D. Equal Channel Angular Extrusion Processing of Alloys for Improved Mechanical Properties

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Objective

- Investigate equal channel angular extrusion (ECAE) as a deformation processing technique to manipulate the microstructure and improve material properties such as strength, formability, fatigue, and corrosion.
- Apply ECAE processing to produce advanced, lightweight materials with enhanced formability, higher strength-to-weight ratio, and higher stiffness, ultimately leading to reductions in vehicle weight and thus more fuel-efficient vehicles.

Approach

- Assess the effects of ECAE processing parameters on the mechanical properties of a magnesium alloy (ZK60A) and aluminum metal matrix composites (MMC). These are lightweight alloys with potential for use in lightweight structural applications in vehicles.
- Determine the optimum ECAE processing schedule for the maximum increase in mechanical properties.
- Characterize the microstructure and mechanical properties of the ECAE-processed material.

Accomplishments

- Demonstrated extreme microstructural refinement in magnesium alloy by low temperature ECAE processing.
- Determined superplastic forming potential of ECAE-processed magnesium alloy through strain rate jump tests to determine strain rate sensitivity parameter.
- Performed superplastic tensile tests at $\leq 250^{o}$C on ECAE-processed magnesium alloy.
- Removed residual porosity from a metal matrix composite (MMC) through ECAE-processing.
- Demonstrated an increase in elastic modulus (>15%) of a powder metallurgy MMC with increasing number of ECAE passes.
  - ECAE improved second phase distribution.

– ECAE improved tensile properties (ductility >10% vs <1% as-received).
– ECAE processed similar commercial MMC's made by casting methods.

Future Direction

- Superplastic tensile testing of ECAE-processed magnesium alloy at >250°C.
- Microstructural characterization of ECAE-processed magnesium alloy before and after superplastic tensile testing.
- Publish results of superplastic testing of ECAE-processed magnesium alloy.
- Compare mechanical properties of ECAE-processed MMC made by powder metallurgy (Dynamet AL6061+10 % B_4C) with those of ECAE-processed commercial MMCs made by casting methods.
- Microstructural characterization of ECAE-processed MMCs.
- Publish results of the ECAE processing study of MMCs.

Introduction

The mechanical properties, especially the yield strength and the ultimate strength, of metallic alloys can be improved through a refinement of the microstructure. The Hall-Petch relationship predicts that the increased yield strength is inversely related to the grain size. Deformation processing, e.g., rolling, forging, swaging, etc., followed by heat treatment is traditionally used to reduce the grain size. These deformation processing methods result in a change in the dimensions of the work piece, typically reducing the work piece dimension in at least one direction while increasing one or more other dimensions. Therefore, these methods are geometrically limited in the amount of deformation that can be imparted to the material. However, equal channel angular extrusion (ECAE) is capable of repeatedly imparting a large amount of deformation into a material without significant dimensional changes. (In ECAE, a billet is forced around an internal angle inside a die. The change in direction, without a change in billet dimensions, imparts the deformation.) The goal of this project is to explore the limits of microstructural refinement and the effects on mechanical properties.

During the previous year the research focus for this program was narrowed to two areas. One area involved the use of ECAE processing of lightweight magnesium alloys to develop an extremely fine microstructure suitable for achieving high strain rate superplasticity. The other area is focused on ECAE processing metal matrix composites (MMCs) to homogenize the distribution of the hard reinforcing phase and, thereby, impart significant ductility to these materials, which are typically very brittle.

Significant progress has been made in both areas. Namely, a very fine grain size has been achieved in the magnesium alloy, ZK60A, through ECAE processing, Figure 1. Tensile samples were made and the superplastic potential of this material was evaluated through strain rate jump tests.

