Technical Report 1500
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Analysis of OSPREY Processes and Conventional Powder Aluminum Alloys

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ADMINISTRATIVE INFORMATION

This work was performed by the Structural Materials Science Branch (Code 932), RTD&E Division of the Naval Command, Control and Ocean Surveillance Center.

Released by
R. K. Fogg, Jr., Head
Structural Materials
Science Branch

Under authority of
C. L. Ward, Jr., Head
Design and Development
Division

JT

EXECUTIVE SUMMARY

OBJECTIVE

Investigate the amount and morphology of oxides and other impurity particles present in samples of a nonstandard 7000 series aluminum alloy made by the OSPREY process and by a conventional powder process. Bulk chemistry, oxygen analysis, metallography, optical and scanning electron microscopy (SEM), and localized energy dispersive x-ray spectroscropy (EDS) were employed in the analysis.

RESULTS

Metallography shows the presence of oxides surrounding the original powder particles of the conventional alloy, but no oxides in the OSPREY alloy. This presence of oxides was confirmed by oxygen analysis which found <0.0005 percent oxygen by weight for the OSPREY alloy and 0.0857 percent by weight oxygen for the conventional alloy. The as-received OSPREY alloy did contain hard particles of meta-stable $FeAl_6$.

CONCLUSIONS

The OSPREY process does result in the formation of fewer oxide impurity particles than conventional powder metallurgical processes.

CONTENTS

FIGURES

TABLES

ANALYSIS

Samples of a nonstandard 7000 series aluminum alloy made by the OSPREY process and by a conventional powder process were analyzed in the Structural Materials Science Branch (Code 932) materials laboratory. The OSPREY process produces high-purity alloys with a spray-deposition, rapid-solidification process carried out in a protective atmosphere. An advantage over conventional powder alloys is the substantial reduction of oxides at the powder particle interfaces. The intent of the analysis was to investigate the amount and morphology of oxides and other impurity particles present. Bulk chemistry, oxygen analysis, metallography, optical and scanning electron microscopy (SEM), and localized energy dispersive x-ray spectroscopy (EDS) were employed in the analysis.

The OSPREY material had been conventionally extruded at a 10- to -1 reduction ratio. The powder alloy had been back extruded at a reduction ratio of something greater than 10 to 1. The samples appeared to be pieces of rectangular bar stock. Both samples have a reported nominal composition of 5.4-percent zinc (Zn), 2.1-percent magnesium (Mg), and 0.25- percent zirconium (Zr).

Bulk chemistry was determined via Inductively Coupled Plasma (ICP) emission spectroscopy on aerosol produced by a conductive solids nebulizer (CSN). The CSN is a standard-spark source that erodes material from solid samples for introduction into the ICP. This analysis method is particularly useful for alloys for which standard optical emission calibrations are not available or the alloy type is initially unknown. The ICP is calibrated with multiple aluminum standards (from Alcoa) from a number of different aluminum alloys. Only one standard with Zr was available, so the Zr content was estimated from a two-point calibration based on Alcoa's SS7050 standard (0.11-percent Zr certified). These estimates are the result of extrapolation and due to the form of the calibration curves, will represent lower bounds of the Zr concentration in these samples. The measured elemental compositions are presented as weight percentages in table 1.

Table 1. Measured elemental compositions.

	Zn	Mg	Zr	Fe	Si	Mn	Cu	Cr	Ti	Be
OSPREY	5.17	2.00	0.21	0.160	0.01	0.000	0.000	0.002	0.00	0.000
Conventional	5.59	2.14	0.27	0.024	0.04	0.001	0.026	0.000	0.00	0.000

The impurity contents of both alloys are quite low indicating that the aluminum ingot was of considerably higher purity than ordinary commercial grade. Because of the overall purity, the iron-silicon-aluminum intermetallic compounds that are typical of commercial purity alloys would not be present. Some iron aluminum intermetallics are expected in the OSPREY alloy because of the relatively high iron content. Figure 1 shows the binary phase diagrams of aluminum and the alloying elements, and figure 2 is an isotherm from the Al-Mg-Zn ternary diagram. The alloy contains four constituents (five if iron is considered in the OSPREY alloy) so exact determination of the phases is difficult. Some idea of the microstructure, however, can be predicted from the phase diagrams.

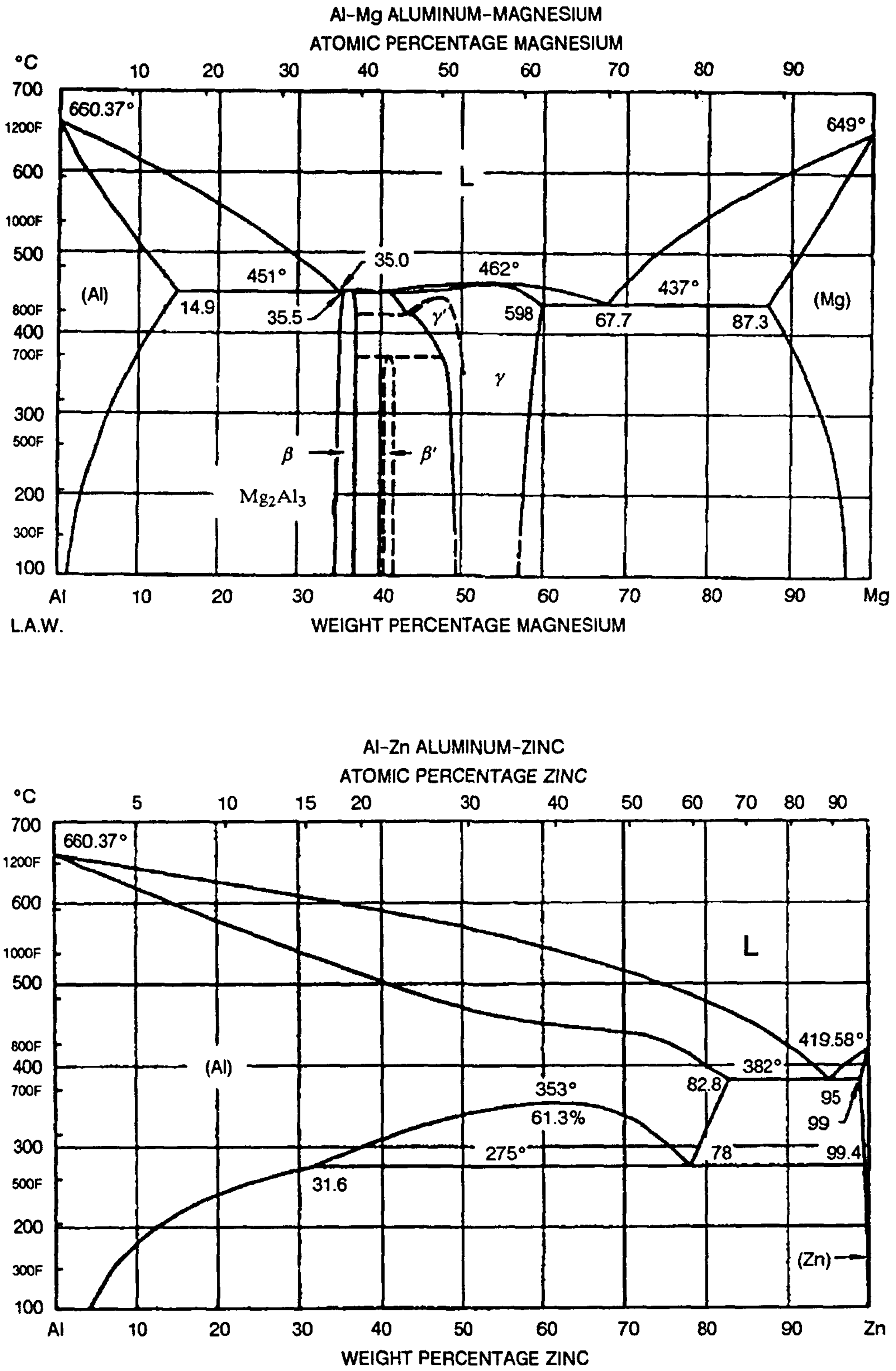


Figure 1. Binary phase diagrams.

