 10^{-7}$	682
250°C (482°F)	25	(172)	$2.25 \times 10^{-6}$	204
250°C (482°F)	27	(186)	$8.61 \times 10^{-6}$	74
400°C (752°F)	5	(35)	$1.82 \times 10^{-8}$	21,362
400°C (752°F)	6.5	(45)	$1.24 \times 10^{-6}$	606
400°C (752°F)	8	(55)	$1.07 \times 10^{-5}$	78

(1) Test discontinued - no failure.

\* Steady-state creep rate

\*\* Rupture time.



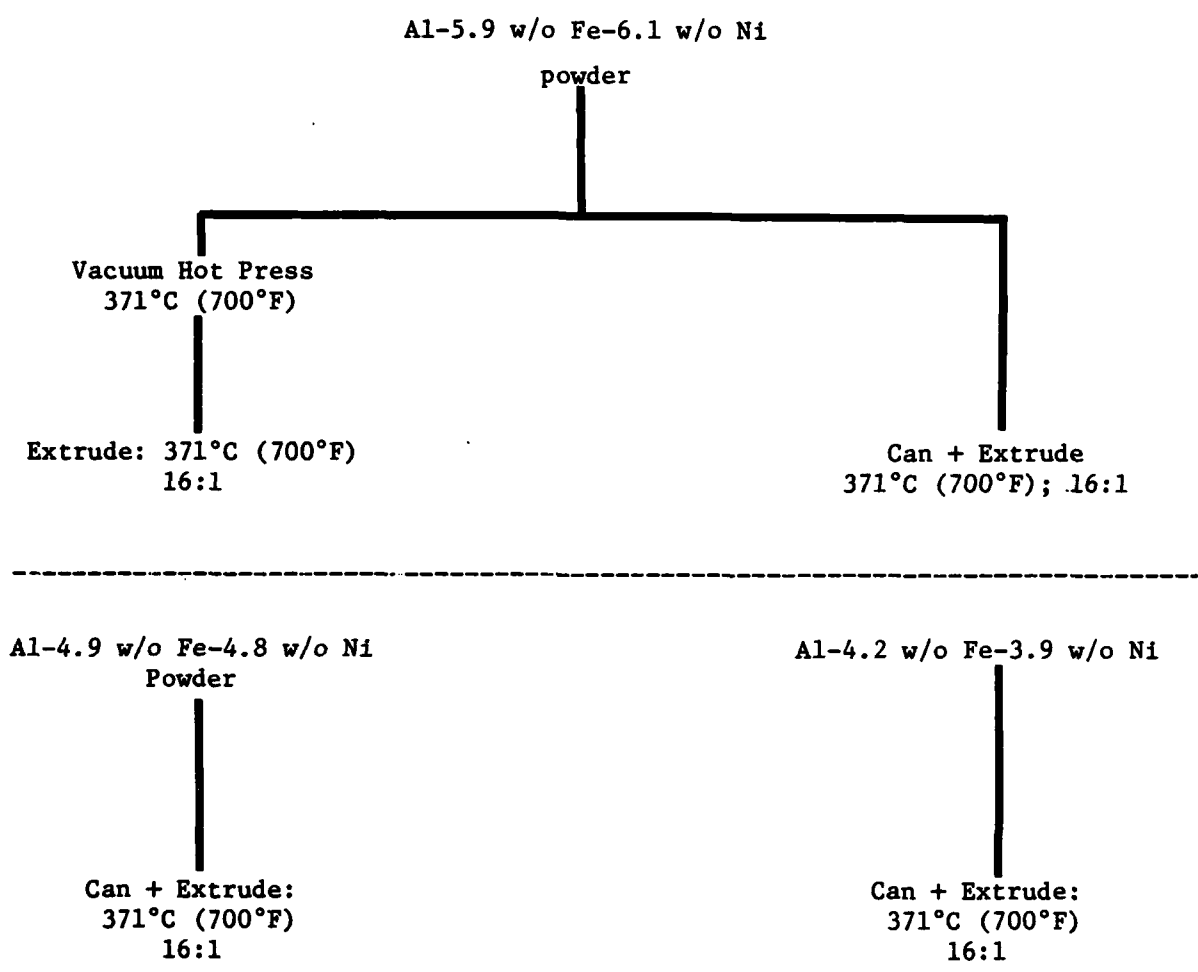
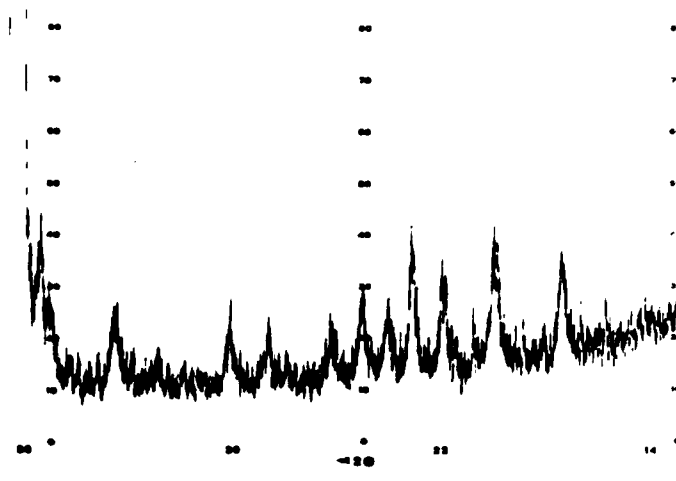
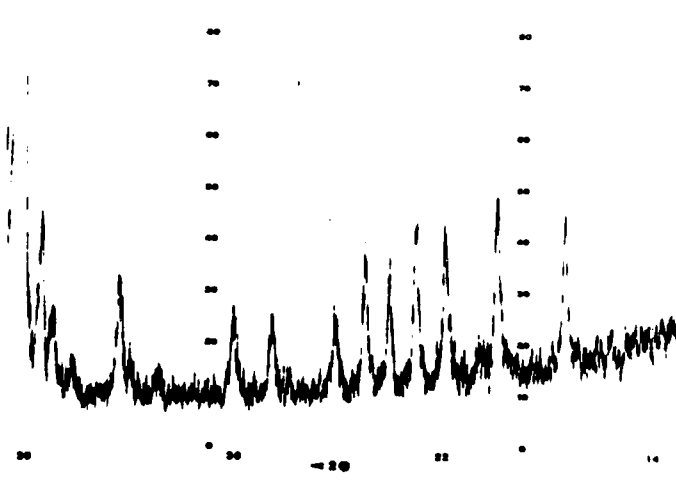


