AN ABSTRACT OF THE THESIS OF

VAUGHN WILLIAM ABBOTT for the MASTER OF SCIENCE
(Name) (Degree)

(Materials Science)
in Mechanical Engineering presented on AUGUST 31, 1967
(Major) (Date)

Title: THE EFFECT OF SMALL AMOUNTS OF MAGNESIUM ON THE SUPERPLASTIC BEHAVIOR OF AN ALUMINUM-ZINC ALLOY

Abstract approved: [redacted]
Olaf G. Paasche

The base Al-Zn superplastic alloy was investigated at 250°C to determine the effect of small amounts of magnesium on mechanical properties. Six alloys of nominal composition 0.00, 0.10, 0.25, 0.50, 0.75, and 1.00 weight percent magnesium, constant 78 weight percent zinc, and variable 21 to 22 weight percent aluminum were each tested in tension at strain rates of 0.02, 0.20, and 2.00 in/min to determine the flow stress and elongation at each strain rate for each composition.

Superplastic elongation occurred at all three strain rates in the specimens containing no magnesium. The addition of any magnesium content investigated destroyed any significant superplastic deformation and led to intercrystalline fracture of all other test specimens. The intermetallic compound Mg_2Zn_{11} was uniformly

distributed throughout the microstructure, limiting plastic deformation. In general, the flow stress was found to increase to a maximum as the magnesium content was increased up to about 0.75 percent, then to start dropping off. At the same time, after the initial drop in elongation due to the change from the superplastic to the intercrystalline fracture mode, the trend in elongation was to decrease slowly with increasing magnesium content. For a given magnesium content, a higher strain rate resulted in a higher value for the flow stress.

The Effect of Small Amounts of Magnesium on the Superplastic Behavior of an Aluminum-Zinc Alloy

by

Vaughn William Abbott

A THESIS

submitted to

Oregon State University

in partial fulfillment of
the requirements for the
degree of

Master of Science

June 1968

APPROVAL:

[redacted]

Professor of Department of Mechanical Engineering

in charge of major

[redacted]

Head of Department of Mechanical Engineering

[redacted]

Dean of Graduate School

Date thesis is presented AUGUST 31, 1967

Typed by Clover Redfern for Vaughn William Abbott

ACKNOWLEDGMENTS

This opportunity is taken to acknowledge several individuals who have contributed much to the development of this thesis. Foremost appreciation is due Professor Olaf G. Paasche. As major professor, his invaluable advice, encouragement, and assistance have been sincerely appreciated throughout the investigation.

The many discussions with Professor William D. McMullen have added much to the background and preparation of this study. Assistance from Mr. John R. Kellogg and Mr. David G. Goodrich is gratefully acknowledged.

The Marshall controller used during the investigation was made available by Mr. Ronald R. Lowery of the U.S. Bureau of Mines, Albany, Oregon.

TABLE OF CONTENTS

	Page
INTRODUCTION	1
THEORETICAL BACKGROUND	3
Soviet Research	3
The Solution-Precipitation Theory	3
The "Metastability" Theory	5
American Research	8
The Competing Processes Theory and Related Corollaries	8
An Alternative Competing Processess Theory	17
A High Temperature Creep Interpretation	19
A Dislocation Model	20
THE EXPERIMENT	24
Selection of the Experimental Alloy	24
Purpose of the Experiment	26
Equipment and Experimental Procedure	26
RESULTS	33
DISCUSSION AND INTERPRETATION OF RESULTS	39
Superplastic Deformation	39
The Effect of Magnesium on Superplastic Deformation and Flow Stress	40
Microstructural Effects	41
Comparison with Previous Work	44
CONCLUSIONS	46
RECOMMENDATIONS FOR FUTURE RESEARCH	47
BIBLIOGRAPHY	49

LIST OF FIGURES

Figure		Page
1.	Graphical illustration of the two competing processes in the Avery-Backofen theory.	11
2.	Possible methods for conserving compatibility between grains at a triple junction during grain boundary shear.	15
3.	Aluminum-Zinc phase diagram.	25
4.	Selected partial ternary diagrams of the Al-Mg-Zn system.	27
5.	Specimen dimensions (inches).	29
6.	Schematic illustration of specimen, testing grips, and furnace in testing position on the Instron testing machine.	30
7.	Breakdown of testing grips and specimen.	31
8.	Experimental test setup showing the Instron testing machine with furnace equipment in place, the Marshall controller, and test monitoring equipment.	31
9.	Representative load-elongation curve for the specimens tested in this investigation.	33
10.	Photomicrograph of typical Al-Zn eutectoid microstructure. Undeformed grip section, 500x.	35
11.	Photomicrograph of typical microstructure of the Al-Zn base alloy containing one percent magnesium. Undeformed grip section, 500x.	35
12.	Comparison of fractures for the set of specimens tested at 0.02 in/min at 250°C.	36
13.	Change in flow stress as a function of the amount of magnesium present in the Al-Zn alloy.	38
14.	Change in elongation as a function of the amount of magnesium present in the Al-Zn alloy.	38

LIST OF TABLES

Table	Page
1. Possible rate controlling processess in superplasticity.	16
2. Experimental results for the alloys tested.	37

THE EFFECT OF SMALL AMOUNTS OF MAGNESIUM ON THE SUPERPLASTIC BEHAVIOR OF AN ALUMINUM-ZINC ALLOY

INTRODUCTION

The phenomenon of unusually large elongations found in crystalline metals and alloys under an applied load is termed superplasticity (2, 8). Superplasticity has been connected with a strong strain rate dependence on flow stress (5, 7, 24) and has been experimentally observed in materials tested at elevated temperatures in the creep range (2, 5, 24), materials having a finely divided microstructure (5), or in materials of eutectic or eutectoid composition (5, 7, 21, 22, 24).

Underwood notes that such unusually large ductility effects during mechanical deformation have been known to occur during phase changes or allotropic transformations for many years. Since many of the more metallurgically important phase changes occur more readily at elevated temperatures, it was perhaps natural that superplasticity was first considered as an elevated temperature phenomenon. One of the earliest studies of such enhanced ductility was conducted by Sauveur in 1924 when he tested iron in both tension and torsion throughout a temperature range from 600°C to 1200°C (24). Due to the economic importance of iron-base alloys, these have since been the subject of continuing research on such ductility effects.

One of the earliest systematic investigations of extremely high ductility in other alloy systems was conducted by Pearson in 1934. He conducted elongation tests on lead-tin and bismuth-tin eutectic alloys discovering elongations approaching 2000% (20).

