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Final Report

INVESTIGATION OF THE ROLE OF TRACE ADDITIONS ON PRECIPITATION, DEFORMATION AND FRACTURE ON ALUMINUM ALLOYS

ONR Grant Number N00014-91-J-1285

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Summary of Research:

The research objectives of this program have been to identify relationships between precipitate structure, grain structure, slip behavior on a micro scale, and strength and bulk deformation and fracture behavior of new aluminum-lithium alloys.

The research on this grant has led to the development of a heat treatment that improves the short-transverse fracture toughness of 8090 by producing a transition in deformation behavior from coarse planar slip to homogeneous deformation (1). We have also developed a low temperature aging treatment for 2090 that significantly improves the fracture toughness without affecting the strength. This treatment reduces the amount of T_1 located on subgrain boundaries (2,3). The low-temperature aging practice, along with a pre-age six percent stretch which was developed in our laboratory under a previous contract (4), has become the standard method of improving the fracture toughness of 2195 for the Super-Light-Weight Tank.

A model that predicts plane strain fracture toughness in Al-Li-X alloys (and any aluminum alloy that has a low volume fraction of constituents and deforms by planar slip) has been developed and correlates nicely with experimental results (5). We have developed general concepts for optimizing mechanical properties of particle-hardened alloys by microstructural design (6,7). Simply stated, we have shown that a more balanced set of mechanical properties can be achieved by controlling the precipitate structure and deformation behavior by proper control of thermal treatments in a number

of Al-Li-X systems and we have identified the desired microstructures. A significant amount of the results of our research on aluminum alloys were highlighted in an invited paper "Application of Modern Aluminum Alloys to Aircraft," by E.A. Starke, Jr. and J.T. Staley, that was published in *Progress in Aerospace Sciences* (8).

We also studied the effect of trace element additions on the nucleation of strengthening precipitates in Al-Cu-Li-X alloys. These alloys are usually worked prior to aging in order to introduce dislocation structures into the matrix, which will subsequently act as preferential nucleation sites for precipitates. However, many product forms do not lend themselves readily to the use of a pre-age stretch, and even when it is feasible, undesirable anisotropy may result. Alternatively, certain trace alloying additions have been found to aid nucleation of the strengthening phases. Trace additions of In, Mg, and Si were analyzed in our study. We believe that we now understand the role that the various trace additions play in the precipitation of the strengthening precipitates in these alloys.

We found that trace additions of indium increase the T6 yield strength of the Al-Cu-Li alloy studied by 25%. The T6 strength of the Al-Cu-Li-In alloy was roughly equal to that of the baseline T86 Al-Cu-Li alloy. Cold work before aging did not increase the strength of the Al-Cu-Li-In alloy. The increase in strength was associated with an increase in the thickness of the {100} precipitates, indicating a shift from θ" to θ'. This occurs after five to ten hours aging at 160°C. Indium promotes the matrix precipitation of the T_1 phase, but in smaller volume fractions than θ'. Indium is believed to be associated with the interface or the crystal structure of the precipitate, thereby lowering the energy barrier for nucleation. We found that removing the low-level of silicon impurities from the alloy did not influence the magnitude of the strength increase associated with indium, but did seem to slow aging kinetics. The microstructure was similar to that of the higher-silicon alloy, but precipitates were larger, with higher aspect ratios, and more T_1 was present.

We also observed that approximately 0.5wt. % addition of Mg to the base-line alloy increased the T6 yield strength by 30%. The increase in strength in the early stages of aging was associated with the precipitation of a high number density of fine θ" and GP zones. As aging proceeded, significant amounts of high-aspect ration T_1 grew, especially near dislocations and subgrain boundaries. The strength of the T86 temper of the Mg-bearing alloy is entirely due to T_1. Mg influences precipitation by interaction with quenched-in vacancies. The increased volume fraction of the {111} precipitates in the Mg-bearing alloy leads to increased anisotropy when compared to the In-bearing and baseline alloys. The details of this research were published in reference (9).

We have also studied the role of an applied stress during the aging process of aluminum alloys. Our fundamental studies on "stress aging" has direct relevance to the "age forming" process that has been recently utilized in the manufacturing of integrally stiffened structures. The combination of integrally stiffened structures and age forming has the potential to improve the performance and to significantly reduce fabrication costs of land, sea and air transportation systems. Our research addressed three aspects of the problem: 1. How an externally applied stress at the age hardening temperature of the alloy affects the resulting microstructure, particularly the dislocation and precipitate structures. 2. How the evolving precipitate structure affects the deformation behavior during the age forming process when stress relaxation and significant deformation occurs. 3. How the process affects the resulting mechanical properties of the age-formed part.