Figure 1: Schematic showing powder consolidation.

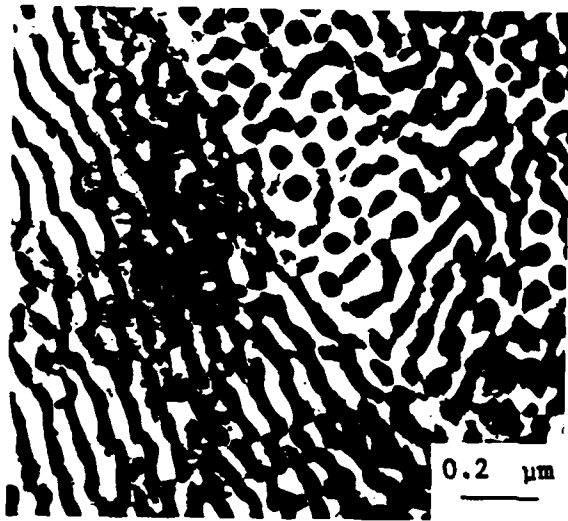


(a)



(b)

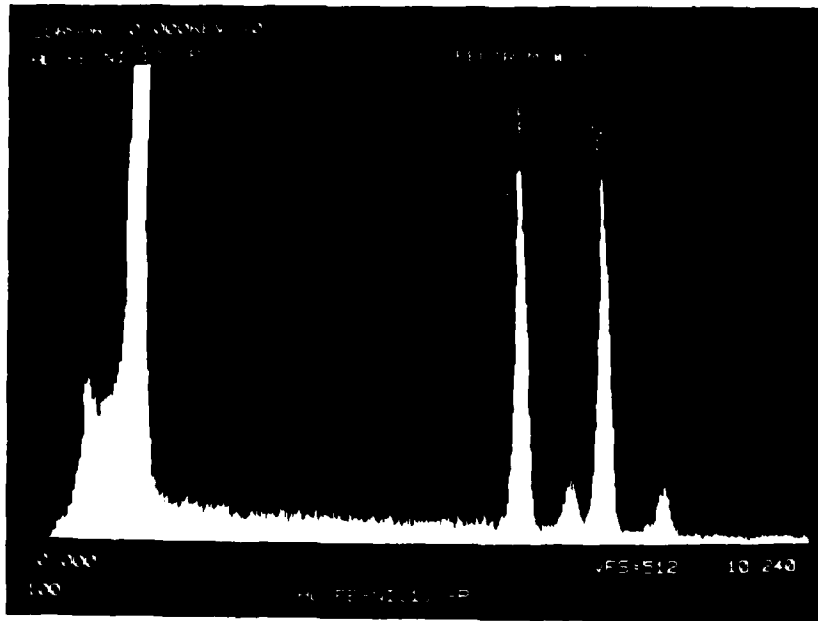
Figure 2: X-ray diffraction traces showing some of the intermetallic