It is significant to note that the phenomenon of superplasticity was discovered primarily in studies of creep resistance. Such studies were concerned with avoiding such extensive ductility and not in the exploration of how to achieve it (2). As in many scientific processes, superplasticity has advanced from the laboratory curiousity stage through the "how to avoid" stage to the research stage as means to use it were found. Present research is pointed toward isolating and identifying the mechanism or mechanisms causing superplasticity. Once this has been satisfactorily accomplished, the superplastic phenomenon may well be exploited for practical engineering and economic advantage. Fabrication processes employing the superplastic materials may well be a useful technological and economic achievement within the next two decades.

It is the objective of this thesis to examine the past and present research findings in the growing field of superplasticity and apply these to additional research on one alloy system.

THEORETICAL BACKGROUND

Soviet Research

The father of modern superplasticity would undoubtedly be the Soviet researcher Bochvar. As Underwood notes (24), a 1945 technical paper by Bochvar and Sviderskaya reported that elongations of up to 650% were not enough to cause failure in a near eutectoid Al-Zn alloy which had been quenched from above the eutectoid reaction horizontal and tested in tension below it. Through his primary experience in the field of high temperature creep resistance, Bochvar developed a theory for plasticity in two-phase alloys. This theory, known as the "solution-precipitation" theory, is the original Soviet model for superplasticity.

The Solution-Precipitation Theory

Introduced in 1948, the solution-precipitation theory of enhanced plasticity in two-phase alloys predicts that relative movement of the two or more phases present is assisted by the mutual solution and precipitation of the phases. The three general requirements appear to be:

1. a fine dispersion of the phases present,
2. a significant variation in composition of at least one of the phases with temperature,

3. high diffusion rates at the deformation temperature (8, 10, 24).

In effect this model predicts that small local temperature variations within the specimen during deformation lead to a solution process at some phase interfaces and to a precipitation process at the others (8). This compositional change requires diffusion which results in viscous deformation or superplasticity. The more pronounced the variation in composition with temperature, the finer the dispersed structure, and/or the higher the diffusion rates, the more superplastic the alloy should be (10).

The American investigator Chaudhari notes, however, that the significant compositional change with temperature is not always needed for superplasticity to occur. Also, he suggests that if temperature gradients due to external environments are excluded, internal temperature fluctuations of long enough duration are not likely present in which extensive mass transfer can take place in the required direction (8). Several Soviet researchers from both the fields of creep and superplasticity have cast doubts as to the generality of Bochvar's superplasticity requirements (10, 24).

On the other hand, American researchers Guy and Pavlick have supported Bochvar's model but have suggested a slightly different order in accounting for their experimental results. In their creep research on the Sn-Sb system, they conclude that, rather than

Bochvar's proposed precipitation and resolution of a second phase within the first which increases the atomic mobility in addition to the usual diffusion effects, the reverse is true. This is, that the local structural effects resulting from the deformation process may allow for non-equilibrium formation of embryos of a second phase within the first. Deformation in the vicinity of the embryos due to this incipient phase transformation then leads to the enhanced creep behavior. The localized deformation could even cause the embryos to redissolve by virtue of an environmental change. They agree with Bochvar in observing that a cycle of precipitation and resolution is necessary to account for the enhanced ductility effects observed (10).

The "Metastability" Theory

The other primary Soviet superplasticity model has its foundation in the observation that superplasticity has been primarily found in eutectic and eutectoid alloys quenched from above their invariant temperatures. Soviet investigators Presnyakov and Chervyakova have associated superplasticity in such alloys with the amount of metastability of the quenched structures (21, 24). They found in testing the Al-Cu eutectic alloy and the Al-Zn eutectoid alloy that the existence of metastability is necessary for superplasticity to be observed. They feel that superplasticity is the result of powerful diffusional movements of atoms accompanying the segregation process of a solid solution in the direction of the applied load. This appears to occur in two separate stages: stage one occurs spontaneously without the need for external influence during the quenching process, and stage two is the complete stabilization of the alloy which can only occur at elevated temperatures under an applied stress. Superplasticity occurs during this second stage and is evidently associated with the weakening of the atomic bond which takes place during reconstruction of the crystalline lattice (21). Presnyakov has investigated a large number of eutectic and eutectoid alloy systems but has yet to systematically correlate superplasticity with metastability (22, 24).

Chaudhari doubts the presence of such a metastable phase as a requirement for superplasticity because no metastable phase has yet been reported. According to Chaudhari's analysis, Gebhardt and Schultz and Sauerwald have found the quenched and appropriately heat-treated Al-Zn alloy to have the same lattice parameter, crystal structure, and composition as the slowly cooled non-superplastic alloy (8). Presnyakov did find such a change in lattice parameter with alloy treatment which he attributed to metastability since the greatest superplasticity occurred at the greatest deviation from the normal lattice spacing (21).

American researchers Avery and Backofen have demonstrated that quenching is not always necessary for superplasticity. Laminar composites containing alternate layers of pure lead sheet and pure tin sheet were rolled and tested at room temperature and found to be superplastic (5). Martin and Backofen also achieved superplasticity in electroplated Sn-Pb layers, again without the benefit of quenching the alloy (18). Chaudhari notes that the importance of quenching is primarily to introduce a fine distribution of the two phases present rather than to introduce a metastable state in the alloy (8).

The two not necessarily conflicting models of Bochvar and Presnyakov form the primary Soviet explanations for superplasticity and conform to the more general Soviet model that the application of stress at elevated temperatures can lead to significant microstructural changes in solid solutions. Such a stressed condition is said to promote an atomic rearrangement that facilitates the segregation, nucleation, and growth of phases which can become stable under the applied stress. An increased atomic diffusion in the direction of the stress gradient then reduces internal stresses in the phases and tends to restore the distorted lattice to equilibrium again. The lattice is again able to undergo further plastic deformation (24). Note that Bochvar's model specifies the mechanism involved in causing such microstructural changes, i.e., the mutual solution and precipitation of the phases. Presnyakov on the other hand offers no specific mechanism to account for the metastability which accounts for superplasticity except to suggest a two stage breakdown of the metastable state.

American Research

American researchers have approached the field of superplasticity from relatively more diverse directions than their Soviet counterparts. Historically the Soviets have conducted superplasticity research since the end of World War II while such interest in this country did not become apparent until the early 1960's. Since their entry into the field, the American investigators have presented several different interpretations of the phenomenon and research has mushroomed. The major findings and theories of these research programs are presented in this section.

The Competing Processes Theory and Related Corollaries

Certainly the most active American investigators in the field of superplasticity have been Professor W.A. Backofen and colleagues at the Massachusetts Institute of Technology. In leading the American assault on the mechanism or mechanisms involved, they suggest that the phenomenon should be understood in relation to conditions for preserving plastic stability under tensile loading. The Soviet basis of structural metastability leading to low strength is not thought possible to entirely account for the extraordinary neck free elongations experimentally observed (7).