We have systematically investigated a model alloy system, Al-Cu, and have observed preferentially aligned precipitate structures under various conditions. Our experiments were conducted on both single crystals and cube-textured polycrystals; having Cu contents that varied from 2.5 to 5 weight percent, at aging temperatures from 150-240°C, and with both tensile and compressive applied stresses from 8 MPa to 100 MPa. We have tested the stability and evolution of the resulting aligned precipitate structures after the stress aging treatment. We have developed a computer simulation method to evaluate the strengthening effects of various precipitate structures, including the combined effects of multiple types of precipitates (10, 11). Compared with previous models, our computer simulation has yielded results closer to those obtained experimentally (12). Using our experimental results as a base, we have developed a model, which predicts the aging response for Al-Cu alloys as a function of applied stress, alloy composition and temperature of aging (13, 14).

The research under this ONR grant has resulted in twenty-four publications in archival journals, fourteen conference proceedings, and numerous invited oral presentations. Also the International Union of Materials Research Societies presented us with an *Innovation in Real Materials Award in 1998* for our research on Al-Li-X alloys, which was primarily supported by ONR Grants.

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Principal Investigator: Professor Edgar A. Starke, Jr.

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Gary H. Bray, Ph.D., Department of Materials Science and Engineering, University of Virginia, May, 1993.

Delwyn L. Gilmore, Ph.D., Department of Materials Science and Engineering, University of Virginia, May, 1996.

Papers in Refereed Journals for Research Sponsored by ONR:

H.J. Roven, E.A. Starke, Jr., Ø. Sødahl and J. Hjelen, "Effects of Texture on Delamination Behavior of a 8090-Type Al-Li Alloy at Cryogenic and Room Temperature," Scripta Met. 24, 421-426 (1990).

C.P. Blankenship, Jr., and E.A. Starke, Jr., "The Fatigue Crack Growth Behavior of the Al-Cu-Li Alloy Weldalite 049," J. Fatigue and Fracture of Engineering Materials and Structures 14, 103-114 (1991).

E. Hornbogen, A.K. Mukhopadhyay, and E.A. Starke, Jr., "An Exploratory Study of Hardening in Al-(Si,Ge) Alloys," Z. Metallkd. 83, 577-584 (1992).

G.H. Bray, A.P. Reynolds, and E.A. Starke, Jr., "Mechanisms of Fatigue Crack Retardation Following Single Tensile Overloads in Powder Metallurgy Aluminum Alloys," Met. Trans. A 23, 3055-3066 (1992).

C.P. Blankenship, Jr., and E.A. Starke, Jr., "Improved Toughness in Al-Li-Cu-Mg-Ag-Zr Alloy X2095," Scripta Met. Mater. 26, 1719-1722 (1992).

G.H. Bray and E.A. Starke, Jr., "The Effect of Elevated Temperature Exposure on Fatigue Crack Growth Retardation," Scripta Met. Mater. 27, 593-598 (1992).

E. Hornbogen, A.K. Mukhopadhyay, and E.A. Starke, Jr., "Precipitation Hardening of Al-(Si,Ge) Alloys," Scripta Met. Mater. 27, 733-738 (1992).

C.P. Blankenship, Jr., and E.A. Starke, Jr., "Mechanical Behavior of Double-Aged AA8090," Met. Trans. A 24, 833-841 (1993).

E. Hornbogen and E.A. Starke, Jr., "Theory Assisted Design of High Strength Low Alloy Aluminum," Overview No. 102, Acta Met. Mater. 41, 1-16 (1993).

E. Hornbogen, A.K. Mukhopadhyay, and E.A. Starke, Jr., "Nucleation of the Diamond Phase in Aluminium-Solid Solutions," J. Mater. Sci. 28, 3670-3674 (1993).

M. Sugamata, C.P. Blankenship, Jr., and E.A. Starke, Jr., "Predicting Plane Strain Fracture Toughness of Al-Li-Cu-Mg Alloys," Mater. Sci. Engr. A163 1-10 (1993).

D.C. Slavik, C.P. Blankenship, Jr., E.A. Starke, Jr., and R.P. Gangloff, "Intrinsic Fatigue Crack Growth Rates for Al-Li-Cu-Mg Alloys in Vacuum," Met. Trans. A 24A, 1807-1817 (1993).

C.P. Blankenship, Jr., E. Hornbogen, and E.A. Starke, Jr., "Predicting Slip Behavior in Alloys Containing Shearable and Strong Particles," Mater. Sci. Engr. A169, 33-41 (1993).

C.P. Blankenship, Jr., and E.A. Starke, Jr., "Structure-Property Relationships in Al-Li-Cu-Mg-Ag-Zr Alloy X2095," Acta Met. Mater. 42, 845-855 (1994).