lines. (a) as atomized powder, (b) after exposure at 500°C.



(a)

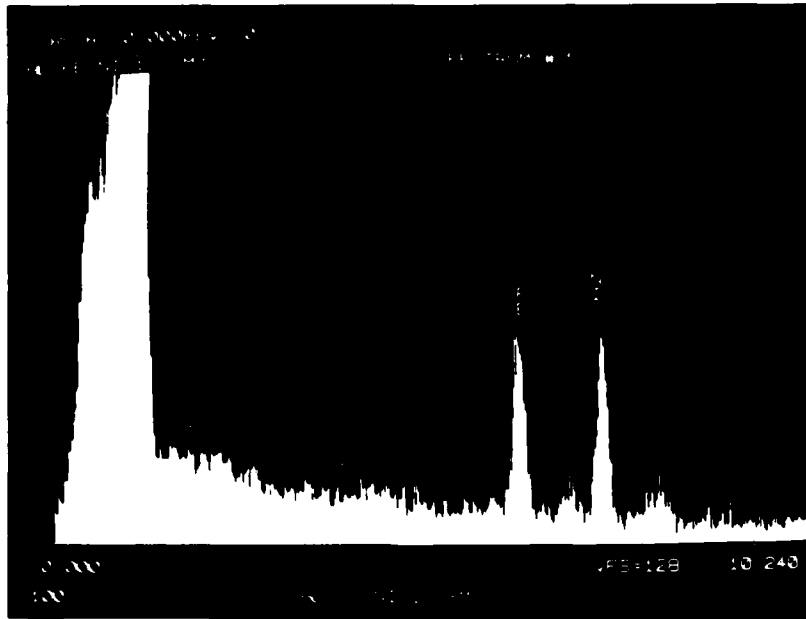


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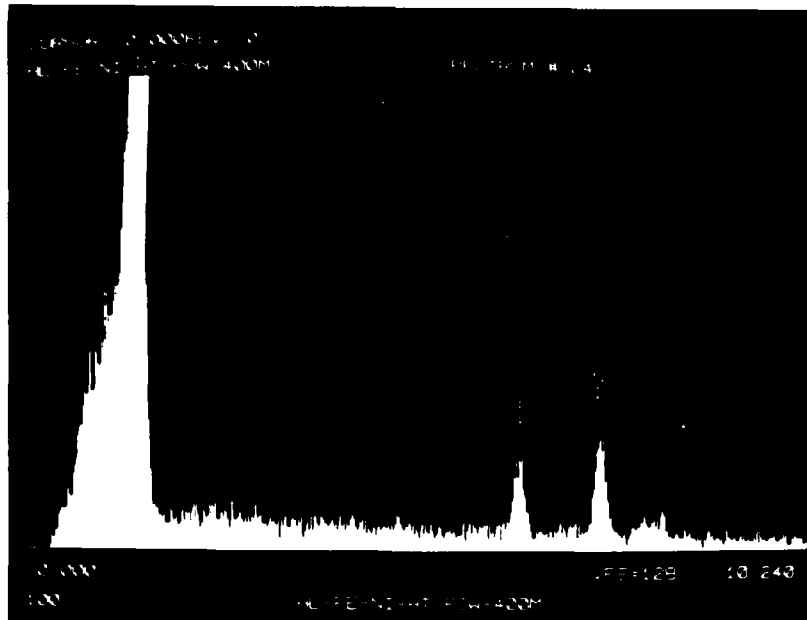


