We gratefully acknowledge the financial support of the Air Force Office of Scientific Research under Grant No. AFOSR-91-0421, which is monitored by Dr. Walter Jones, and the National Science Foundation, under Grant No. DMR-92-57465, which is monitored by Dr. Bruce Mac-Donald.

REFERENCES

- 1. A. Guinier: X-Ray Diffraction in Crystals, Imperfect Crystals, and Amorphous Bodies, W.H. Freeman and Co., New York, NY, 1963, pp. 121 and 240-248.
- 2. B.E. Warren: X-Ray Diffraction, Addison-Wesley, Reading, MA, 1969, p. 251.
- 3. R.C. Batra and C.H. Kim: Int. J. Plasticity, 1992, vol. 8, pp. 425-52.
- 4. S.P. Timothy: Acta Metall., 1987, vol. 35, pp. 301-06.
- 5. H. Dève and R.J. Asaro: Metall. Trans. A, 1989, vol. 20A, pp. 579-93.
- 6. C.A. Pampillo: J. Mater. Sci., 1975, vol. 10, pp. 1194-1227.
- E.J. Kramer: J. Polymer Sci., Polymer Phys. Ed., 1975, vol. 13, pp. 509-25.
- 8. P.B. Bowden and J.A. Jukes: J. Mater. Sci., 1972, vol. 7, pp. 52-63.
- 9. J.E. Carsley, J. Ning, W.W. Milligan, S.A. Hackney, and E.C. Aifantis: NanoStr. Mater., 1995, vol. 5, pp. 441-448.
- G.W. Nieman, J.R. Weertman, and R.W. Siegel: J. Mater. Res., 1991, vol. 6, pp. 1012-27.
- 11. I.A. Ovid'ko: J. Phys. D, Appl. Phys., 1994, vol. 27, pp. 999-1007.

Grain Structure and Quench-Rate Effects on Strength and Toughness of AA7050 Al-Zn-Mg-Cu-Zr Alloy Plate

R.C. DORWARD and D.J. BEERNTSEN

The fracture toughness of heat-treatable Al-Zn-Mg-Cu aluminum alloys is influenced by a number of microstructural features, which are controlled by chemistry and processing. The effects of constituent particles (insoluble and soluble) and hardening precipitates are well known and understood.[1,2,3] Also well accepted are the independent effects of quench rate and recrystallization, whereby slack quenches reduce toughness,^[4] as do recrystallized structures.^[3,5] Less is understood, however, about possible interactions between these factors. It has been claimed that the rate of loss in toughness with decreasing quench rate is higher for recrystallized products because of precipitation on high-angle boundaries.^[6] Although this contention is partially supported by loss-of-ductility data,^[7,8] the prior results were obtained on Cr/Zr-free alloys with essentially equiaxed grain structures. And, as Unwin and Smith noted,^[9] tensile elongation in such materials does not necessarily correlate with fracture toughness.

To examine the inter-relationships between quench rate and recrystallization as they affect both strength and fracture toughness, a systematic study was conducted in which AA7050 plates with various grain structures were solution heat-treated using a number of quench rates. The starting material was a 150-mm-thick F temper plate (composition shown in Table I), which was hot-rolled to 14-mm-thick plates. By varying the final (exit) rolling temperature from 245 °C to 385 °C, levels of recrystallization ranging from 15 to 80 pct at the center of the plates were achieved after solution heat treating at 475 °C for 1 hour. Recrystallization was estimated visually after a 5 minute etch in 10 pct H_3PO_4 at 50 °C. The photomicrographs in Figure 1 show that the unrecrystallized condition is characterized by a

Table I. Chemical Composition of 7050 Plate

Weight Percent*						
Si**	Fe	Cu	Mg	Zn	Ti	Zr
0.04	0.07	2.25	2.11	6.43	0.04	0.10

*By inductively coupled plasma spectroscopy, except silicon (others < 0.01 pct each).