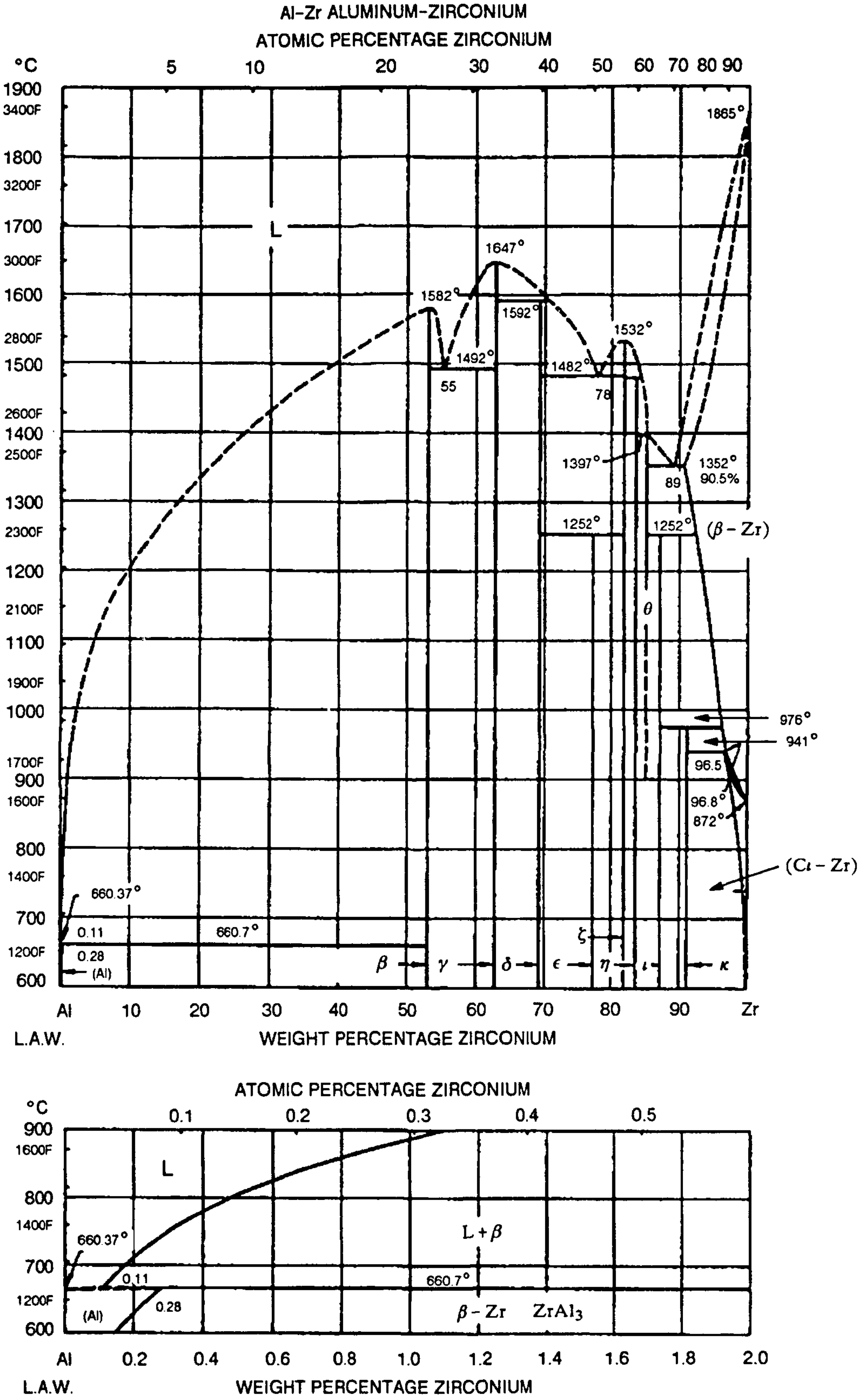


Figure 1. Binary phase diagrams. (continued)

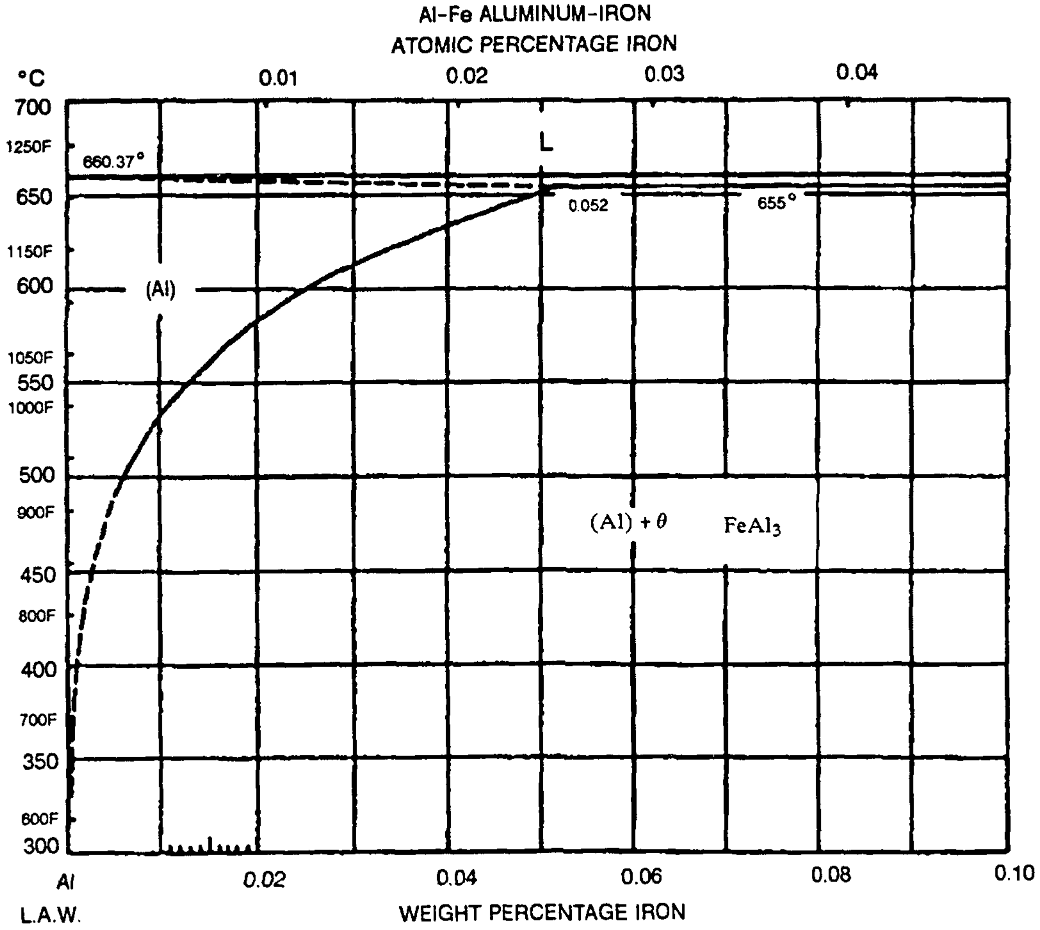


Figure 1. Binary phase diagrams. (continued)

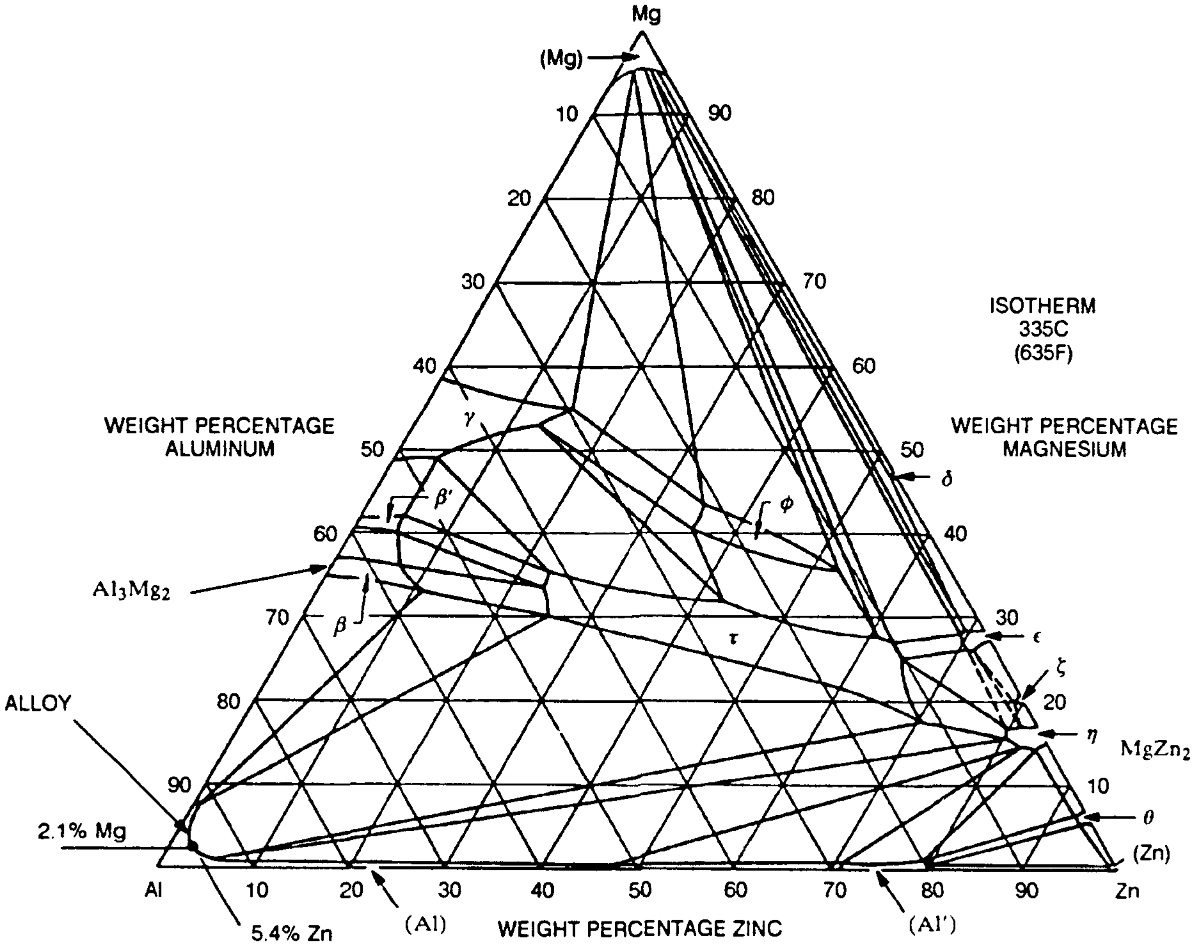


Figure 2. Al-Mg-Zn (Aluminum-Magnesium-Zinc).