(c)

Figure 3: (a) and (b) - TEM of fine structure in atomized powder;  
(c) EDS of the dispersoid.



(a)



(b)

Figure 4: EDS from the matrix of the powder (a) as-atomized; (b) after exposure at 400°C (752°F) for 1 hour.

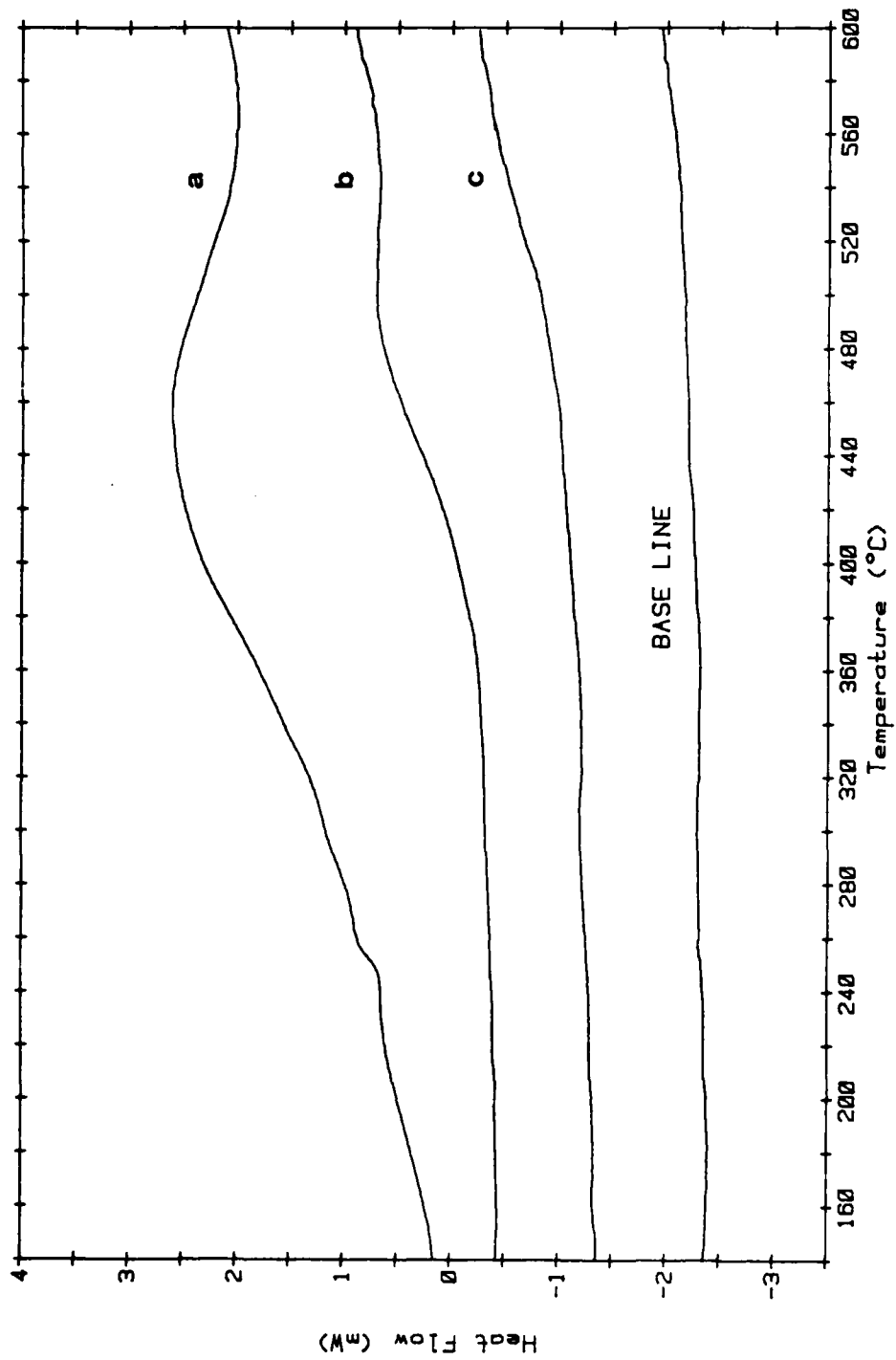