In the work with the MMC it was demonstrated last year that ECAE processing increased the density and ductility of a metal matrix composite, AL6061+10 wt% B_4C, by compacting, sintering and hot isostatic pressing AL6061 and boron carbide powders. The

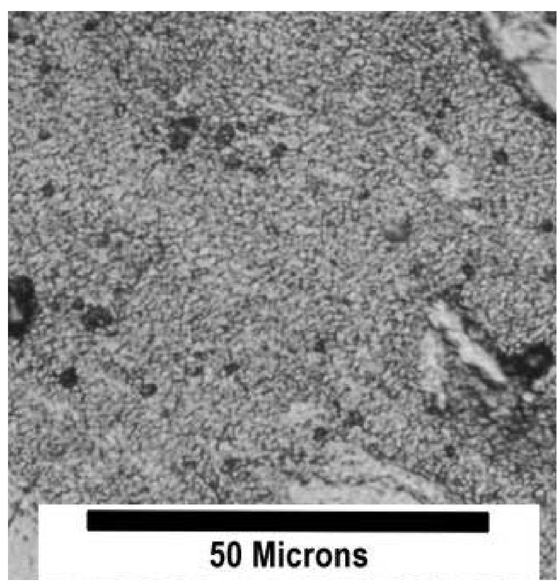

Figure 1. The etched microstructure of ECAE-processed ZK60A.

distribution of the boron carbide phase showed significant signs of homogenization during ECAE and the tensile ductility increased from <1% elongation in the as-received condition to greater than 10% after ECAE processing. ECAE also improved the relative density and the elastic modulus, although these values were still somewhat lower than theoretical. So, new ECAE processing parameters were used to process additional material in an effort to further improve the ductility as well as increase the elastic modulus. Also two commercially available aluminum alloy MMCs made by *casting techniques* were added to the test matrix and ECAE processed.

Both of the materials studied are lightweight and could significantly reduce vehicle weight, increasing fuel efficiency. The goal is to develop lightweight alloys with good formability and high stiffness with significant ductility for application in the transportation sector.

ECAE Processing of Magnesium Alloys

The magnesium alloy billets for superplastic evaluation were ECAE-processed according to Table 1. The ECAE processing schedules shown in Table 1 were meant to explore the effects of processing temperature, processing route and number of ECAE passes on the superplastic forming behavior. Additionally, sample ZK60-S4, -S6 and -S7 attempted to look at the effect of precipitation from solid solution *during* ECAE processing.

Strain rate jump tests were performed at 180, 225, 250 and 275°C on the ECAE-processed samples from the first four billets in Table 1 to assess the potential for superplastic behavior. The strain rate sensitivity factor, *m*, was calculated from:

$$m = \log(\sigma_2/\sigma_1)/\log(\dot{\varepsilon}_2/\dot{\varepsilon}_1) \quad , \qquad (1)$$

where $\dot{\varepsilon}$ was the strain rate, σ was the flow stress and the subscripts 1 and 2 referred to the values before and after the strain rate jump, respectively.

The strain rate sensitivity factor has been plotted as a function of test temperature in Fig. 2 for the first four samples in Table 1.

Table 1. ZK60A ECAE processing schedules

Billet ID	Initial billet condition	ECAE temperature (°C)	ECAE route	Back stress level, ksi
ZK-1	T5	150	8B	15.1
ZK-R1	T5	150 135 120	2B 1B 1B	15.1 15.1 19.7
ZK-R5	T5	150 130	2B 4B	15.1 15.1
ZK60-S4	Solution treated	200 150	2B 6B	10.6 21.2
ZK60-S6	Solution treated	200 150 140 120	2B 2B 2B 2B	11.2 14.0 18.3 22.5
ZK60-S7	Solution treated	200 150 150	2A 1B 1A	11.2 14.0 14.0
ZK-4N	T5	150 140 130 120	2B 2B 2B 2B	16.8 16.8 16.8 22.5

The plots for ZK-R1 and ZK-R5 (4 and 6 ECAE passes, respectively) are quite regular. The strain rate sensitivity passes through a maximum as the temperature increases. Also, as the strain rate increases, the maximum m value occurs at higher temperatures. In fact, for samples, ZK-R1 and ZK-R5, the maximum m value for strain rates $\geq 3 \times 10^{-4}$/second appears to occur at temperatures higher than those tested, probably around 300-325°C.