Suggested as phenomenological explanation of such neck free elongation is a strong strain rate dependence on the flow stress, increasing the necking resistance (5, 7). As a quantitative measure of strain-rate sensitivity, Backofen and colleagues have initiated the use of the strain-rate sensitivity index, "m". The expression relating the flow stress and strain rate is

$$\sigma = k\dot{\epsilon}^{m}, \tag{1}$$

where

σ = stress,

$\dot{\epsilon}$ = strain rate,

k = constant (7).

They have shown that as "m" becomes greater than about 0.3, or roughly the upper limit as found in more non-superplastic material at the same temperature, necking resistance rapidly increases (5).

Avery and Backofen have proposed that Nabarro-Herring creep and dislocation climb are the two competing mechanisms at the heart of superplasticity (5). Nabarro-Herring creep is the name given to a diffusional flow of matter occurring within each crystal grain causing local yielding which can be macroscopically described by a viscous flow proportional to the square of the linear grain dimensions (13). This vacancy migration within grains can be mathematically expressed as

$$\dot{\epsilon} = \frac{A\sigma}{L^2} , \quad (2)$$

where

L = length of the vacancy diffusion path, or roughly equal to the metallographic mean-free path between interphase boundaries,

A = a constant for any given temperature related to the diffusion constant and atomic volume,

σ = stress,

$\dot{\epsilon}$ = strain rate (5).

As the second competing mechanism, Avery and Backofen note that climb regulated movement of dislocations across the grains is reasonably expected in materials in which the constituent phases are deforming together--excluding dispersion and precipitation hardened materials (5). Mathematically this process may be represented as

$$\dot{\epsilon} = B \sinh \beta\sigma , \quad (3)$$

where

B = a constant for any given temperature related to the diffusion constant, moving dislocation density, interjog distance, and the Burgers vector,

β = a constant for any given temperature related to the activation volume,

σ = stress,

$\dot{\epsilon}$ = strain rate (5).

When combined, Equations 2 and 3 form the mathematical model of the Avery-Backofen theory,

$$\dot{\epsilon} = \frac{A\sigma}{L^2} + B \sinh \beta\sigma \quad . \tag{4}$$

Figure 1 graphically illustrates the individual contribution each term makes on stress-strain rate axes.

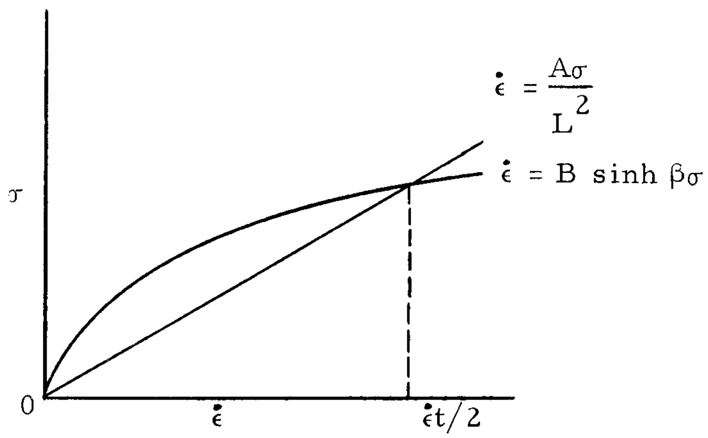


Figure 1. Graphical illustration of the two competing processes in the Avery-Backofen theory.

Avery and Backofen have further defined a transition strain rate as the strain rate where the contribution of each competing process is equal. They propose this transition strain rate to be an indicator of superplasticity and that the greater its value, the greater the probability superplasticity can be observed at normal strain rates rather than strain rates in the creep range. Upon passing from the low to the high side of the transition strain rate, a change in "m" is expected from a high value representing a strong Nabarro-Herring creep contribution to a lower "m" value representing a more normal dislocation climb to be the rate controlling process. At the same time, the trend is away from superplasticity toward a more normal type of plasticity (5).

To strengthen their hypothesis, Avery and Backofen tested extruded Sn-Pb eutectic alloys of varying grain sizes at different strain rates. By decreasing the metallographic mean free path, the transition strain rate increased, showing high "m" behavior to higher strain rates. They have concluded that superplasticity is associated with a relatively high transition strain rate below which strongly viscous flow is observed and above which the more conventional climb regulated dislocation movement occurs (5).

Their previous experiment with the Al-Zn eutectoid alloy did not agree with predictions made with their competing processes theory. In rationalizing this anomaly, Avery and Backofen suggested that strain enhanced diffusion takes place, overriding the strain-rate contribution of dislocation climb (5). In a critique of Avery and Backofen's Sn-Pb eutectic work, Jones and Johnson agree that diffusional flow between closely spaced sinks and sources is the rate controlling process in superplasticity but disagree as to the diffusion path. They suggest that Nabarro-Herring creep takes place by grain boundary diffusion rather than by intercrystalline diffusion. Substantiating their interpretation, they point out that the self-diffusion coefficients calculated using Avery and Backofen's results in the Nabarro-Herring relation are several orders of magnitude different than those found from tracer experiments on the base metals (16). In reply, Avery and Backofen have suggested that the diffusion path taken is the one requiring the least diffusion time regardless of whether by grain boundaries or through the grains. In practice they suggest it would include both and would then still be proportional to the metallographic mean free path and thus still retain the squared relationship in their superplasticity mechanism model (16).

In more recent experiments on the Al-Cu eutectic alloy, Holt and Backofen have modified their model by pointing out that although Nabarro-Herring creep may be the rate controlling process, it cannot alone account for the superplastic deformation. They rationalize this by noting that theoretically, Nabarro-Herring flow would lead to elongated grains relative to the elongation of the structure. Since viscosity increases rapidly with a lengthening diffusion path, this process becomes self-exterminating. Although in his earlier study Backofen rules out grain boundary shear as a possible mechanism, he concludes that this is the rate controlling mechanism for the Al-Cu alloy. But to conserve compatibility, at least one other process is necessary to relax stress concentrations at triple points. It should be noted that if indeed grain boundary shearing were to become the rate controlling process, the stress would be a linear function of grain size rather than the squared function as found in Nabarro-Herring creep (14).