******Quantometer estimate.



+ 20 µm (*a*)



(b)

Fig. 1—Microstructures of 7050 plates: (a) 15 pct recrystallized and (b) 80 pct recrystallized. High-angle boundaries in (a) actually define relatively large elongated grains as revealed by the Barker's etch insert (1/5 magnification of the 10 pct H_1PO_4 etch photomicrographs).

R.C. DORWARD, Program Manager, Aircraft Products, is with the Center for Technology, Kaiser Aluminum & Chemical Corporation, Pleasanton, CA 94566. D.J. BEERNTSEN, formerly Senior Staff Research Metallurgist, Aircraft Products, Center for Technology, Kaiser Aluminum & Chemical Corporation, is retired.

Manuscript submitted September 20, 1994.



Fig. 2—Transmission electron photomicrograph showing precipitates in subgrain boundaries of slowly quenched plate (8 °C/s).



Fig. 3-Effect of quench rate on the strength of 7050-T6 plate.

well-developed subgrain structure, which is common to zirconium-containing alloys. The grains defined by high-angle boundaries have an elongated, pancake shape, as revealed by polarized light after a Barker's etch. When these regions recrystallize, they form considerably smaller, subgrain-free grains with much lower aspect (length-to-thickness) ratios.

Samples of each plate were quenched from the solutionizing treatment in agitated water baths held at 20 °C, 60 °C, 80 °C, and 95 °C, which provided cooling rates of about 150 °C/s, 50 °C/s, 20 °C/s, and 8 °C/s, respectively, through the 400 °C to 285 °C temperature range. Transmission electron microscopy showed little boundary precipitation at the fast quench rate; at 8 °C/s, precipitates rich in zinc, magnesium, and copper were evident in both high-angle grain boundaries and in subgrain boundaries, particularly those oriented in the longitudinal direction (Figure 2). After aging for 12 hours at 120 °C plus 12 hours at 155 °C (T6 temper), the samples were tested for longitudinal tensile properties



Fig. 4-Effect of recrystallization on the strength of 7050-T6 plate.

(6.35-mm diameter by 25-mm length specimens) and L-T fracture toughness using chevron-notched specimens (ASTM E-1304). Both tests were conducted with at least two specimens for each condition.

Figure 3 shows the effect of quench rate on the 0.2 pct yield strengths of 15 and 80 pct recrystallized plates. Both materials were unaffected until the quench rate fell below 20 °C/s; however, the extensively recrystallized plate appeared more quench sensitive in the low-rate region, losing about 12 pct of its strength at an 8 °C/s quench rate vs. 6 pct for the nearly unrecrystallized material. Figure 4 shows yield strength decreasing by only 15 MPa (2 pct) over the 15 to 80 pct recrystallization range for the fast quench rate vs 45 MPa (8 pct) at the slowest rate. The corresponding decreases in strength at the intermediate quench rates were essentially identical to those at 150 °C/s. Tensile elongation was not highly sensitive to either recrystallization (Figure 5) or quench rate. In all cases, however, the slowest quench rate gave the lowest elongation value.

Figure 6 shows fracture toughness decreasing with decreasing quench rate. The least recrystallized material had the highest toughness (as expected) and was most affected by quench rate (perhaps unexpected). The specific influence of recrystallization on toughness at each quench rate is shown in Figure 7. Toughness decreased almost linearly with the increasing degree of recrystallization, the effect being greatest in plates quenched at the fastest rate. Recrystallization had little effect on slowly quenched material. A multiple regression analysis^[10] of the fracture toughness data gave the following "best-fit" relation between quench rate (Q) and percent recrystallization (R_x):

$$K_{lv} = 14.1 + 7.63 \ln Q - 0.041 R_x \cdot \ln Q$$
 [1]

with a correlation coefficient (r^2) of 0.95 and an estimated standard error of 1.5 MPa \sqrt{m} . An *F*-test of significance at the 95 pct confidence level was used to establish the num-



Fig. 5-Effect of recrystallization on the ductility of 7050-T6 plate.



Fig. 6-Effect of quench rate on the fracture toughness of 7050-T6 plate.

ber of terms to carry in the equation. Note that recrystallization is included only in an interaction term.