The other two samples, which have the most ECAE passes, are somewhat more ambiguous. ZK-1 (8 ECAE passes) exhibits a minimum between 225 and 250°C, while the ZK60-S4 sample (8 ECAE passes on a solutionized billet) seems to show the maximum m values occur below 180°C for strain rates *less than* 3×10^{-4}/second while the maximum m values for strain rates of 1.6×10^{-3}/second, 7.8×10^{-3}/second and 3.4×10^{-2}/second occur around 225°C.

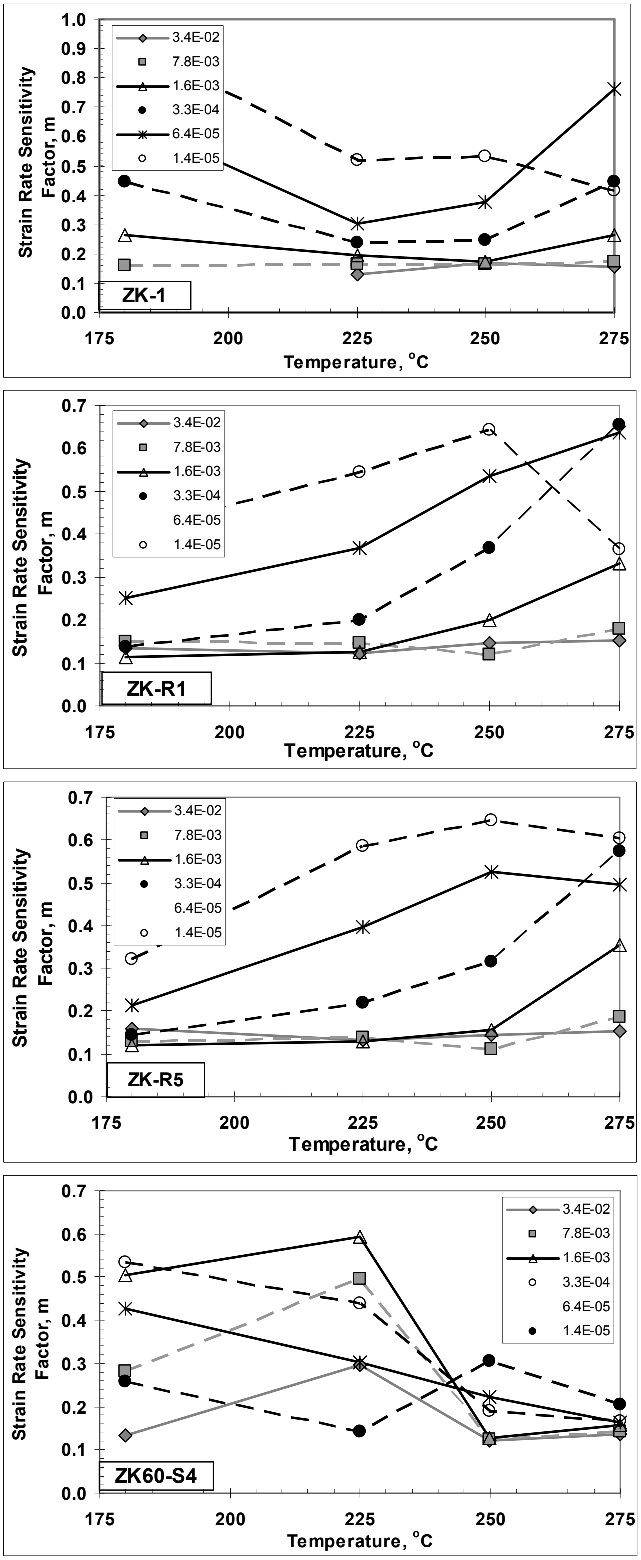

Figure 2. Strain rate sensitivity of ECAE-processed ZK60A at various temperatures.