Holt and Backofen propose the four possibilities shown in Figure 2 as methods for preserving compatibility during grain boundary shear (14). During Nabarro-Herring flow, Figure 2a shows lattice diffusion along stress gradients and stress is the squared function of grain size. As has been already noted, Jones and Johnson have suggested the diffusion path lies along the grain boundaries, in which case the grain size dependence on stress would be to the third power (16). And recall that grain boundary shear is a linear function of the stress. The other possibilities illustrated are slip with recovery (2b), slip followed by boundary migration (2c), and recrystallization (2d) (14).

It is now evident that American researchers have proposed three different rate controlling processes based upon atomic movement. These are summarized in Table 1 (14).

Thus as a general requirement, the mechanism or mechanisms involved must account for the high strain rate sensitivity of the flow stress which is also proportional to L^a where "a" must be greater than zero. Holt and Backofen use this to point out that

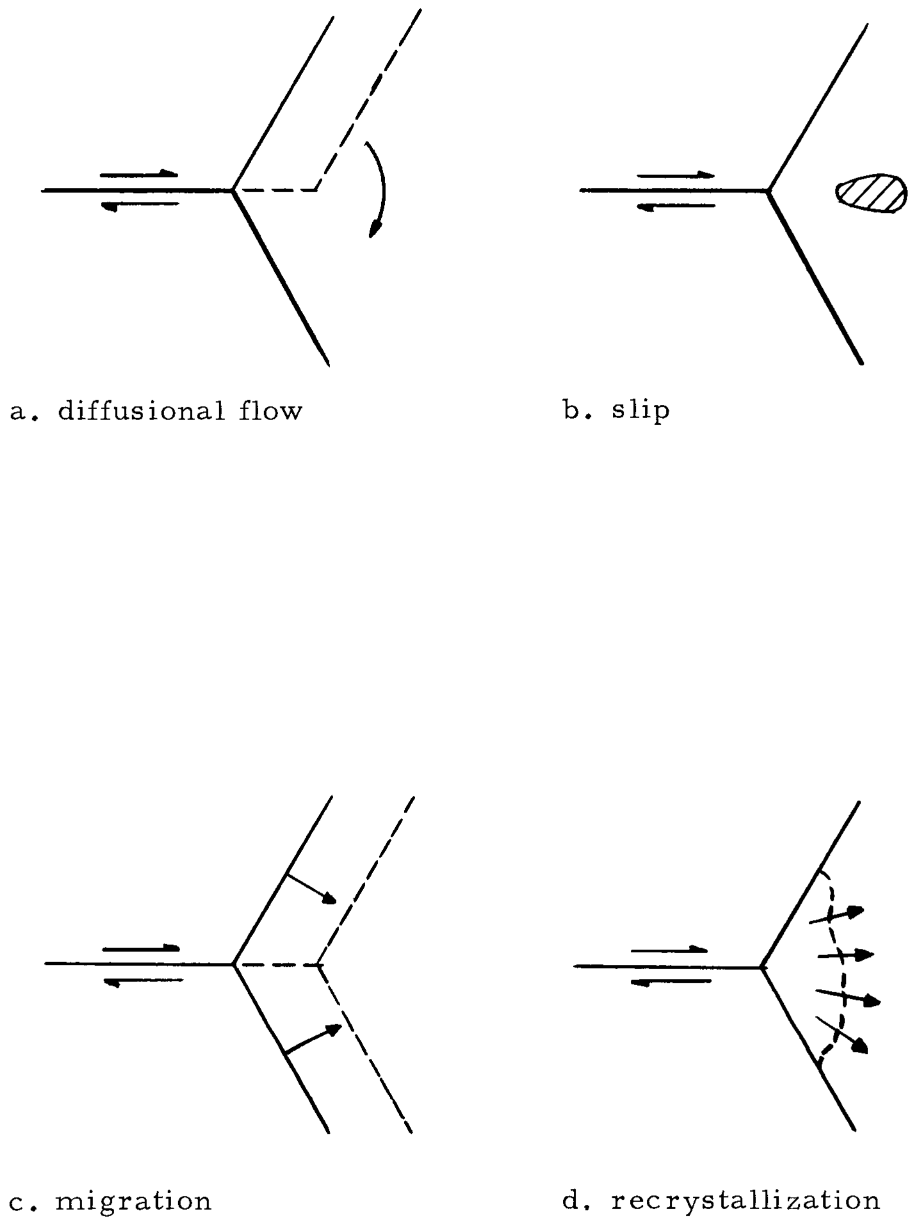


Figure 2. Possible methods for conserving compatibility between grains at a triple junction during grain boundary shear (14).

dislocation arguments alone are not satisfactory in predicting superplastic behavior in either case since "m" values are usually less than 0.3 and "a" values are less than zero (14).

Table 1. Possible rate controlling processes in superplasticity.

Process	"a"	"m"
Grain boundary shear	1	1 (assumed)
Nabarro-Herring creep	2	1
Viscous creep from boundary diffusion	3	1

where $\sigma = L^a$

Source (14).

In conclusion, Holt and Backofen propose that in the Al-Cu eutectic alloy, grain boundary shear is the rate controlling process. In relating a high strain rate sensitivity as a direct result of such grain boundary shear, they suggest that mechanical obstructions to sliding are reduced by strain enhanced migration and recrystallization, (Figures 2c and 2d), (14). They found at low strain rates that boundary obstructions are present and "m" is low while at higher strain rates, boundaries become smoother and stress is determined by viscous boundary drag. They also found, however, that strain rate sensitivity went through a maximum and then decreased as strain rate increased yet higher. It was concluded that at both high and low strain rates the low rate sensitivity observed was mainly due to deformation of bulk material. At intermediate strain rates, high rate sensitivity is due to deformation by grain boundary shear connected with boundary migration (14).

In a more recent experiment, Holt worked with the Al-Zn eutectoid alloy in an effort to determine how grain boundary shear varies with strain rate (15). He concludes that at both high and low strain rates where "m" is low, grain boundary shear made up less than 30 percent of the total deformation, the rest being due to slip within the grains. At intermediate rates and high "m", the contribution is on the order of 60 percent. He notes that at low strain rates boundary shear is limited due to the need for deformation at boundary ledges and triple points. Rate sensitivity then reflects the strain rate sensitivity of the bulk material. His observations of boundary shear being greatest when the strain rate sensitivity of flow stress is highest and that boundary migration enhances such shear support the model proposed in his work on the Al-Cu eutectic alloy (15, 14).