The separate and combined effects of recrystallization and quench rate on mechanical properties can be rationalized by a number of possible mechanisms. Quench sensitivity relative to strength is well understood: slow cooling rates promote precipitation on grain boundaries and incoherent dispersoids. As Bryant and Thomas^[11] observed, the outer (recrystallized) regions of extrusions are more quench sensitive from a strength viewpoint than the inner (unrecrystallized) regions. Elongations, however, were relatively unaffected by grain structure. It has also been argued that



Fig. 7-Effect of recrystallization on the fracture toughness of 7050-T6 plate.

recrystallization results in a crystallographic reorientation such that the normally coherent Al₃Zr dispersoids become incoherent, thereby acting as effective nuclei for MgZn₂ precipitation during the quench.^[12,13] Transmission electron microscopy observations that precipitates in recrystallized grain boundaries were larger than those in unrecrystallized regions support the former mechanism, although a contribution from incoherent Al₃Zr dispersoids cannot be ruled out.

Although alloy 7050 is considered relatively quench insensitive from a strength viewpoint, its fracture toughness response is quite sensitive (Figure 6). And, contrary to the strength situation, unrecrystallized grain structures are affected most (at least for the L-T orientation). Explanations based on prior studies, generally model systems of equiaxed higher purity alloys, are not necessarily applicable. As noted earlier, recrystallization in the present system replaces large pancake-shaped grains containing a well-developed subgrain-structure with less elongated subgrainfree grains.

Fracture in equiaxed recrystallized Al-Zn-Mg-(Cu) alloys is predominantly intergranular (IG) in nature. The amount of IG fracture increases with area fraction (A_i) of grain boundary precipitate,^[9] with the fracture energy inversely related to $\sqrt[n]{A_{e}}^{[14]}$ Notably, large variations in grain boundary precipitate density (and fracture toughness) can occur without significant changes in tensile properties.^[9] The presence of subgrains in unrecrystallized (and elongated) structures leads to a combination of transgranular and intersubgranular (ISG) fracture; and as the amount of precipitate on the subgrains increases, the relative amount of ISG fracture increases, with a concomitant decrease in toughness (analogous to the equiaxed recrystallized situation). Slowly quenched material, therefore, has low toughness independent of recrystallization, since fracture is either intergranular or intersubgranular, with the fracture energy lowered by



Fig. 8—Strength-adjusted fracture toughness vs quench rate assuming a relation of the form $Klv = AQ^m$.

boundary precipitates in both cases. At higher quench rates, boundary precipitation is minimized, in which case a nearly equiaxed recrystallized structure is inherently more susceptible to IG fracture when stressed in the longitudinal and long-transverse directions.^[5]

Since fracture toughness has been theoretically and experimentally related to fractional grain boundary coverage, it may be instructive to estimate the effect of quench rate on A_{f} using a simple growth model. At homologous temperatures (T/Ts) below 0.9 (Ts = absolute solidus temperature), lengthening of grain boundary allotriomorphs in the Al-Cu system proceeds by a collector plate mechanism involving both volume and grain boundary diffusion.[15,16] The precipitate length (or diameter in the grain boundary plane) is related to (time)^{1/4} with a temperature-dependent proportionality constant comprised of concentration, diffusion, and surface energy terms. Under continuous cooling conditions, the principle of "additivity" may apply;[17] i.e., the nucleation sites saturate early and the instantaneous reaction rate depends only on the temperature, not on the thermal path. The grain boundary fractional area coverage is then given by

$$A_f = \left(\int_0^t kNdt\right)^{1/2}$$
 [2]

where N is the number of nuclei per unit grain boundary area and k is the aforementioned rate constant. If the cooling rate, Q, *i.e.*, dT/dt, is reasonably linear, then

$$A_{f} = \frac{1}{Q^{1/2}} \left(\int_{T_{1}}^{T_{2}} kNdT \right)^{1/2}$$
 [3]

where the integral has a constant (time-independent) value. Since fracture energy, G, is proportional to $1/\sqrt{A_f}$ and $G \propto K_i^2$, then K_i should be proportional to $Q^{1/8}$. As Figure 8 shows, strength-adjusted toughness data (~1 MPa \sqrt{m} per 7 MPa in yield strength) are reasonably consistent with such a relationship.