Y. Mou, J. M. Howe, and E. A. Starke, Jr., "Grain Boundary Precipitation and Fracture Behavior of an Al-Cu-Li-Mg-Ag Alloy," Met. Trans A, 26A, 1591-1594 (1995).

C. P. Blankenship, Jr., G.H. Bray, L.R. Kaisand and E.A. Starke, Jr.,"Low Cycle Fatigue Behavior of Two Al-Li Alloys," Fatigue and Fracture of Engineering Materials and Structures Vol 18 No 5, pp. 551-564 (1995).

E. A. Starke, Jr., and J. T. Staley, "Application of Modern Aluminum Alloys to Aircraft," Progress in Aerospace Sciences, Vol 32, pp. 131-172 (1996).

D.L. Gilmore and E.A. Starke, Jr., "Trace Element Effects on Precipitation Processes and Mechanical Properties in an Al-Cu-Li Alloy," Metallurgical and Materials Transactions A, Vol 28A, pp. 1399-1415 (1997).

H. Hargarter, M.T. Lyttle and E.A. Starke, Jr., "Effects of Preferentially Aligned Precipitates on Plastic Anisotropy in Al-Cu-Mg-Ag and Al-Cu Alloys," Materials Science & Engineering A, Vol. 257, pp. 87-99 (1998).

A.W. Zhu, A. Csontos and E.A. Starke, Jr., "Computer Experiment on Superposition of Strengthening Effects of Different Particles," Acta Materialia Vol. 47, pp. 1713-1721 (1999).

A.W. Zhu and E.A. Starke, Jr., "Strengthening Effect of Unshearable Particles of Finite Size: A Computer Experimental Study," Acta Materialia Vol. 47, pp. 3263-3269 (1999).

A.W. Zhu, J. Chen and E.A. Starke, Jr., "Precipitation Strengthening of Stress-Aged Al-xCu Alloys," Acta Materialia, Vol. 48, pp. 2239-2246 (2000).

A.W. Zhu and E.A. Starke, Jr., "Stress-Aging of Al-xCu Alloys: Experiments," Acta Materialia. (in press).

A.W. Zhu and E.A. Starke, Jr., "Stress-Aging of Al-xCu Alloys: Computer Modeling," Acta Materialia. (in press).

Papers in Conference Proceedings on Research Sponsored by ONR:

C.P. Blankenship, Jr., H.J. Roven, and E.A. Starke, Jr., "The Fracture and Fatigue Behavior of Al-Li-X Alloys," Fatigue 90, eds. H. Kitagawa and T. Tanaka, MCE Publications Ltd., Birmingham, United Kingdom, 937-955 (1990).

C.P. Blankenship, Jr., and E.A. Starke, Jr., "Fracture Behavior of Aluminum-Lithium-X Alloys," Aluminium-Lithium, eds. M. Peters and P.-J Winkler, DGM Informationsgesellschaft mbH, Oberursel, FRG, 187-201 (1992).

H.J. Roven and E.A. Starke, Jr., "Microstructural Effects on Fatigue and Fracture Behavior at Cryogenic and Room Temperature of an Al-Li 8090 Alloy," Mechanical Behaviour of Materials-VI, July 29-August 2, 1991, Kyoto, Japan, 25-29 (1992).

E. Hornbogen, A.K. Mukhopadhyay, and E.A. Starke, Jr., "Precipitation Hardening and Microstructural Stability in Al-Si-Ge-Cu Alloys," Aluminium Alloys: Their Physical and Mechanical Properties, Proceedings of the Third International Conference on Aluminum Alloys (ICAA3), Vol. I, eds. L. Arnberg, O. Lohne, E. Nes, and N. Ryum, The Norwegian Institute of Technology, Trondheim, Norway, 199-207 (1992).

E.A. Starke, Jr., E. Hornbogen, and C.P. Blankenship, Jr., "Theory and Design of Ideal Microstructures in Particle Hardened Aluminum Alloys," Aluminium Alloys: Their Physical and Mechanical Properties, Proceedings of the 3rd International Conference on Aluminium Alloys (ICAA3), Vol. III, eds. L. Arnberg, O. Lohne, E. Nes, and N. Ryum, The Norwegian Institute of Technology, Trondheim, Norway, 279-299 (1992).

G.H. Bray and E.A. Starke, Jr., "Fatigue Crack Retardation in a Dispersion-Strengthened PM Aluminum Alloy," "Proceedings of Fatigue '93, 3-7 May 1993, Montreal.

E.A. Starke, Jr., and C.P. Blankenship, Jr., "Aluminum-Lithium Alloys," Metallic Materials for Lightweight Applications: Proceedings of the Sagamore Army Materials Research Conference, M.G.H. Wells, E.B.