An Alternative Competing Processes Theory

Another American approach to the superplasticity phenomenon has been taken by Packer and Sherby (19). They have analyzed Avery and Backofen's experimental work with the Sn-Pb eutectic alloy and have discovered what they consider to be three inconsistencies in the theory. First they show a lack of agreement between diffusivities calculated from the Nabarro-Herring process using Avery and

Backofen's data and those obtained from tracer experiments. Second, they point out that the strain rate sensitivity index "m" approaches 0.5 at low strain rates and not 1.0 as if the Nabarro-Herring model is appropriate. The third inconsistency is that since the climb regulated dislocation process term predominates at all strain rates tested, the Avery-Backofen model does not describe the true superplastic behavior of eutectic alloy systems. They conclude that the Nabarro-Herring creep process probably is not the rate controlling factor in plastic flow behavior of this alloy (19).

Instead, they suggest an alternative model of two competing processes, one term accounting for a recrystallization or grain boundary migration process and the other term being Weertman's theoretically derived expression describing the creep behavior of metals at high strain rates when dislocation climb is the rate controlling process. This relation mathematically appears as

$$\dot{\epsilon} = \frac{A'\sigma^2}{L^3} + B'\sigma^2 \sinh \beta'\sigma^{2.5} \tag{5}$$

where

A', B', β' = material constants,

σ = creep stress,

L = grain diameter,

$\dot{\epsilon}$ = creep rate (19).

Packer and Sherby feel the most demanding test for a superplasticity relation is its ability to predict both the strain rate to stress and the strain rate to "m" behavior. According to their analysis, Avery and Backofen's model does not meet this standard. Using their new model, Packer and Sherby find their predictions meet this requirement (19).

Packer and Sherby also conducted an experiment on the Al-Zn eutectoid alloy in which they found evidence interpreted as recrystallization or grain boundary migration taking place during the superplasticity process (19).

A High Temperature Creep Interpretation

The American researcher Alden has approached superplasticity from a different direction (2). He interprets his results from experiments on Sn-Bi alloys as being consistent with the literature in the field of high temperature creep. Two distinct deformation mechanisms are established that are rate controlling at high and low strain rates. The dominant low strain rate mechanism is grain boundary shear while the high strain rate mechanism is slip. The region of transition where grain boundary shear is just becoming a significant deformation mode is the region of superplasticity. However, he points out that superplasticity is associated with a rapid drop in flow stress occurring when any additional mechanism of deformation becomes significant and does not limit himself to grain broundary shear. Qualitatively he notes that other accomodation processes such as boundary migration and local slip are necessary by-products of grain boundary shearing. Another result of his experimental work is that no recrystallization occurs, contrary to the findings of Holt and Backofen (2).

A Dislocation Model

Another unique analysis is that made by Chaudhari (8). His research on the effect of stress, temperature, and heat-treatment on the strain rate of the Al-Zn alloy have yielded yet another proposed mechanism theory for superplasticity. In a particularly thorough analysis of the Avery-Backofen and Packer-Sherby competing processes models, he notes that as both processes in each model increase monotonically with an increasing stress, the strain rate sensitivity index "m" may be expected to show only a decrease with further increase in stress. Two such functions cannot yield a region where "m" increases with increasing strain rate because the term which has a stronger dependence on stress dominates with any further increase in strain rate. The results on the Al-Zn and Sn-Bi systems have shown such an increasing "m" value with increasing strain rate (2, 7). Hence the results cannot fully be explained on a simple two competing processes model without unreasonable postulation (8).

He also notes that grain boundary shear and Nabarro-Herring creep have been proposed to account for the large superplastic elongations. Although he points out that grain boundary shear can make a contribution to the total deformation by relaxing the requirements of five independent slip systems, they cannot account for the extensive elongations without flow of the bulk material. And he also notes that the values of strain rate and its variation with grain size, stress, and temperature do not agree with predictions made from the Nabarro-Herring creep equation (8).

Instead, Chaudhari suggests a single thermally activated deformation mechanism as controlling the strain rate and proposes the expression shown below for use above 200°C. Numbers given are for the Al-Zn eutectoid alloy.

$$\sigma = \alpha \dot{\epsilon}^{a} + \beta \dot{\epsilon}^{c} \quad \text{or} \quad \sigma = B\exp\left(aQ/RT\right)\dot{\epsilon}^{a} + C\exp\left(cQ/RT\right)\dot{\epsilon}^{c} \tag{6}$$

where

$a = 0.17 - 0.20$,

$c = 0.46 - 0.50$,

B, C = functions of grain size only,

Q = activation energy,

R = gas constant,

T = absolute temperature (8).

σ = stress

$\dot{\epsilon}$ = strain rate

This mathematical model is based on the motion of mobile dislocations in an internal stress field generated by neighboring dislocations and operates in the following manner. Flow behavior is regulated by dislocation motion and at a given steady state strain rate, the dislocation density is independent of strain. A given dislocation density has an internal stress associated with it so that any effective driving force is equal to the applied stress minus the internal stress. An increase in steady state strain rate results in a subsequent increase in dislocation density and thus increases the internal stress. Chaudhari chooses the motion of jogged screw dislocations as the mechanism controlling dislocation velocity. In conclusion he notes that with his assumption of jogged screw dislocations being the rate controlling factor, his thermally activated process model qualitatively predicts an increase in strain rate sensitivity index with an increasing strain rate (8).

The preceding examination of the major studies to date in the field of superplasticity illustrates the close similarity to the field of high temperature creep. The Soviet scientists prefer to account for the superplasticity phenomenon by metastability while the American scientists have basically accounted for it in terms of strain rate sensitivity with flow stress. It would appear that results of some investigations have directly conflicted with the results of others, while some, achieving similar results, are interpreted in different ways by different researchers. The search for a structural mechanism or mechanisms goes onward; to paraphrase Backofen, there is indeed still uncertainty in theory (7).

THE EXPERIMENT

Selection of the Experimental Alloy

To date, superplasticity has been observed in at least the following alloy systems: Al-Cu (14, 21), Al-Zn (7, 8, 15, 19, 21), Al-Ni (24), Al-Si (24), Al-Fe (24), Cu-Zn (24), Cu-Ni (24, Sn-Bi (2, 20), Sn-Pb (5, 18, 20), Cr-Co (23), titanium and zirconium alloys, (17), brasses (24), magnesium alloy ZK 60 (6), two-phase Ni-Fe-Cr alloys (12), and several other iron-base alloys (24).

In choosing an alloy for this superplasticity project, a necessary criterion was finding a suitable alloy which could be readily fabricated and tested using equipment available. The Al-Zn eutectoid alloy meets this requirement by having a homogeneous, fine-grained microstructure which is obtained without the need for hot working (15). Due to the relatively low melting point of the alloy, special high temperature apparatus is not required in either fabrication or testing. Another factor aiding selection of this system as the base material in this project is the relatively large amount of previous work which has been done (7, 8, 15, 19, 21).