Based on these strain rate jump tests, certain ECAE processing schedules should produce superplastic behavior. (Superplastic behavior is usually associated with m values greater than 0.5.) Elevated temperature tensile tests were begun to elevate the extent of superplastic deformation possible. From a commercial aspect, a material may be superplastically formed, economically, if a minimum elongation of 200-300% can be attained at a strain rate on the order of 10^{-2}/second or greater. Figure 3 shows an example of the elevated temperature tensile results for a strain rate of 10^{-2}/second. An elongation of greater than 150% has been obtained at 250°C. Table 2 summarizes the results for the samples tested so far. Based on the strain rate jump tests, greater elongations are expected for higher

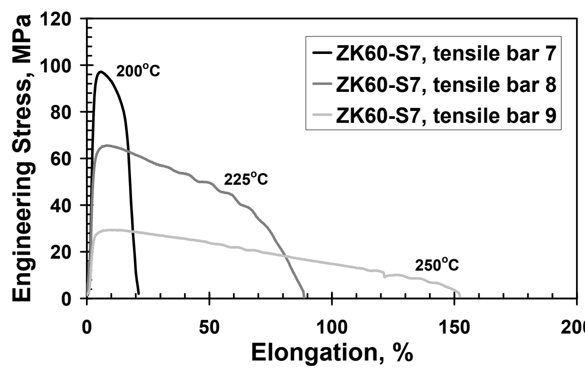

Figure 3. Example of elevated temperature tensile behavior of ECAE processed alloy ZK60. Strain rate = 10^{-2}/second

Table 2. Elevated Temperature Tensile Results

Billet ID	Elongation, %	Test Temperature, °C	Strain Rate, s^{-1}
ZK -1	176	200	0.006
	90	200	0.013
	83	200	0.025
ZK-R1	90	200	0.006
	45	200	0.025
ZK-R5	156	200	0.006
	98	200	0.013
	92	200	0.025
ZK60-S4	188	200	0.006
	181	200	0.013
	150	200	0.025
ZK60-S7	21	200	0.01
	89	225	0.01
	153	250	0.01

temperatures which are planned for early in FY06. Therefore, high-strain-rate superplasticity may still be found in this ECAE-processed magnesium alloy. This would facilitate the use of magnesium alloys in automotive applications. Superplastic forming would allow net-shape fabrication of components and minimize waste of this relatively expensive alloy, making it more economically competitive with traditional iron-based materials. High-strain-rate superplastic forming also offers the potential for rapid fabrication, resulting in higher productivity and making this alloy more competitive with conventional automotive structural materials. Ultimately, vehicles incorporating magnesium alloys would be lighter than those that rely on traditional alloys, and therefore more fuel-efficient.

ECAE Processing of Aluminum Alloy Composites

Two commercial metal matrix materials were added to this research area during the first half of FY05. The materials were made by Duralcan and were obtained from Dr. Darrell Herling at PNNL. The first material, designated F3S.20S-T6**,** consisted of an aluminum alloy 356 matrix with 20 wt% SiC particulate and was made by a casting process. In the as-received condition, this MMC exhibited an elastic modulus (106.8 GPa) that was approximately 55% greater than typical aluminum alloys. The other material, designated W6A.15A-T6, consisted of an AL6061 matrix with 15 wt% Al_2O_3 particles. This material was made by extrusion of powdered material and exhibited an elastic modulus approximately 28% greater than typical aluminum alloys. Both materials were fully dense and exhibited well-dispersed particulate reinforcement, Figure 4. The particles in the SiC composite are on the order of 10-15 microns while those in the Al_2O_3 composite are on the order of 20-30 microns. The boron carbide particle size in the AL6061/B_4C composite made by Dynamet is on the order of 50 microns. The commercial composites will be used to compare the effects of ECAE on the prototypical Dynamet material with MMCs made by other means. The goal is to determine the utility of ECAE processing of MMCs of all types.