The Al-Mg-Zn ternary alloy forms a quasibinary system between aluminum and $MgZn_2$ and has a pseudobinary eutectic reaction in it. The Mg- to Zn-atomic ratio is 1-to-1 in this alloy so $MgZn_2$ precipitate would be expected. The excess Mg may remain in solution, form Mg_2Al_3 intermetallics, or form into a ternary-intermetallic compound. The ternary-phase diagram indicates that the alloy is a solid solution above about 400°C so the alloy can be solution annealed, quenched to retain the solid solution, and age hardened.

Zirconium is added to 7005 aluminum to reduce stress corrosion susceptibility and that is probably its intended purpose here. The Al-Zr phase diagram indicates that Zr at a concentration of 0.25 percent will dissolve at a solution anneal temperature and may add to the age-hardening effect. At the lower aging temperatures, it forms $ZrAl_3$. $ZrAl_3$ is reported to be coherent with the aluminum phase and may not be visible under ordinary light microscopy.

Iron is generally an impurity in aluminum alloys and is present in the OSPREY alloy. It forms $FeAl_3$ under equilibrium conditions but, the metastable $FeAl_6$ phase will form in rapidly cooled alloys. Iron can be dissolved in aluminum at ordinary solution anneal temperatures, although it does not have a significant age-hardening effect by itself. The form or shape of the intermetallic compounds would be expected to change with higher temperature heat treatments.

Three perpendicular sections of each sample were mounted and metallographically polished. Figure 3 shows a longitudinal section of each alloy in the as-polished condition at 800X. In the as-polished condition, "hard" second phases such as intermetallic compounds or oxides can usually be observed. The OSPREY alloy has a considerable amount of irregular hard phase in the form of light grey stringers in the longitudinal direction. The conventional powder alloy shows almost no evidence of hard compounds at this magnification. The general matrix shows evidence of a dispersion of very fine particles; however, this may be a polishing artifact.

The as-polished samples were examined in a scanning electron microscope (SEM). No uniformly distributed second phases were found in the conventional alloy. The hard phase in the OSPREY alloy was easily located. Figure 4 is a secondary electron image, and the second phase shows up as the bright stringers. Figure 5 is an EDS spectrum of the second phase and indicates that it is an iron aluminum intermetallic compound. A standardless analysis program computed the atomic aluminum-to-iron ratio as 6 to 1, identifying the compound as the metastable $FeAl_6$.

The metallographic samples were etched with Keller's etch to reveal the general structure. Figures 6 and 8 are composite photomicrographs of the alloys showing the grain structure in the three perpendicular planes. Figures 7 and 9 are high-magnification photomicrographs of the three different metallographic sample planes for each sample. Both alloys have an apparent elongated and flattened grain structure typical of an extrusion with a rectangular bar or a tubular configuration.

In the OSPREY material, the $FeAl_6$ particles are darkly etched as shown in figure 6. In figure 7, shown at high magnification, the OSPREY material has very cleanly etched grain boundaries showing the elongated structure. Depending on the grain orientation, some grains etch more than others, and appear less bright. The grain boundaries are quite irregular, but this is typical of a 7000 series aluminum extrusion. Inside the large elongated grains is a very fine subgrain structure. This type of structure is reported in 7000 series alloys and is believed to be an effect of a dispersion of very fine precipitation, most likely $ZrAl_3$ in this case. The subgrains could be due to subtle differences in alloy composition that etch to form this structure; possibly a type of "coring" effect resulting from the OSPREY forming process.

What appear at low magnification to be grain boundaries in the conventional powder alloy are actually a delineation of the original powder particle structure. The rounded shape of the particles is apparent in the frontal view of figure 8. In figure 9, at higher magnification, it is apparent that the particles are outlined by a second-phase network. This etched sample was examined in the SEM and the second-phase network was identified as a string of etch pits. An EDS spectrum of the surface inside the pits revealed a typical spectrum for the bulk alloy. Whatever material was at the etch-pit site, has been

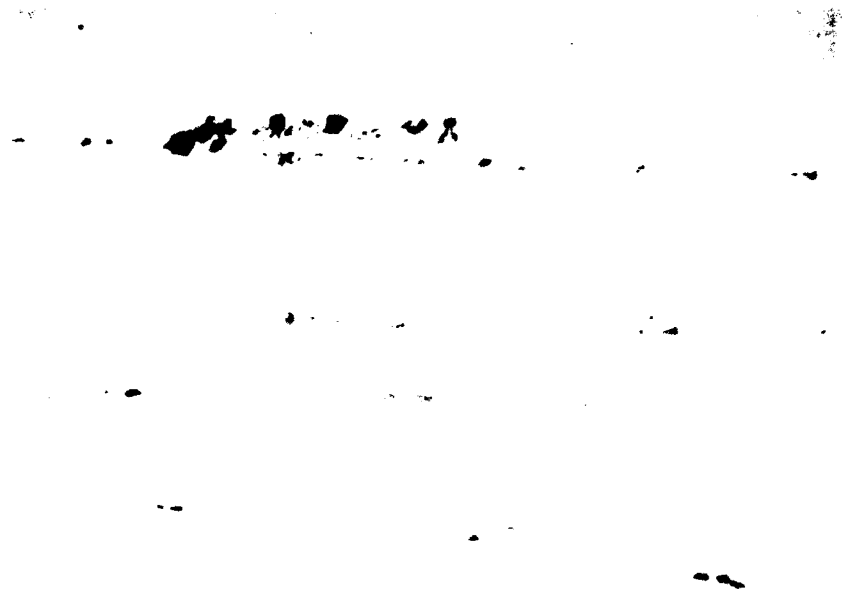
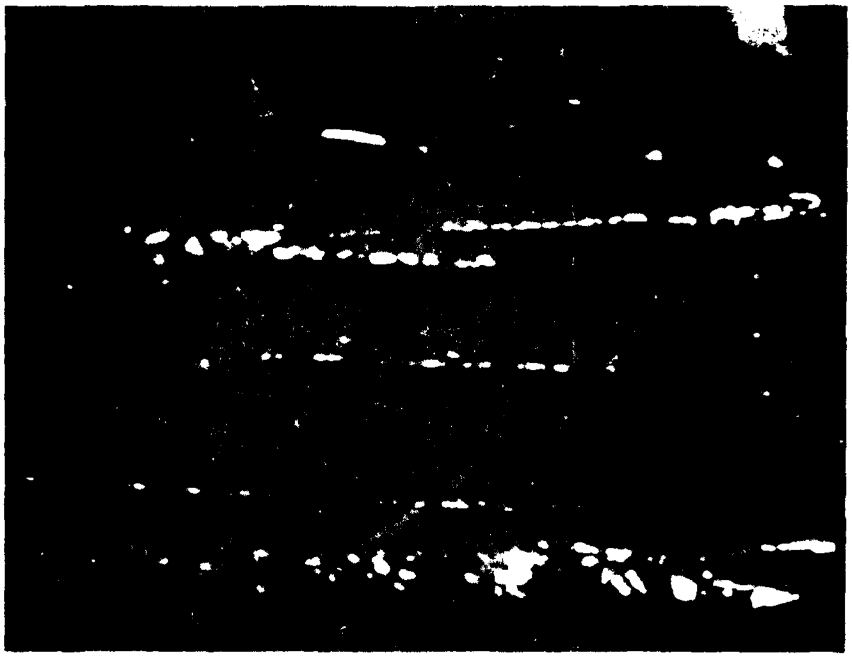


Figure 3a. OSPREY alloy, as polished, "hard" phase particles.

Figure 3b. Conventional powder alloy, as polished.



1200X

Figure 4. OSPREY alloy, SEM image of "hard" phase particles

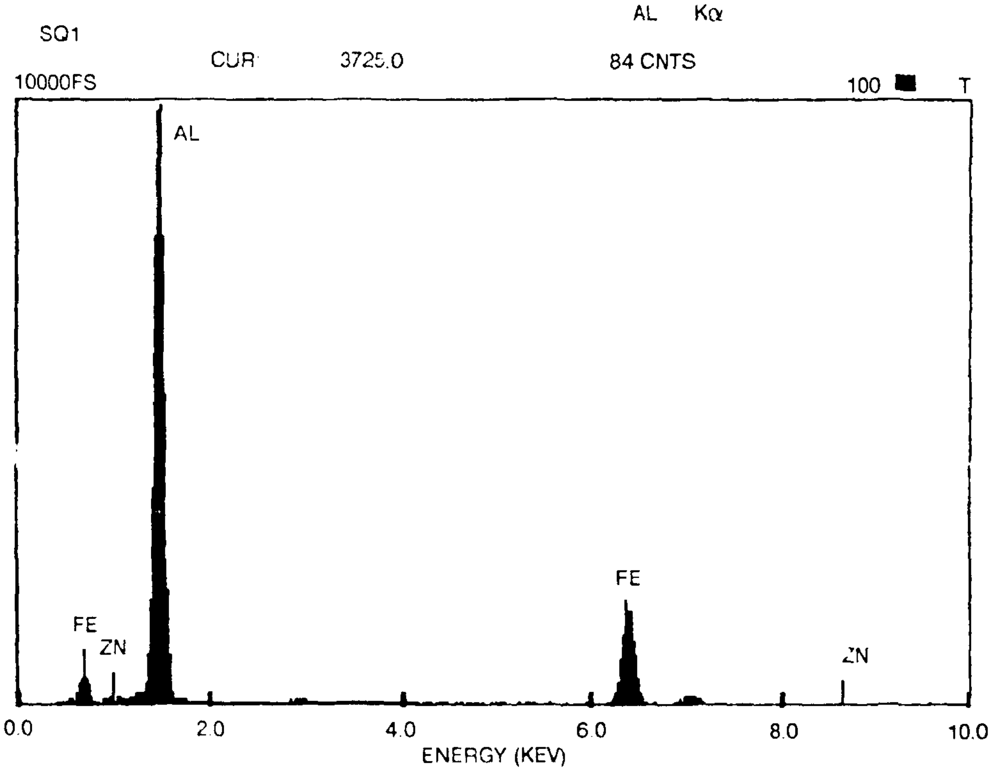


Figure 5. EDS spectra of "hard" phase, identified as $FeAl_6$.