Figure 3 shows the basic Al-Zn equilibrium diagram. The eutectoid transformation involves decomposition of the α' face-centered cubic phase containing 78 weight percent Zn and 22 weight percent Al (59.4 and 40.6 atomic percent, respectively) to an Al-rich face-centered cubic α solid solution and a Zn-rich hexagonal close packed β solid solution. When quenched from the high temperature solid solution region, the α' phase disintegrates spontaneously, evolving heat which can raise the temperature of the freshly quenched specimen by as much as 50°C within a few minutes (7, 9, 21). During this high temperature phase breakdown, Garwood notes no intermediate structure formed and that the final result is one of α and β primary solid solutions in equilibrium concentrations (9).

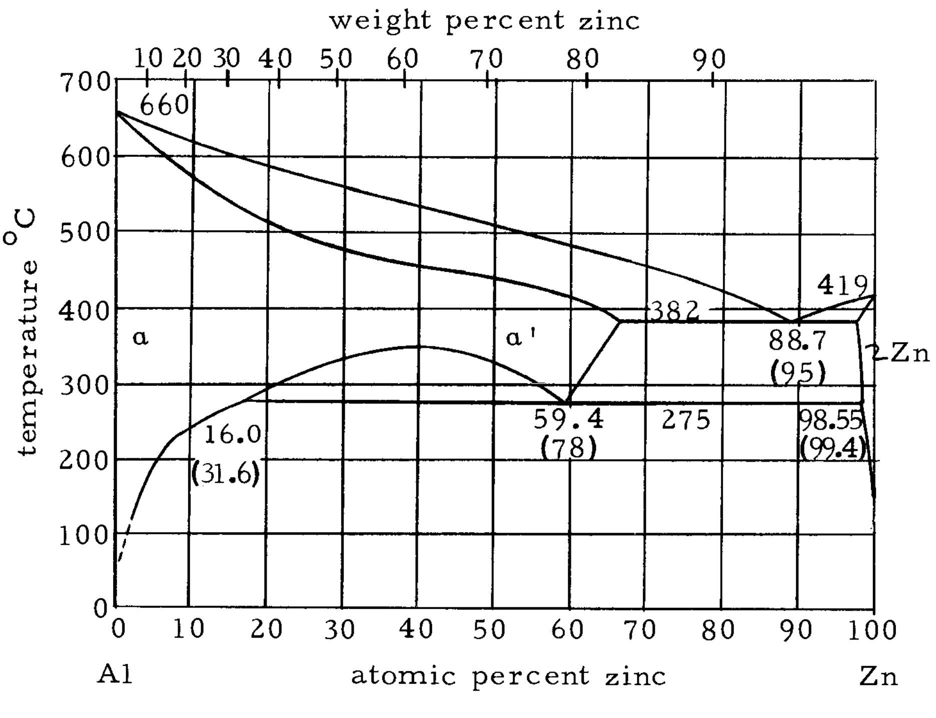


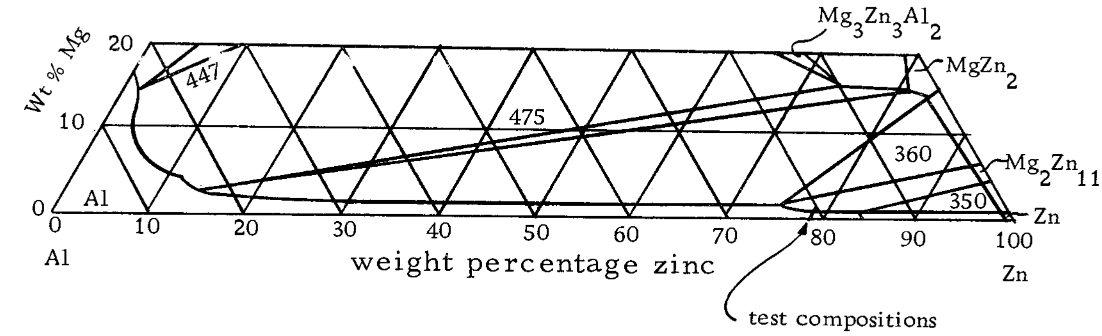
Figure 3. Aluminum-Zinc phase diagram (after Hansen, 11, p. 149).

Purpose of the Experiment

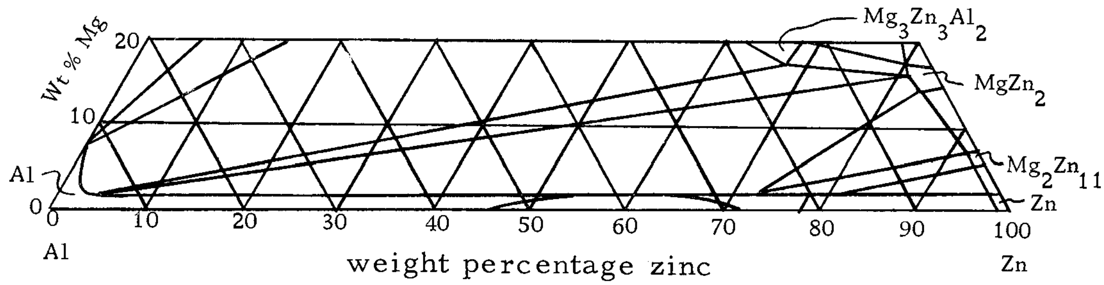
The purpose of this investigation was to note the effect of a third element to the Al-Zn eutectoid composition. Small amounts of magnesium have been used to increase the yield strength of both aluminum and zinc alloys (4). Although no present commerical alloy exists near this composition, the project was directed toward discovering the change that selected quantities of magnesium had on the superplastic behavior and flow stress of the Al-Zn eutectoid alloy. The partial ternary diagrams shown in Figure 4 show the effect of magnesium on the Al-Zn metallurgical system at the solidus, the 330°C isotherm, and the 100°C isotherm (3, 25). In reference to the spontaneous heat evolution of the breakdown of the α' phase upon quenching, Garwood mentions, in passing, that magnesium delays this reaction at room temperature (9).

