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ELECTRON PROBE MICROANALYSIS OF CONCENTRATION SEGREGATION IN AL--ETC(U)  
AUG 77 G JUDD

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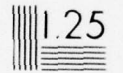
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FINAL REPORT

Contract No. N00014-75-C-0504, NR 031-737

ELECTRON PROBE MICROANALYSIS

OF

CONCENTRATION SEGREGATION IN Al-Zn-Mg

*See 1473*

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August 1977

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The purpose of this program has been to quantitatively determine the localized concentration gradients present during aging of precipitation hardened alloys and to study the effects that trace element additions have on the chemical specie distribution, the alloy microstructure, mechanical and corrosion properties of these alloys. Particular focus of this study has been the alloy element distribution as a result of the precipitation process, the formation of precipitate free zones (PFZ), the microstructure associated with PFZ, and the nature and distribution of grain boundary precipitation. The PFZ and grain boundary precipitation phenomena have been reported to be involved in the marked deterioration of the mechanical and corrosion resistant properties of aluminum precipitation hardened alloys.

The experimental portion of this study centered upon electron probe microanalysis coupled with studies of substructural details obtained from transmission electron microscopy. Mechanical and stress corrosion tests were employed to relate structural parameters to alloy properties. Several different Al-Zn-Mg ternary alloys were studied during the course of the program and special emphasis was placed on the role of titanium additions to the ternary. In addition, a powder metallurgy alloy was studied to compare the properties and structure of that alloy with conventionally prepared alloys for comparison purposes.

By carefully determining the chemical composition distribution, the effect of the titanium alloy addition, and the resulting micro-

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structures, the results of this study provided a better understanding of the mechanisms in controlling the precipitation process in the Al precipitation hardened alloys.

In terms of the microstructure in precipitation hardened alloys, of particular significance was the modification of PFZ formation theories as developed during the course of this program. A substantial modification in the quenched-in vacancy profile, due to vacancies generated by grain boundary precipitation was suggested for ternary alloys. The formation of the PFZ was thus felt to be governed by the vacancy profile resulting from the quench as modified by vacancies generated due to grain boundary precipitation. An expansion of this model was reported upon to include the effect of titanium and to explain observations in two-stage aging. The modified model took into account the solute and the vacancy concentration coupled with a time and temperature effect to explain variations in the PFZ with observed ternary alloys as a function of time and temperature of the first aging treatment in a two-stage aging treatment. The titanium addition itself was shown to reduce the amount of grain boundary precipitation and the width of the PFZ. It also affected the morphology of matrix precipitation in single aging treatments. In two-stage aging, the Ti addition alloy was less sensitive to changes in microstructure or PFZ width. These effects could all be explained in terms of Ti interacting with vacancies and/or in solute atoms causing the retardation in the precipitation phenomenon and changes in the concentration profile.

As a result of the electron probe microanalysis performed, both the specific characterization of microsegregation in the ternary and ternary with Ti addition alloys was characterized and analytical methods of characterization of microsegregation were developed. A statistical parameter was employed to be a measure of the microsegregation solute elements on a dimensional scale of the electron beam size. This technique was found to be widely applicable to the measurement of segregation in solids. Solute segregation was measured in both the ternary and the Ti addition alloys. It was found that Ti had a measurable retardation of the microsegregation phenomena both in terms of quenched-in microsegregation and the development of microsegregation that occurred in aging. Development of microsegregation was found to be dominated by the initial effects of solution temperature and quench rate. These heat treatment parameters controlled the vacancy concentration and thus affect the redistribution of solute.

The program showed the extreme sensitivity of the Al-Zn-Mg system to both composition and processing variables covering changes in the resultant microstructure and microsegregation levels. At the same time, the study gave insight into the mechanisms controlling the precipitation process and developed analytical techniques for measuring the microsegregation involved.

#### Technical Reports

The following technical reports were issued:

Technical Report #1-"Transmission Electron Microscopy Study of Al-Zn-Mg,"  
by G. Judd and C. R. Shastry, 1970.

4.

Technical Report #2-"An Electron Microprobe Analysis of Solute Segregation in Grain Boundaries in Al-Zn-Mg Alloy", by G. Judd and C. R. Shastry, 1971.

Technical Report #3-"Precipitation and Precipitate Free Zone Formation at the Grain Boundary in an Al-Zn-Mg Alloy", by C. R. Shastry and G. Judd, 1972.