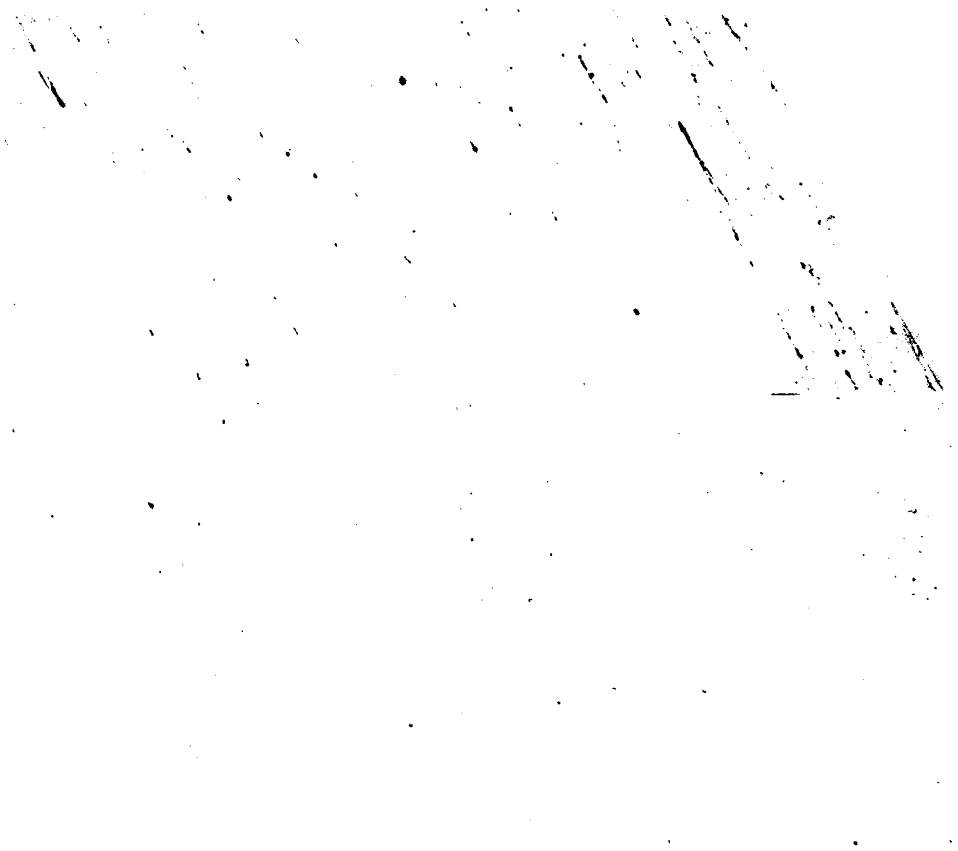
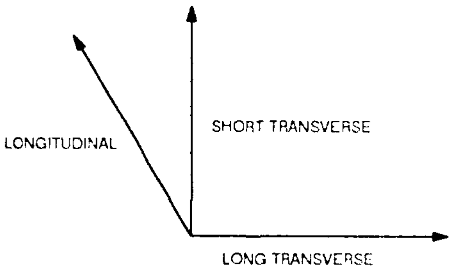
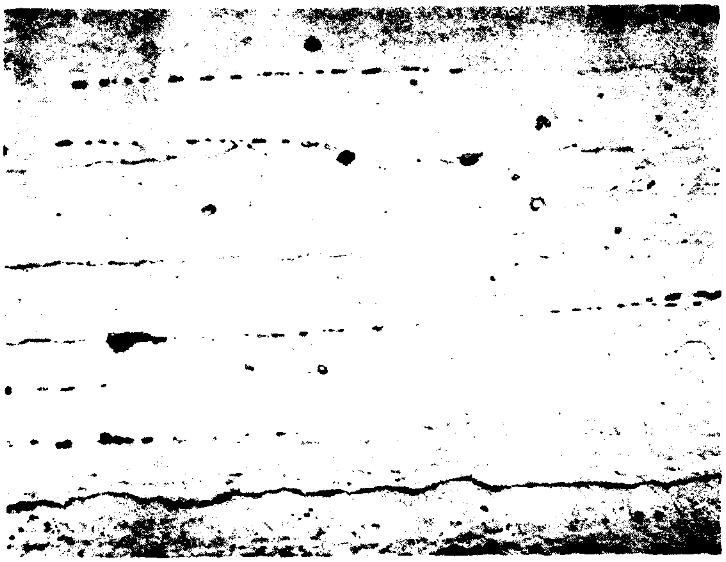


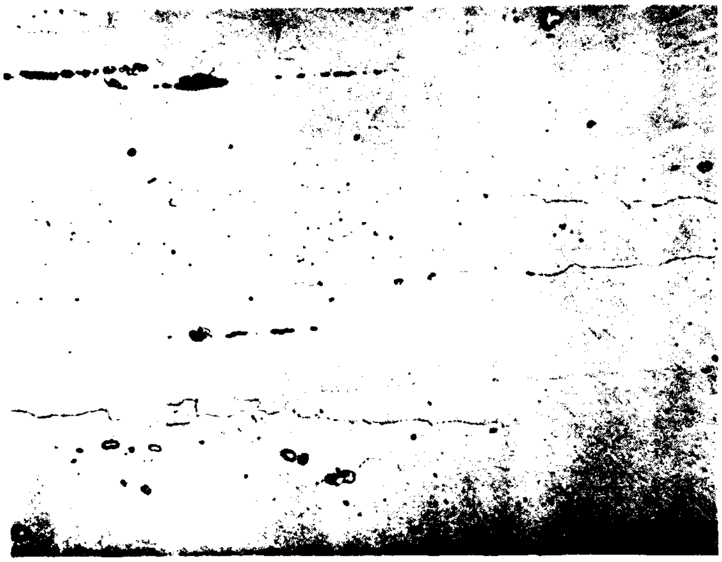
Figure 6. Composite photomicrograph of OSPREY alloy.





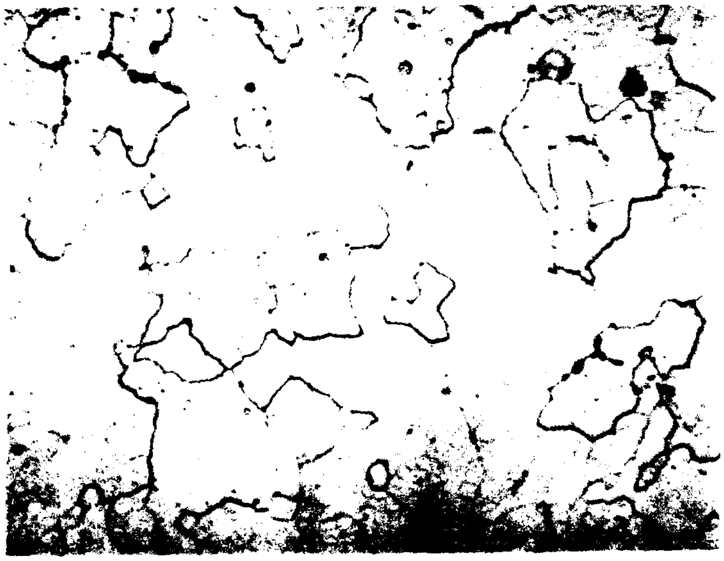
800X

Figure 7a. OSPREY alloy, L-ST section.



800X

Figure 7b. OSPREY alloy, L-LT section.



800X

Figure 7c. OSPREY alloy, ST-LT section.