In summary, slow quench rates and recrystallization reduce strength and fracture toughness of 7050-T6 plate, as expected, and the effects on toughness are much greater than those on strength. Recrystallization has the largest influence on the toughness of well-quenched material. Loss of toughness is associated with precipitation on both highangle and subgrain boundaries, and the results are qualitatively consistent with fracture energy being inversely dependent on fractional area coverage.

REFERENCES

- 1. I. Kirman: Metall. Trans., 1971, vol. 2, pp. 1761-70.
- 2. G.T. Hahn and A.R. Rosenfield: Metall. Trans. A, 1975, vol. 6A, pp. 653-68.
- J.T. Staley: in Properties Related to Fracture Toughness, ASTM STP 605, 1976, pp. 71-96.
- J.T. Staley: Fracture Toughness and Microstructure of High Strength Aluminum Alloys, AIME Meeting, Pittsburgh, 1974.
- 5. D.S. Thompson: Metall. Trans. A, 1975, vol. 6A, pp. 671-83.
- 6. J.T. Staley: Mater. Sci. Technol., 1987, vol. 3, pp. 923-35.
- D.S. Thompson, S.B. Subramanya, and S.A. Levy: *Metall. Trans.*, 1971, vol. 2, pp. 1149-60.
- P.C. Varley, M.K.B. Day, and A. Sendorek: J. Inst. Met., 1957-58, vol. 86, pp. 337-51.
- P.N.T. Unwin and G.C. Smith: J. Inst. Met., 1969, vol. 97, pp. 229-310.
- 10. The Statistician, Quant Systems, Charleston, SC, 1981.
- 11. A.J. Bryant and A.T. Thomas: J. Inst. Met., 1972, vol. 100, pp. 40-44.
- H. Suzuki, M. Kanno, and H. Saitoh: *Keikinzoku*, 1983, vol. 33, pp. 399-406.
- 13. S. Kikuchi, H. Yamazaki, and T. Otsuka: J. Mater. Processing Technol., 1993, vol. 38, pp. 689-701.
- 14. J.D. Embury and E. Nes: Z. Metallkd., 1974, vol. 65, pp. 45-55.
- 15. H.B. Aaron and H.I. Aaronson: Acta Metall., 1968, vol. 16, pp. 789-98.
- J. Goldman, H.I. Aaronson, and H.B. Aaron: *Metall. Trans.*, 1970, vol. 1, pp. 1805-10.
- 17. J.W. Cahn: Acta Metall., 1956, vol. 4, pp. 572-75.

Microstructural Anomalies in a W-Ni Alloy Liquid Phase Sintered under Microgravity Conditions

YIXIONG LIU, RONALD G. IACOCCA, JOHN L. JOHNSON, RANDALL M. GERMAN, and SHIRO KOHARA

The gravitational role in liquid phase sintering (LPS) is a problem of great interest in both materials science and engineering practice. Gravity-induced microstructural gradients in grain size, grain shape, and solid volume fraction have been well documented in liquid phase sintered tungsten heavy alloys^[1,2,3] and have been analyzed by a number of theoretical models.^[1,4] However, gravity may have many unknown effects on LPS, which can only be revealed by experiments conducted under microgravity conditions. This article reports

Manuscript submitted September 19, 1994.

YIXIONG LIU, Research Associate, RONALD G. IACOCCA Director, Materials Characterization, JOHN L. JOHNSON, Director, Materials Development, and RANDALL M. GERMAN, Brush Chair Professor in Materials, are with the Engineering Science and Mechanics Department, The Pennsylvania State University, University Park, PA 16802-6809. SHIRO KOHARA, Professor, is with the Department of Materials Science and Technology, Faculty of Industrial Science and Technology, The Science University of Tokyo, Noda, Chiba 278, Japan.