Kula, J.H. Beatty, eds., Plymouth, MA, 30 August - 2 September, pp. 139-157 (1993).

E.A. Starke, Jr., and C.P. Blankenship, Jr., "Aluminum-Lithium Alloys," Aluminum-Lithium Alloys for Aerospace Applications Workshop, B.N. Bhat, T.T. Bales & E.J. Vesely, Jr., eds., NASA Marshall Space Flight Center, AL, May 17-19, pp. 6-16 (1994).

D.L. Gilmore and E.A. Starke, Jr., "Trace Element Effects on Precipitation Processes and Mechanical Properties in Al-Cu-Li-X Alloys," Aluminum Alloys: Their Physical and Mechanical Properties, Proceedings of the 4th International Conference on Aluminum Alloys (ICAA4), ed. T. H. Sanders, Jr., and E.A. Starke, Jr., Atlanta, GA, 11-16 September 1994, Vol II, 313-320.

D. L. Gilmore and E. A. Starke, Jr., "The Effects of In and Mg Trace Additions on Precipitation Processes and Mechanical Properties in an Al-Cu-Li Alloy," Light Weight Alloys for Aerospace Applications III, eds. E.W. Lee, N.J. Kim, K.V. Jata and W.E. Frazier, TMS, Warrendale, PA, 1995, 3-16.

D.L. Gilmore and E.A. Starke, Jr., "The Effects of In, Mg and Impurity Trace Additions on Precipitation, Anisotropy, and Related Properties in Al-Cu-Li Alloy", Aluminium Alloys, Their Physical and Mechanical Properties - Proceedings ICAA5, July 1-5, 1996, Grenoble, France, 877-882.

E.A. Starke, Jr., (Invited) "The Present and Future of Aluminum Alloys in Transportation Systems," Proceedings of the International Forum on Creation of Super Metallic Materials Consisting of Amorphous, Nonoscale and Mesoscopic Structures (FCSMM), Tokyo, Japan, 7 February (1997), 39-48.

A.W. Zhu and E.A. Starke, Jr., "A Finite Element Analysis of Strengthening Effects of Plate-Like Particles in a Metal Matrix," Materials Science Forum, Vols. 331-337, 1279-1284 (2000).

A.W. Zhu and E.A. Starke, Jr. "Strengthening Effects of Precipitates and C-Nanotubes in Aluminum Alloys/Composites: Dislocation-Slip Simulations, to be presented as an invited Keynote Lecture at the Eight International Conference on Composites Engineering, ICCE/8, August 5-11, 2001 in Tenerife, Spain.

References:

1. C.P. Blankenship, Jr., and E.A. Starke, Jr., *Met. Trans. A.,* Vol. 24, 1993, p. 833.
2. C.P. Blankenship, Jr., and E.A. Starke, Jr., *Scripta Met. Mater.,* Vol. 26, 1992, p. 1719.
3. C.P. Blankenship, Jr., and E.A. Starke, Jr., *Acta Met. Mater.,* Vol. 42, 1994, p. 845.
4. W.A. Cassada, G.J. Shiflet and E.A. Starke, Jr., *Met. Trans. A.,* Vol. 22, 1991, p. 299.
5. M. Sugamata, C.P. Blankenship, Jr., and E.A. Starke, Jr., *Mater. Sci. Engr.,* Vol. A163, 1993, p. 1.
6. E. Hornbogen and E.A. Starke, Jr., *Acta. Met. Mater.,* Vol. 41, 1993, p. 1.
7. C.P. Blankenship, Jr., E. Hornbogen and E.A. Starke, Jr., *Mater. Sci. Engr.,* Vol. A169, 1993, p. 33.
8. E.A. Starke, Jr., and J.T. Staley, *Prog. Aerospace Sci.,* Vol. 32, 1996, p. 131.
9. D.L. Gilmore and E.A. Starke, Jr., *Metall. Mater. Trans.,* Vol. 28A, 1997, p. 1399.
10. A.W. Zhu, A. Csontos and E.A. Starke, Jr., *Acta Mater.,* Vol. 47, 1999, p. 1713.
11. A.W. Zhu and E.A. Starke, Jr., *Acta Mater.,* Vol. 47, 1999, p. 3263.
12. A.W. Zhu J. Chen and E.A. Starke, Jr., *Acta Mater.,* Vol. 48, 2000, p. 2239.
13. A.W. Zhu and E.A. Starke, Jr., "Stress-Aging of Al-xCu Alloys: Experiments," *Acta Mater.* (in press).
14. A.W. Zhu and E.A. Starke, Jr., "Stress-Aging of Al-xCu Alloys: Modeling," *Acta Mater.* (in press).

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