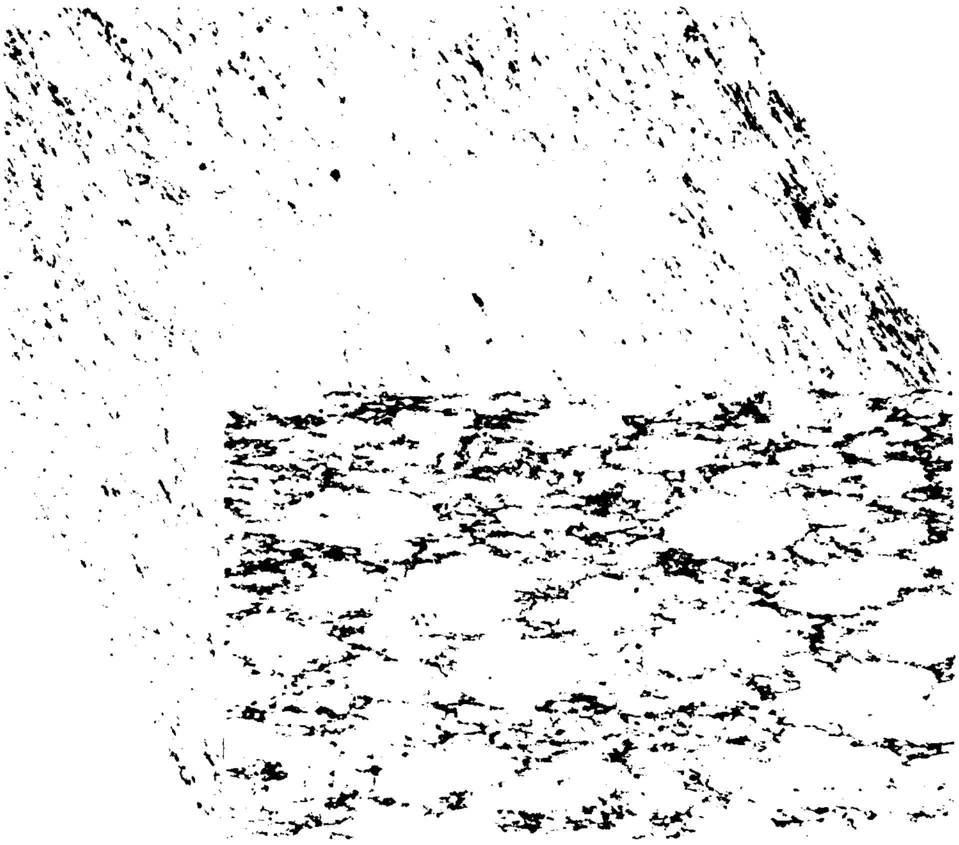
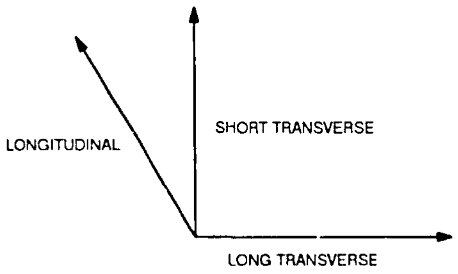


Figure 8. Photomicrograph of conventional powder alloy.

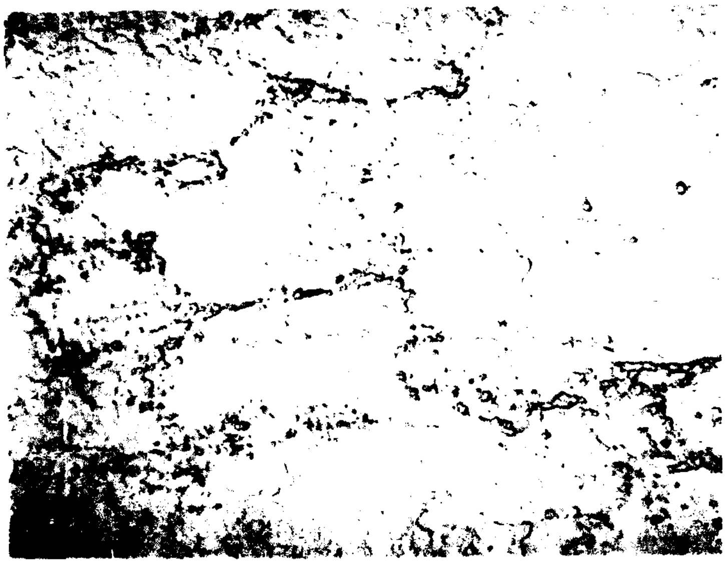


800X

Figure 9a. Conventional powder alloy, L-ST section.

800X

Figure 9b. Conventional powder alloy, L-LT section.



800X

Figure 9c. Conventional powder alloy, ST-LT section.

removed by the etch process. As discussed previously, this network could not be identified with the SEM in the as-polished condition. We concluded the pits are formed along a layer of contamination, probably very small oxide particles that are a result of the original powder-marking process.

In figure 9, a very fine grain or subgrain network is visible within the original powder particles. Comparing the two alloy samples, the fine grain boundary structure of the conventional material resembles the fine subgrain structure of the OSPREY material and the two may be the same. In the conventional powder alloy, the network is probably true grain boundaries because no clearly marked larger grain boundaries are present. In figure 9a, this network does appear to be a very fine recrystallized grain size structure.

To provide more insight into the structure of the two alloys, samples were heat treated and metallographically examined. One pair of samples was heat treated for one-half hour at 1000°F and furnace cooled over several hours. This put the material in the "0" temper. A second pair of samples was heat treated for 24 hours at 1000°F and water quenched, putting the material in the "T4" condition. The samples were mounted, polished, and etched as before.

The "0" temper samples are shown in figures 10 and 11. A generalized precipitation has occurred throughout as evidenced by the mottled appearance at low magnification. At high magnification, individual precipitate particles are visible, particularly in the OSPREY material. The particles are assumed to be $MgZn_2$ precipitation. The overall precipitation has suppressed the etch delineation of the grain boundaries, but the grains can be easily determined in the OSPREY material by the different orientation of the precipitate particles in each grain.

The Fe-Al intermetallic compounds are not visible in the OSPREY material. From the Al-Fe phase diagram, we expected that the $FeAl_6$ would transform into $FeAl_3$ and show up in the microstructure as perhaps a different shape particle and somewhat dispersed. The presence of the $MgZn_2$ precipitation perhaps has affected the appearance of the etched surface. The iron aluminum intermetallic compounds were not looked for in the as-polished condition. The subgrain structure is not easily seen, but can be faintly resolved in figure 10b. The subgrain structure could either have been modified by the heat treatment, or the etching effect masked by the $MgZn_2$ precipitation, or both.

In the conventional material, the fine grain size can be seen at high magnification. Some precipitation has occurred at the grain boundary intersections as shown in the large-powder particle in the lower half of figure 11b. This indicates that the grain size in the conventional material is very fine, and provides precipitation initiation sites for the $MgZn_2$. The second-phase network that outlined the original powder particles is still highly visible and apparently was not affected by the heat treatment. This heat treatment did not cause recrystallization or significant grain growth.

The T4 temper samples are shown in figures 12 and 13. The OSPREY material is essentially single phased, except that the Fe-Al intermetallic compounds are visible. Partial recrystallization has occurred as indicated by the stringers of smaller grains mixed in with the the overall larger grain structure. The sharp subgrain structure is no longer

100X

Figure 10a. OSPREY alloy, "0" temper L-LT section.

800X

Figure 10b. OSPREY alloy, "0" temper L-LT section.

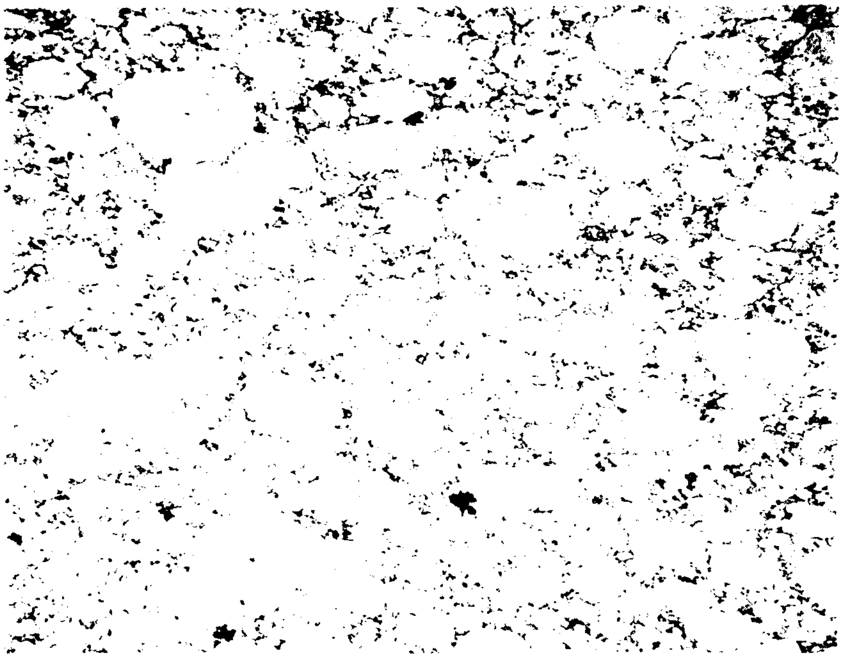


Figure 11a. Conventional powder alloy, "0" temper ST-LT section.

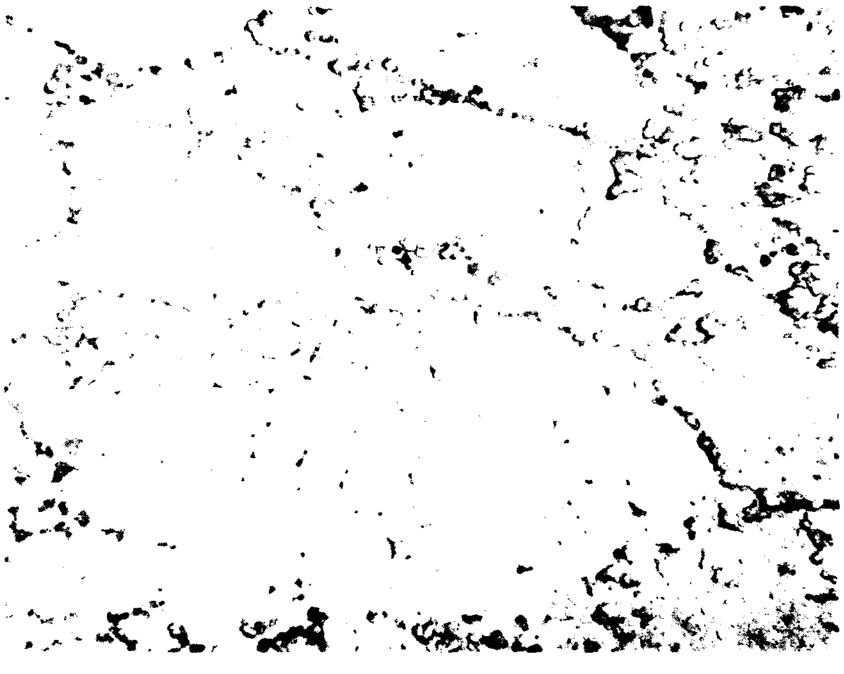


Figure 11b. Conventional powder alloy, "0" temper ST-LT section.