Equipment and Experimental Procedure

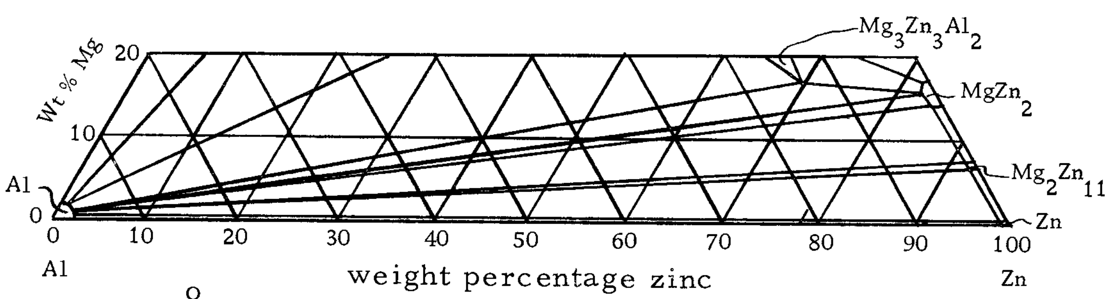
Six nominal alloy compositions of 0.00, 0.10, 0.25, 0.50, 0.75, and 1.00 weight percent magnesium, constant 78 weight percent zinc, and variable 21 to 22 weight percent aluminum were prepared from 99.99%$^{+}$ pure zinc, electrical conductor (EC) aluminum (99.45% minimum aluminum content), and primary magnesium (99.8% minimum magnesium content). Approximately 200 gram charges were placed



a. solidus surface (3, p. 1247)



b. 330°C isotherm (25, p. 392)



c. 100°C isotherm (3, p. 1247)

Figure 4. Selected partial ternary diagrams of the Al-Mg-Zn system.

in a zirconia crucible and heated in a furnace to $825^{\circ}C$ for 30 minutes before casting. The long holding time at temperature prior to casting follows from a notation in the Metals Handbook (4, vol. 1, p. 1170) that any aluminum and silicon compounds (presumably oxides) and aluminum-iron compounds float to the surface of the melt given several minutes at temperature once the aluminum has melted.

Casting was done in a cast iron mold heated to $315^{\circ}C$ prior to the pour. A pyrex stirring rod was used to mix the melt, then to skim any slag away during the pour. Finished castings measured $4\frac{1}{2} \times 3 \times \frac{3}{16}$ inches.

Each casting was then solution heat treated in air at $340^{\circ}C \pm 6^{\circ}C$ for 50 hours then quenched in brine at room temperature. Castings were removed from the brine prior to the heat evolution accompanying the decomposition of the high temperature phase (7, 9, 21). Two tensile specimens, of the dimensions shown in Figure 5, were then machined from each homogenized casting. These specimens were then annealed in air at $250 \pm 5^{\circ}C$ for one hour prior to testing. Annealing was followed by a room temperature brine quench.

Testing was performed on an Instron testing machine within a 16-inch long resistance wound Marshall tube furnace. Specimens placed in the furnace were brought to temperature in not over 15 minutes and testing began 30 minutes after initial placement. Temperature was controlled by a Marshall controller at mid-gage length to

$\pm$ 4^{o}C and the gradient throughout the middle nine inches of the furnace was not greater than 1^{o}C per inch.

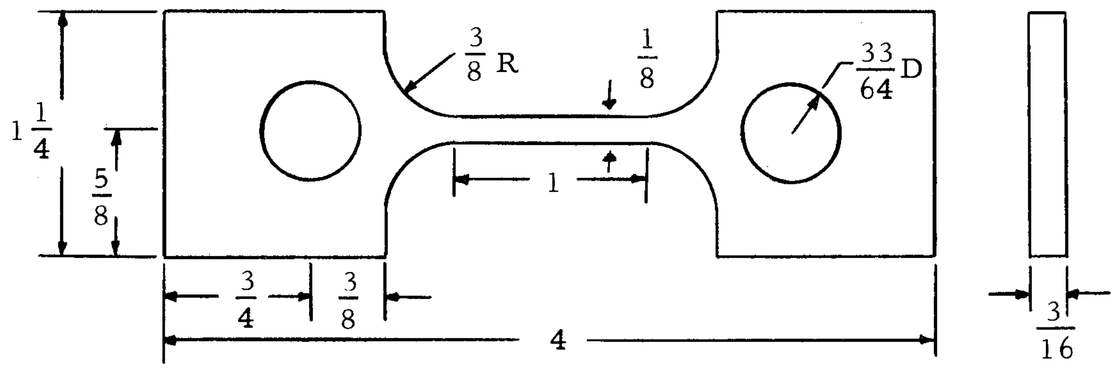


Figure 5. Specimen dimensions (inches).

Figure 6 shows a schematic view of the furnace, testing grips, and specimen in test configuration. The furnace was mounted on a vertical mounting bracket and could be adjusted to any height within the testing well. Figure 7 shows a pictorial breakdown of the testing grips and specimen. Figure 8 shows the Instron testing machine with furnace equipment in place, the Marshall controller, and test monitoring equipment.

Specimens of each alloy composition were tested in tension at a nominal 250^{o}C at crosshead velocities of 0.02 inches per minute, 0.20 inches per minute, and 2.00 inches per minute to determine the elongation and flow stress. Testing time per specimen varied with alloy composition and strain rate from but a few seconds to nearly two hours. After failure, the specimens were removed from the