Figure 5: DSC traces, (a) as-atomized powder, (b) hot pressed, (c) hot pressed + extruded.

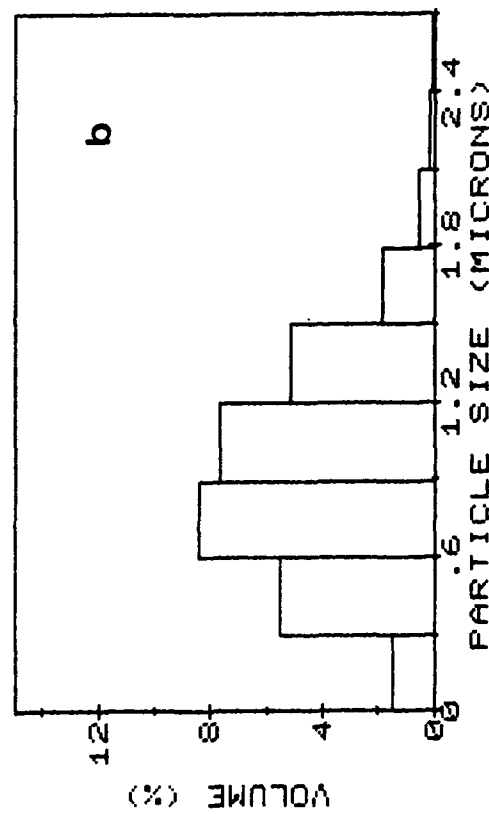
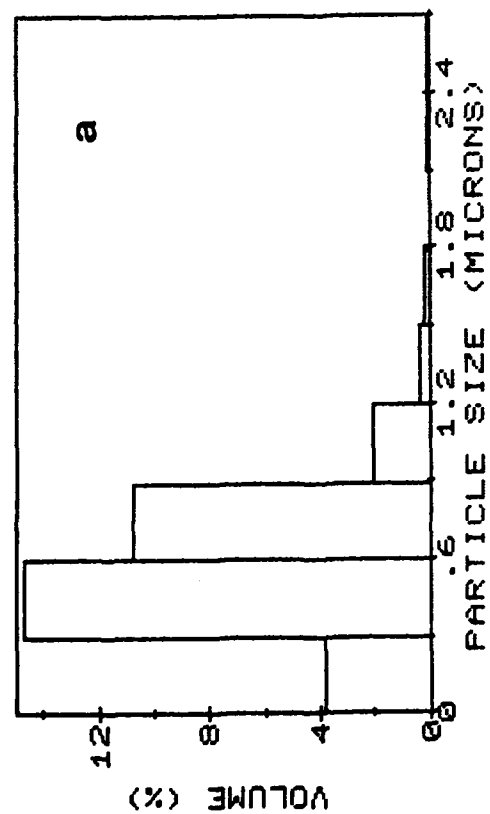
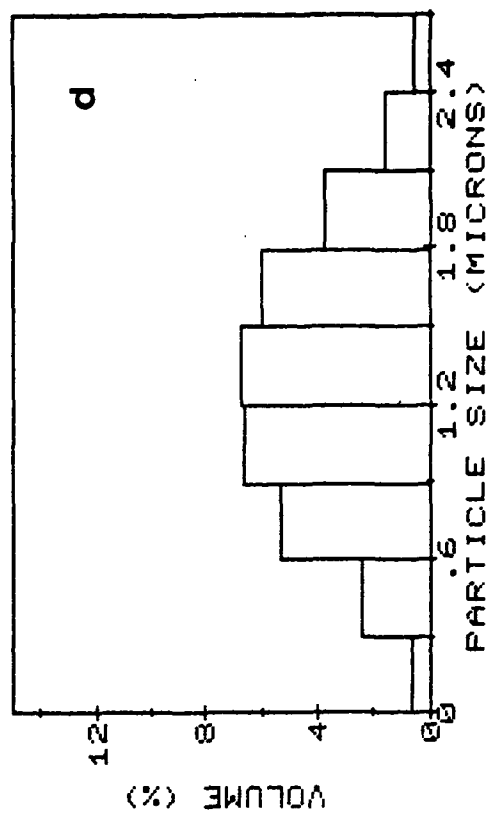
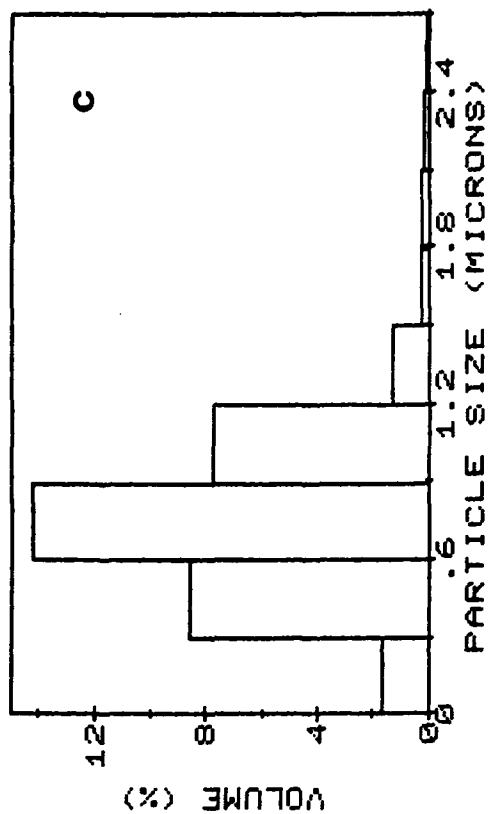