Figure 12a. OSPREY alloy, "T4" temper L-LT section.

Figure 12b. OSPREY alloy, "T4" temper L-LT section.

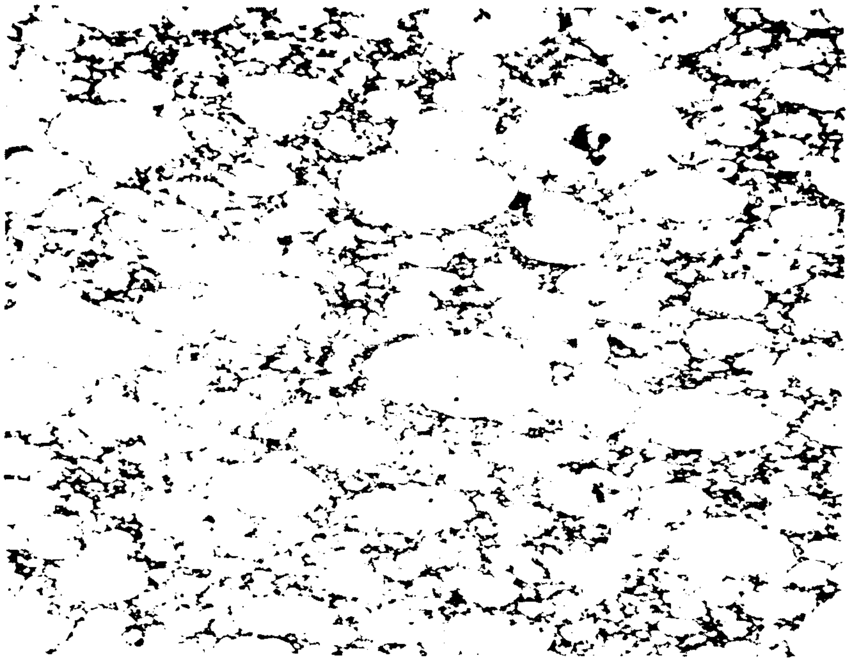


Figure 13a. Conventional powder alloy, "T4" temper ST-LT section.

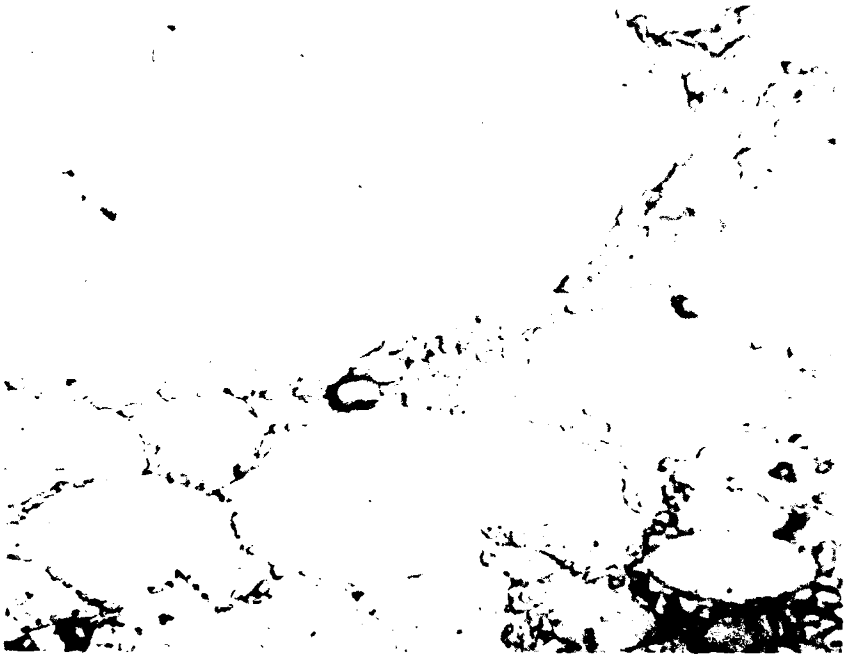


Figure 13b. Conventional powder alloy, "T4" temper ST-LT section.

present; however, some substructure is still visible as can be seen in the lower large grain shown in figure 12b. The long-term solution anneal has dissolved the majority of any dispersed precipitates and homogenized any composition gradients that may have been present.

The T4 temper sample of the conventional alloy is clearly single phased except for the network that outlines the original powder particles. The particle network was not affected by the long-term soak of the T4-heat treatment, indicating that these particles and the resultant etch effect is due to oxides surrounding the original powder particles. Aluminum oxides are insoluble in the matrix and are very stable at the heat-treat temperatures. The grain boundaries inside the individual powder particles are clean and sharply defined, and considerable grain growth occurred during the heat treatment.

To support the premise that the particle boundary etch effect is due to oxides, the alloys were analyzed for oxygen content. Samples of each material were analyzed with a Leco R014DR6 oxygen analyzer. The average weight percentage values of oxygen based on three determinations are presented:

OSPREY	<0.0005 percent by weight
Conventional	0.0857 percent by weight, 0.0017 standard deviation

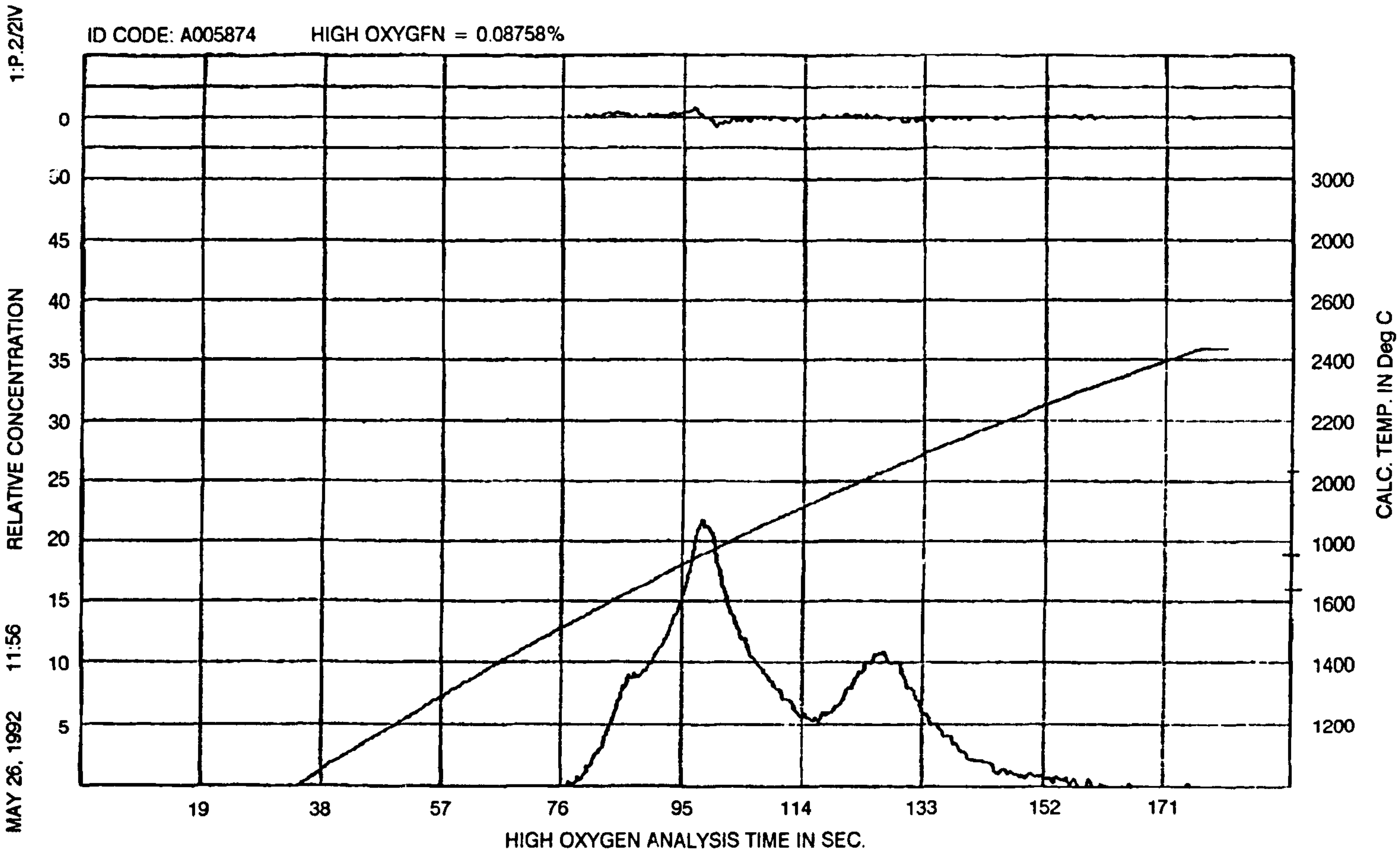
The oxygen content in the OSPREY alloy is unmeasurable. The conventional alloy has a significant amount of oxygen indicating that the etch effect that delineates the powder particles is due to oxides.