The specific schedules used are shown in Table 3. The ECAE-processed billets remained intact and were free of cracks and other flaws commonly found

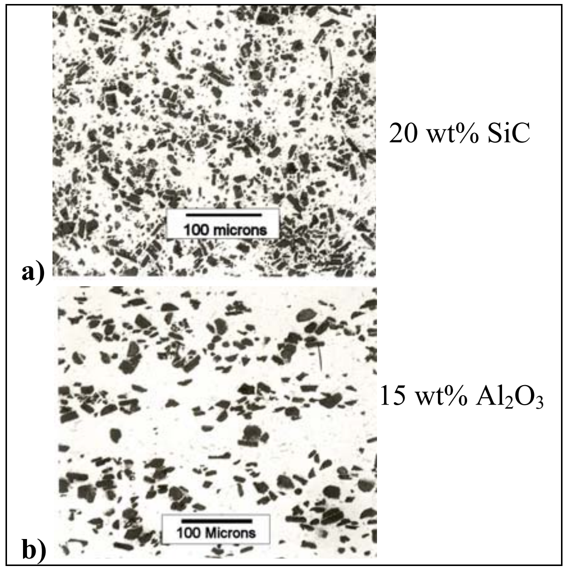

Figure 4. The as-received microstructure of a) F3S.20S-T6 and b) W6A.15A-T6

Table 3. Processing schedules for Commercial MMCs

Sample	Initial condition	ECAE temperature (°C)	ECAE route	Back stress (ksi)
F3S.20S-2	solutionized	300	2B	14.0
		150	2A	19.7
		100	1A	21.1
W6A.15A-1	solutionized	300	2B	5.6
		150	2A	19.7
		100	1A	21.1
B3528-10	T0	300	2B	9.8
		150	2A	21.1
		100	1A	21.1

in materials of low ductility that are ECAE-processed without back pressure. Tensile bars were made and tested for B3528-10 (Dynamet material). Tensile bars for the other two ECAE-processed MMCs are being made and will be tested in early FY06. This sample set will allow the benefits of ECAE processing on MMCs to be determined.

However, the effect of ECAE processing parameters on the elastic modulus and the density for the different MMCs was determined after each ECAE pass of the processing schedule using ultrasonic methods and the water immersion technique, respectively. Figure 5 shows the variation of these quantities as the billets were ECAE processed. Unfortunately this behavior was not determined for

B3528-10 so sample B3528-7 has been included for reference. The obvious difference is that ECAE processing seems to have quite a large initial effect on the prototypical Dynamet material (B3528-7) where as ECAE processing has little effect on the commercial MMCs. ECAE processing of the Dynamet material removes residual porosity and increases the relative density which results in a significant increase in the elastic modulus over the as-received billet. After the first pass through the ECAE die, subsequent passes do not have as great an effect on either the density or elastic modulus for B3528-7, i.e., the ECAE effect becomes similar to those observed in the fully dense commercial MMCs. Tensile testing will determine whether ECAE processing has produced a beneficial refinement of the microstructure which yields improved mechanical properties such as strength and/or ductility.

Conclusions

ECAE processing has the potential to refine the microstructure and improve the mechanical and/or formability of commercial lightweight alloys. Work during this fiscal year has shown that ECAE processing may enable a magnesium alloy to be superplastically formed at relatively high strain rates ($\geq 10^{-2}$/second). Net shape superplastic forming, with little material waste, may enable the use of this, and other, ECAE-processed magnesium alloys in transportation applications to reduce vehicle weight and increase fuel efficiency.

ECAE processing may also be used to remove residual porosity of lightweight, high stiffness metal matrix composites produced by powder metallurgy techniques. Additional benefits of ECAE processing of MMCs appear to be improved distribution of the elastically hard, particulate phase as well as increase in the ductility of the MMC. It remains to be seen if ECAE processing has similar effects on MMCs made by casting or extrusion. Work in early FY06 should provide insight into this issue.