Figure 6: Dispersoid size distribution by volume in hot-pressed + extruded material. (a) 550°C (1022°F)/5 hours; (b) 550°C (1022°F)/166 hours; (c) 600°C (1112°F)/5 hours; (d) 600°C (1112°F)/100 hours.

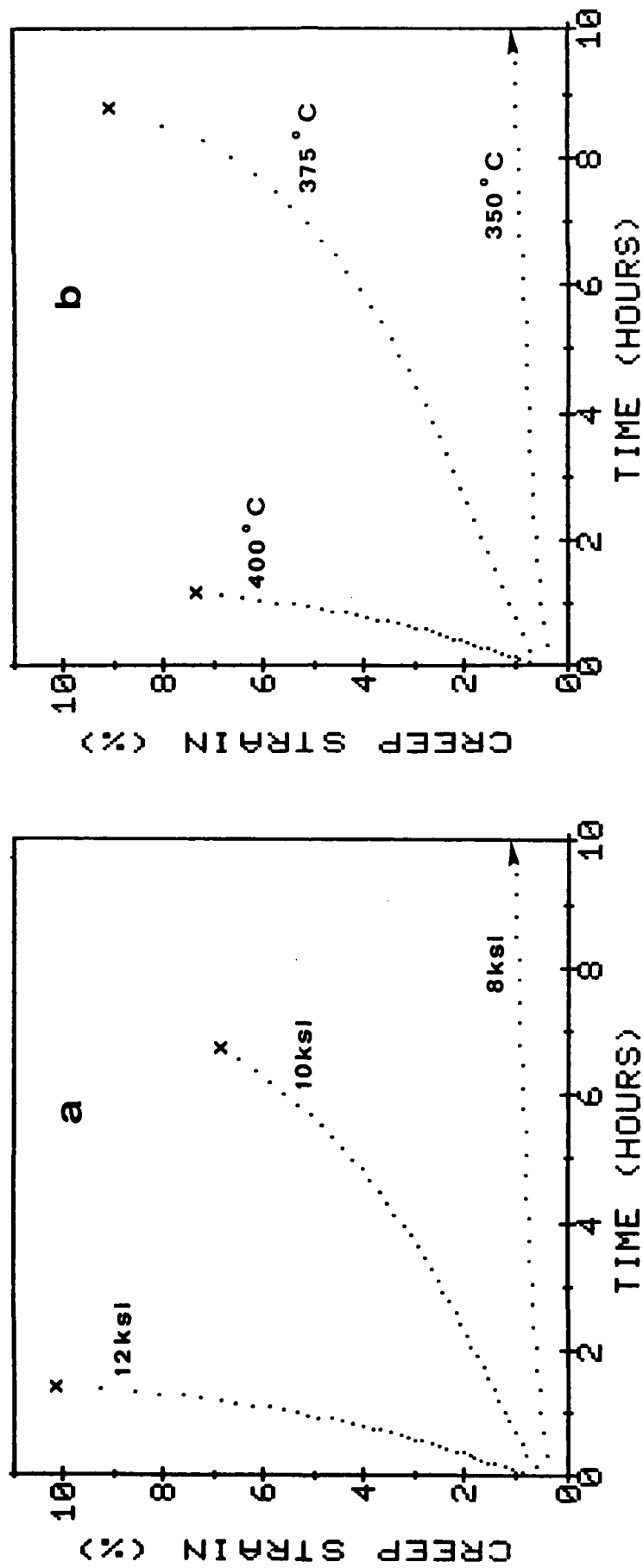


Figure 7: Creep response of hot-pressed + extruded material. (a) Effect of stress level at 350°C (662°F); (b) Effect of temperature at 8 ksi (55 MPa).

## PUBLICATIONS

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Lawley, A., "Aluminum Alloy Powder Consolidation", in Rapidly Solidified Powder Aluminum Alloys, ASTM STP, in press.

"Elevated Temperature Stability of Powder Metallurgy Al-Fe-Ni Alloys", M. Premkumar, A. Lawley and M.J. Koczak, submitted to Met. Trans.

"Elevated Temperature Microstructural Stability and Properties of P/M Al-Fe-Ni Alloys", M. Premkumar, Ph.D. Dissertation, in progress.

## PERSONNEL

A. Lawley - Professor and Co-Principal Investigator

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

M. Premkumar - Ph.D. Student



## COUPLING ACTIVITIES

### a) Presentations - Lawley

"Powder Metallurgy and Rapid Solidification Technology", Case Western Reserve University, Cleveland, Ohio, October 1982.

"Rapid Solidification and Powder Metallurgy Processing Technology", Detroit AIME Section, November 1982.

"Powder Metallurgy and Rapid Solidification Technology", Arkansas/Louisiana/Texas Chapter ASM, Shreveport, Louisiana, November 1982.

"Powders and Powder Production", TRW, Cleveland, Ohio, November 1982.

"Cold Sintering - A New Powder Consolidation Process", MPIF Annual Meeting, New Orleans, Louisiana, May 1983.

"Microstructure and Mechanical Properties of a Cold Sintered P/M Aluminum Alloy", MPIF Annual Meeting, New Orleans, Louisiana, May 1983.

### Presentations - Koczak

"Fatigue in P/M Aluminum Alloys"

- "Powder Metallurgy Processing"

"Fundamental Concepts of P/M"

These lectures were given between October 1982 and September 1983 at various universities, industrial and government laboratories in Australia, China, Japan and Korea, while Professor Koczak served as the ONR Scientific Liaison Officer in the Far East.

### 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:

Alcoa Technical Center - F.R. Billman, W.S. Cebulak,  
H.G. Paris, G.J. Hildeman

AFML/AFWAL - A.M. Adair, W.M. Griffith

NADC - J.J. DeLuccia, G.J. London, J. Waldman

Northwestern University - M.E. Fine, J.R. Weertman

Purdue University - T.E. Sanders

University of Virginia - E.A. Starke, Jr.

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