Technical Report #4-"Effect of Titanium Additions on the Aging Characteristics of Al-Zn-Mg Alloys", by C. Grove and G. Judd, 1973.

Technical Report #5-"Stress Corrosion Cracking Susceptibility in Aging Characteristics of Al-Zn-Mg-Ti Alloys", by C. Chen and G. Judd, 1973.

Technical Report #6-"Microstructural Characterization and Stress Corrosion Susceptibility of Al-Zn-Mg-Ti Alloys", by C. Chen and G. Judd, 1974.

Technical Report #7-"A Microsegregation Parameter Study of Precipitation in Al-Zn-Mg Alloys", by A. Wirsing and G. Judd, 1975.

Technical Report #8-"Microstructure Study on the Effect of Titanium in the Aging Treatment of an Al-Zn-Mg Alloy", by C. Chen and G. Judd, 1976.

#### Publications

"Precipitation and Precipitate Free Zone Formation at the Grain Boundary in an Al-Zn-Mg Alloy", Met. Trans., 2, 3283-3287, 1971, C. R. Shastry and G. Judd.

"A Microanalysis Study of Solute Distribution in the Grain Boundary Region in an Al-Zn-Mg Alloy", Met. Trans., 3, 779-782, 1972, C. R. Shastry and G. Judd.

"Effects of Titanium Additions on the Aging Characteristics of an Al-Zn-Mg Alloy", Met. Trans., 4, 1023-1027, 1973, C. Grove and G. Judd.

"An Investigation of the Solute Distribution in the Grain Boundary Region in an Al-Zn-Mg Alloy", Proc. 5th Nat'l. Conf. on Electron Microprobe Analysis, July 1970, 23A-C, C. R. Shastry and G. Judd.

"Microsegregation in Al-Zn-Mg Alloys", to be published in the Proc. of the 12th Nat'l. Conf. on Electron Microprobe Analysis, August 1977, A. Wirsing and G. Judd.

Publications (cont.)

"An Electron Microprobe Study of Microsegregation in Al-Zn-Mg Alloys", submitted for publication to the 8th Internat'l. Conf. on X-Ray Optics and Microanalysis, A. Wirsing and G. Judd, 1977.

"Microstructure Study of the Effect of Titanium in the Aging Treatment of an Al-Zn-Mg Alloy", submitted for publication to Met. Trans., C. Chen and G. Judd, 1977.

Presentations

The following presentations were made supported under this grant:

"Effect of Heat Treatment on Solute Distribution in the Grain Boundary Region in an Al-Zn-Mg Alloy", presented at 1970 TMS Meeting, Cleveland, 1970, C. R. Shastry and G. Judd.

"Effect of Quench Rate on Precipitate Distribution in an Al-Zn-Mg Alloy", presented at 1970 TMS Meeting, Cleveland, 1970, C. R. Shastry and G. Judd.

"Characterization of Microsegregation by Electron Probe Microanalysis", presented at 1972 Materials Engineering Congress, G. Judd.

"Effect of Titanium Additions on the Aging Characterization of an Al-Zn-Mg Alloy", presented at the AIME Annual Spring Meeting, Philadelphia, 1973, C. Grove and G. Judd.

"The Effects of Titanium Additions on Aging Characteristics and Stress Corrosion Cracking Susceptibility of Al-Zn-Mg Alloys", presented at the AIME Spring Meeting, Toronto, 1975, C. Chen and G. Judd

"Microstructure Study of the Effect of Titanium in Two-Stage Aging in Al-Zn-Mg", 105th AIME Annual Meeting, Las Vegas, 1976, C. Chen and G. Judd.

"Microsegregation in Al-Zn-Mg Alloys", 105th Annual AIME Meeting, Las Vegas, 1976, A. Wirsing and G. Judd.

"Microsegregation in Al-Zn-Mg Alloys", to be presented at the 8th Internat'l. Conference on X-Ray Optics and Microanalysis, Boston, 1977, A. Wirsing and G. Judd.

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Students Supported

During the course of the contract, the following students were supported, receiving the degrees listed:

T. Sargent	M.S.	1971
C. Chen	M.S.	1973
C. R. Shastry	Ph. D.	1971
C. Chen	Ph. D.	1977
A. Wirsing	Ph. D.	1977

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) A summary report is presented on the studies of microsegregation and microstructure in Al-Zn-Mg-Ti alloys performed under the contract entitled, *Electron Probe Microanalysis of Concentration Segregation in Aluminum-Zinc-Magnesium.*		

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