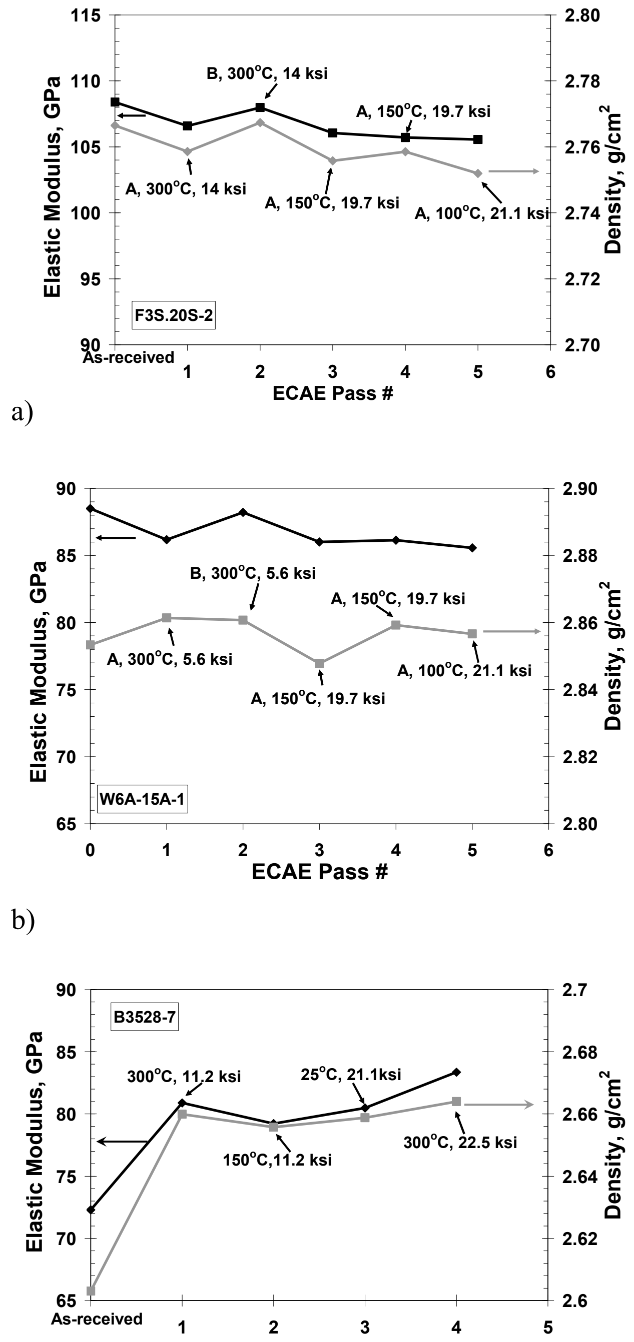

Figure 5. Effect of ECAE processing on elastic modulus and density. a) F3S.20S-2, b) W6A.15A-1, c) B3528-7

Publications and Presentations During FY05

T.M. Lillo, "Enhancing Ductility of AL6061+10wt% B_4C Through ECAE Processing", poster session in the Langdon Symposium at the 2005 TMS Annual Meeting & Exhibition in San Francisco, CA. A manuscript has been accepted for publication in Materials Science and Engg. A.

S.R. Agnew, P. Merhota, T.M. Lillo, G.M. Stoica, P.K. Liaw, "Texture Development During the Equal Channel Angular Pressing of Magnesium Alloys AZ31, AZ80, Mg-4wt% Li, WE43 and ZK60", presented at 2005 TMS Annual Meeting & Exhibition in San Francisco, CA. Manuscript has been prepared for proceedings.

D.P. Field, R.C. Eames, and T.M. Lillo, "The Role of Shear Stress in the Formation of Annealing Twin Boundaries in Copper" submitted to Scripta Materialia.

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Agnew, S.R., Mehrotra, P., Lillo, T.M., Stoica, G.M., "Texture evolution of five wrought magnesium alloys during route A equal channel angular extrusion: Experiments and simulations", *Acta Materialia,* Volume: 53, Issue: 11, June, 2005, pp. 3135-3146.