Figure 14 is an oxygen separation diagram generated by the oxygen analysis equipment. The apparatus essentially reduces the oxides of the sample with carbon and then measures the amount of carbon monoxide generated. The concentration of the oxygen emitted by the sample and temperature are plotted against time. This plot shows three different peaks in oxygen concentration corresponding to temperatures of 1650, 1750, and 2030°C. These peaks are usually associated with the breakdown of different species of oxides in a given alloy. In this case, the alloy is primarily aluminum, and Al_2O_3 is the only oxide formed at ambient temperatures and pressures. According to the Leco laboratories, a double peak in oxygen emission has commonly been observed in samples of aluminum powder alloys. Separate peaks have also been observed in alloys containing additions of the refractory alloys; zirconium in this case.

Hardness measurements were made on the alloy samples in the as-received condition and also on the heat-treated samples. The results are presented in table 2. The conventional powder alloy is harder in all temper conditions than the equivalent tempered OSPREY alloy. This hardening affect may be attributed to a dispersion hardening from the oxide inclusions. Both alloys were considerably harder in the as-received condition than after either of the heat treatments, indicating that the as-received alloy samples had been age hardened.

Table 2. Results of hardness measurements.

	As Received		"O" Temper	T4 Temper
	HRB	HRE	HRE	HRE
OSPREY	85	107	69	68
Conventional	87	108	72	76



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High Oxygen	1 0-115 Sec	0.05805%
High Oxygen	2 115-180 Sec	0.02953%
	High Oxygen =	0.08758%

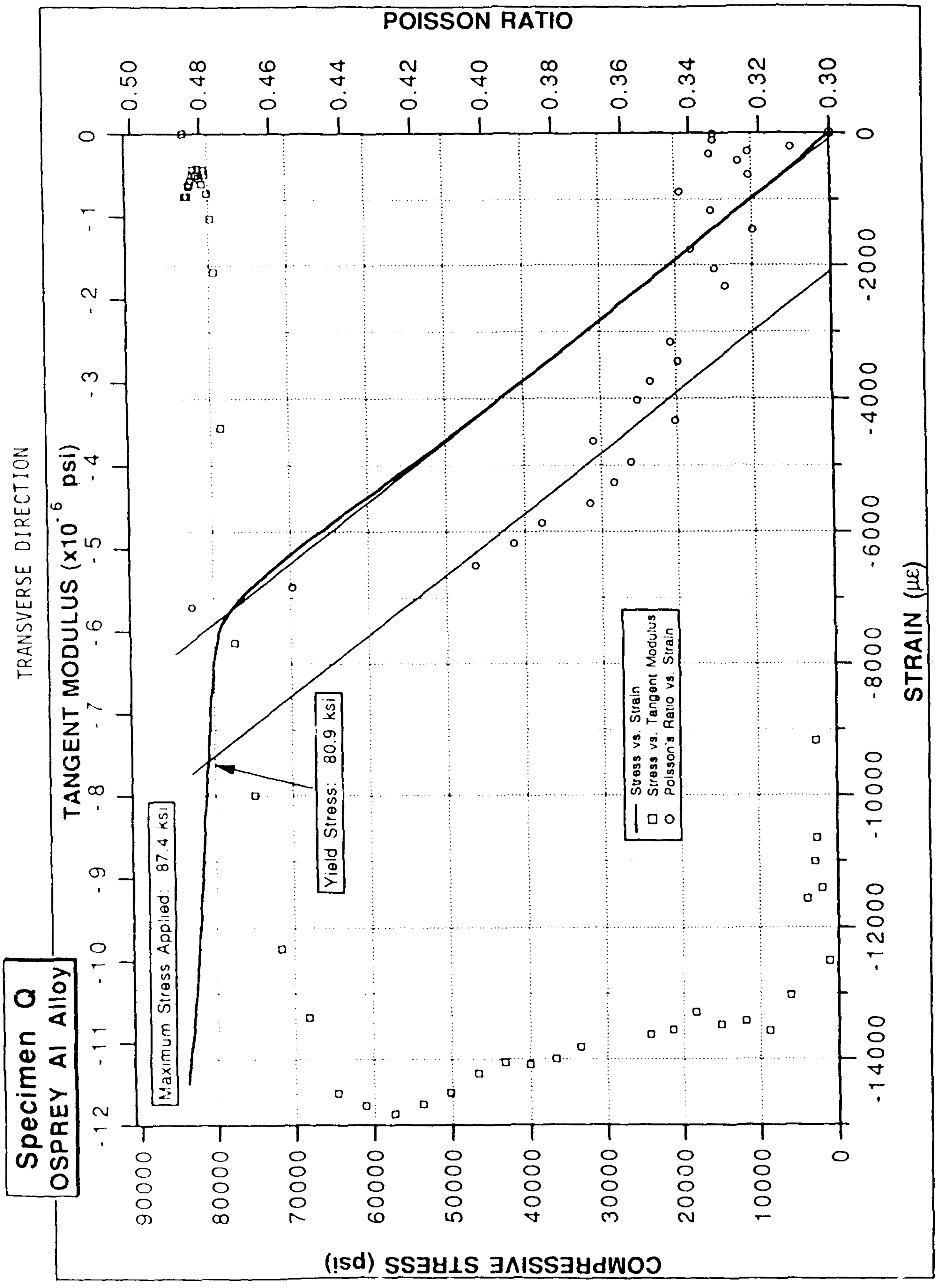
Figure 14. Oxygen separation diagram conventional powder sample.

Appendix

COMPRESSIVE TEST DATA

Appendix

COMPRESSIVE TEST DATA



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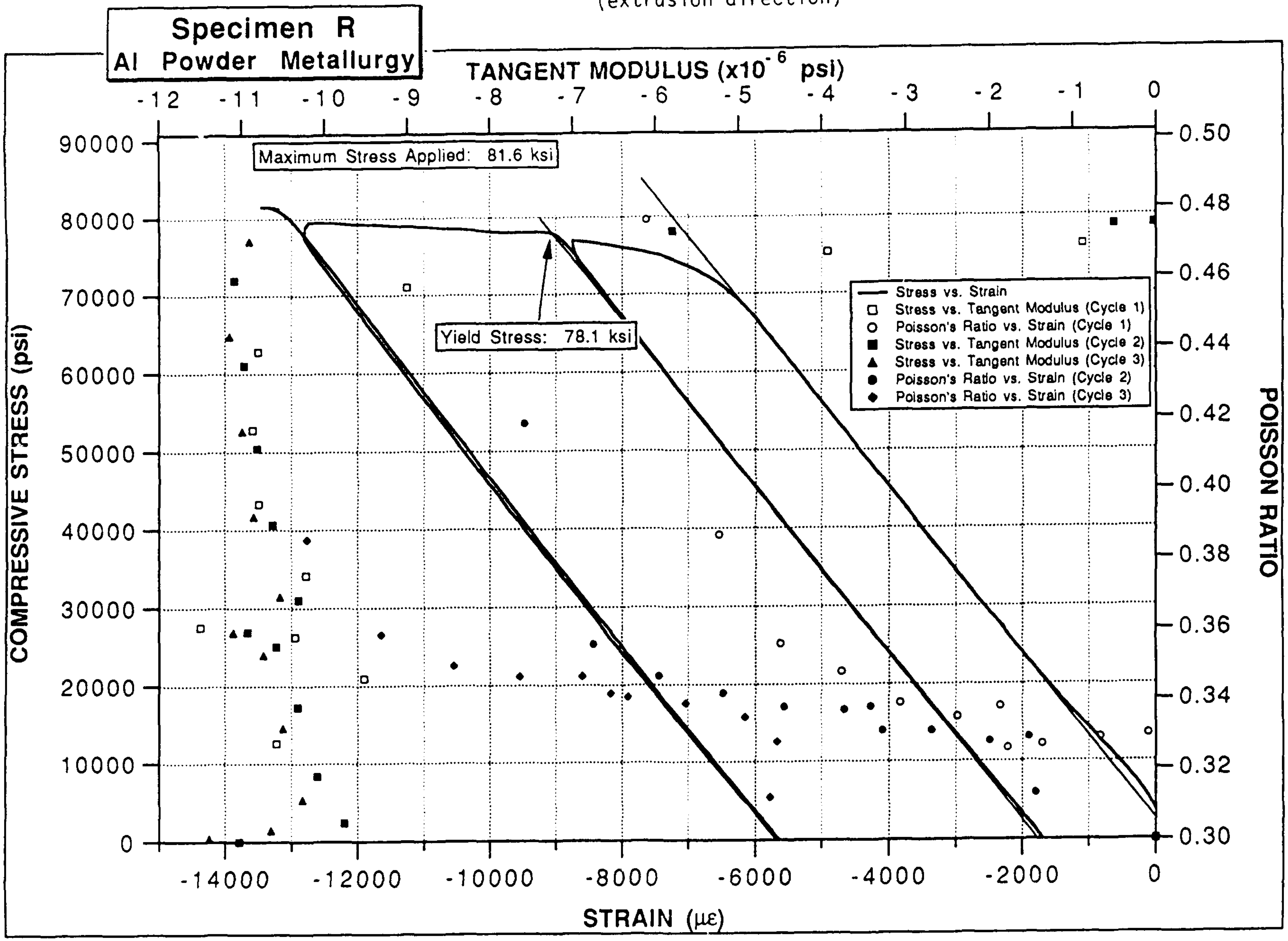

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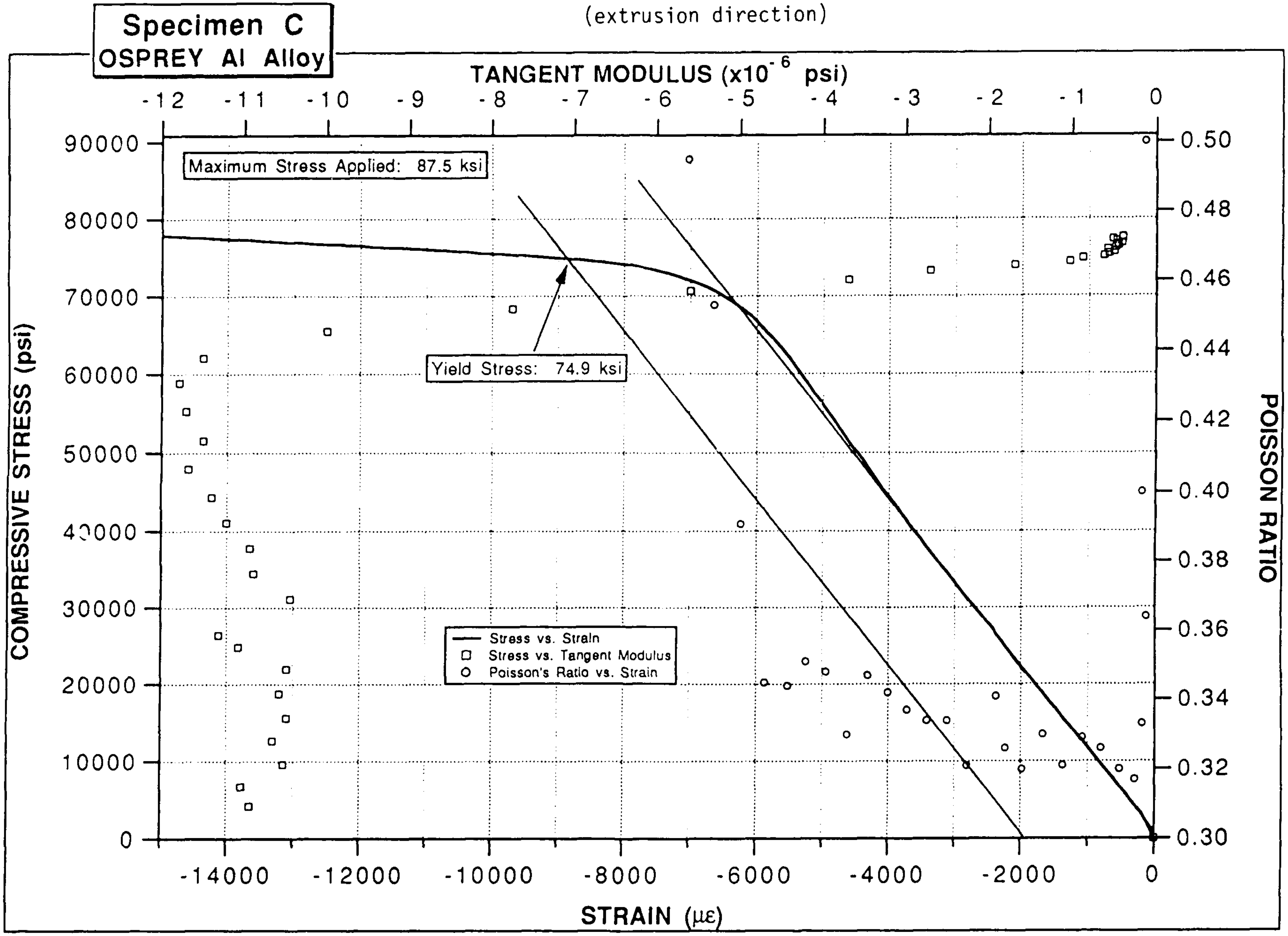

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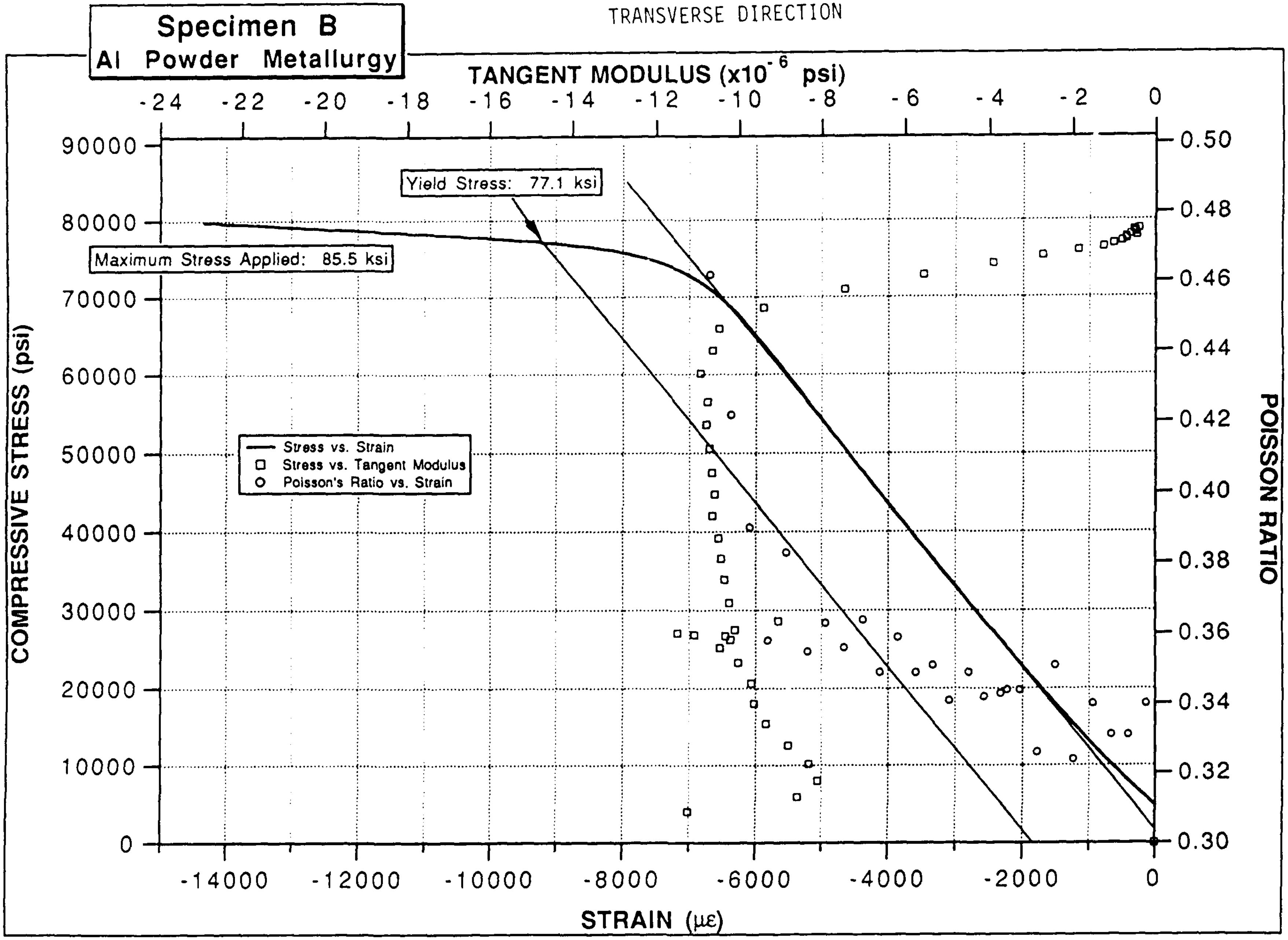

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. AGENCY USE ONLY *(Leave blank)*

2. REPORT DATE
April 1992

3. REPORT TYPE AND DATES COVERED
Final

4. TITLE AND SUBTITLE
ANALYSIS OF OSPREY PROCESSES AND CONVENTIONAL POWDER ALUMINUM ALLOYS

5. FUNDING NUMBERS
PR: ET35
PE: 0602234N
WU: DN088688

6. AUTHOR(S)
G. G. Chase and W. E. Glad

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
Naval Command, Control and Ocean Surveillance Center (NCCOSC)
RDT&E Division (NRaD)
San Diego, CA 92152–5000

8. PERFORMING ORGANIZATION REPORT NUMBER
NRaD TR 1500

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
Naval Surface Weapons Center
White Oak Lab
Silver Spring, MD 20910

10. SPONSORING/MONITORING AGENCY REPORT NUMBER

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION/AVAILABILITY STATEMENT
Approved for public release; distribution is unlimited.

12b. DISTRIBUTION CODE

13. ABSTRACT *(Maximum 200 words)*

The materials laboratory analyzed samples of nonstandard 7000 series aluminum alloy made by the OSPREY process and by a conventional powder process.

14. SUBJECT TERMS
CIVAPP: composite materials
CIVAPP: metal matrix
ordnance structures

15. NUMBER OF PAGES
33

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
UNCLASSIFIED	UNCLASSIFIED	UNCLASSIFIED	SAME AS REPORT

UNCLASSIFIED

21a. NAME OF RESPONSIBLE INDIVIDUAL W. E. Glad	21b. TELEPHONE *(Include Area Code)* (619) 553–3222	21c. OFFICE SYMBOL Code 932

NSN 7540-01-280-5500

Standard form 298 (BACK)

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