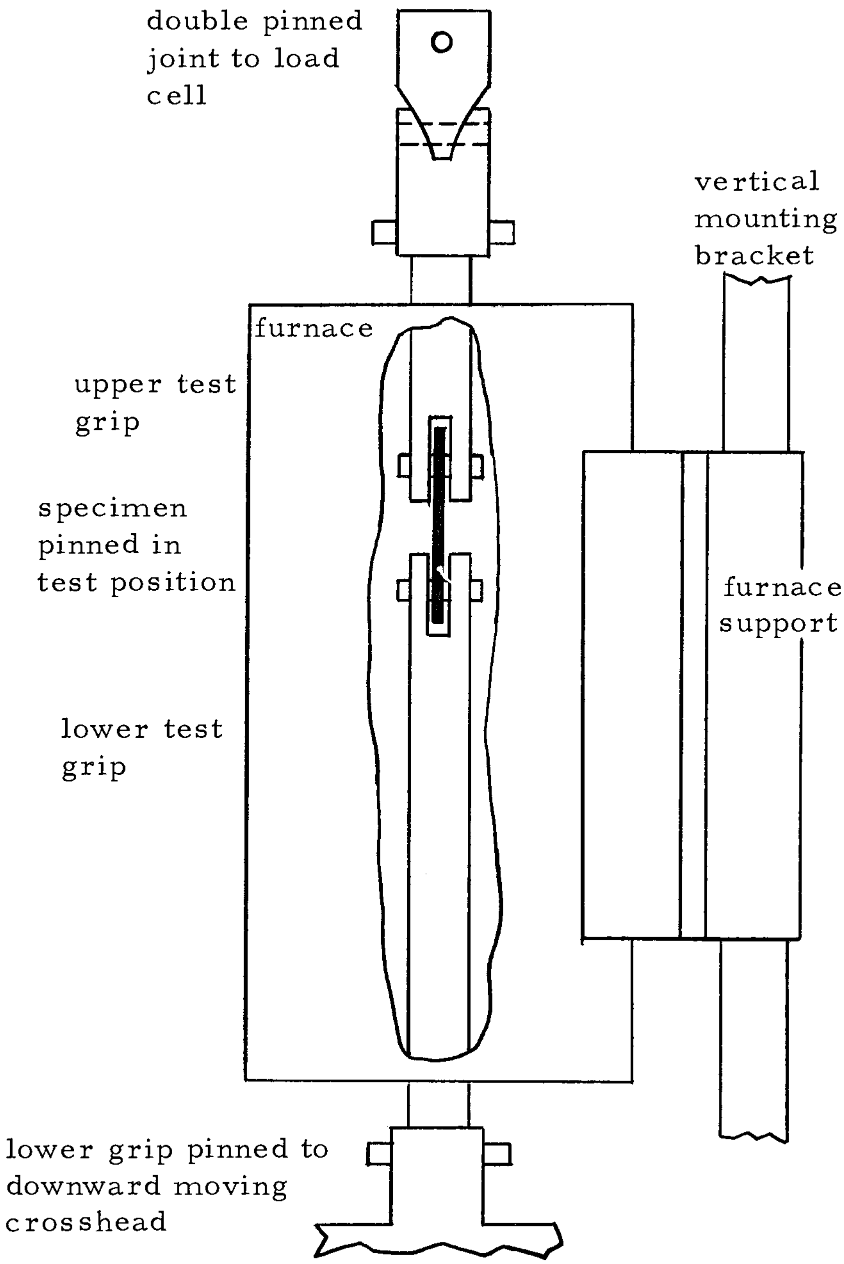


Figure 6. Schematic illustration of specimen, testing grips, and furnace in testing position on the Instron testing machine.

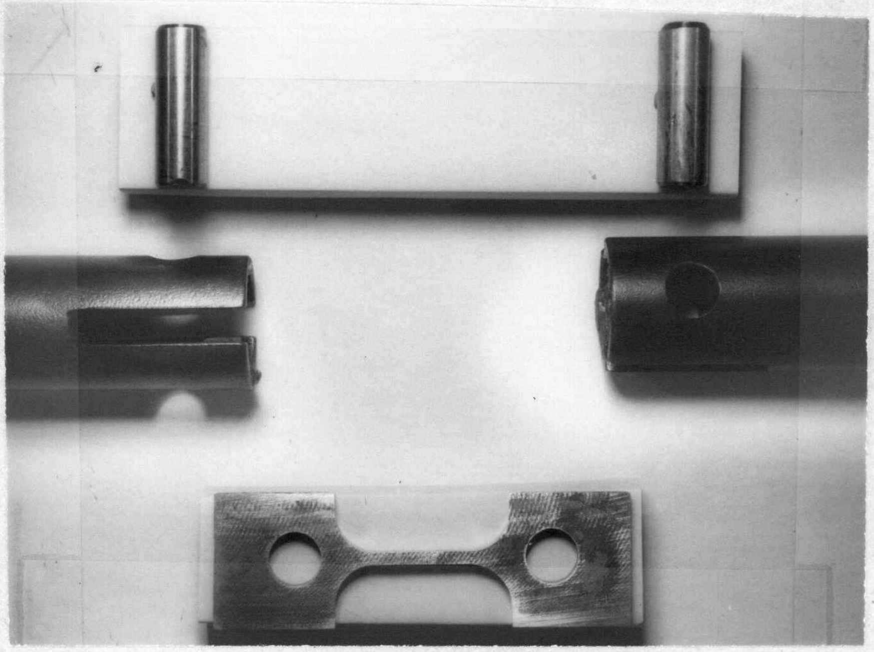


Figure 7. Breakdown of testing grips and specimen.

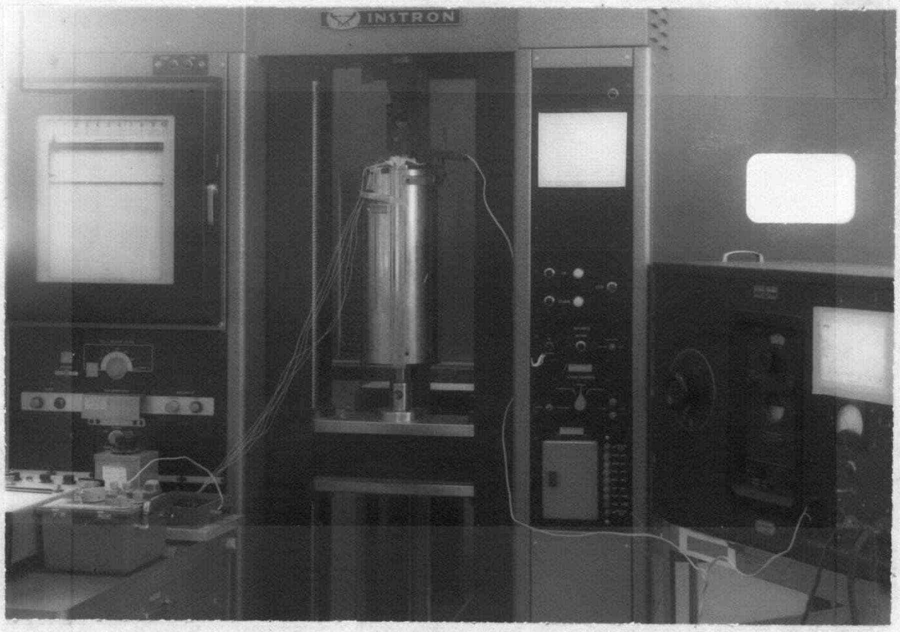


Figure 8. Experimental test setup showing the Instron testing machine with furnace equipment in place, the Marshall controller, and test monitoring equipment.

furnace, air cooled, and samples were then taken from selected specimens for metallographic study. The samples were cold mounted and metallurgically polished using standard metallographic techniques. Samples were etched using a 10% NaOH solution.

RESULTS

The experimental results are tabulated in Table 2, page 37. In this table, the heading "magnesium content" refers to the nominal magnesium composition of each specimen tested. Each specimen also contains a nominal 78 weight percent Zinc with the balance being Aluminum. "Flow stress" here signifies the engineering ultimate stress as calculated by dividing the original gage cross-sectional area into the maximum load carried. The load-elongation curve as traced by the strip chart recorder connected to the Instron testing machine followed the general form as shown below in Figure 9. A higher strain rate compressed the curve along the elongation axis while a lower strain rate generally expanded it. "Percent elongation" refers to the amount of extension the gage section developed over its original one inch length.

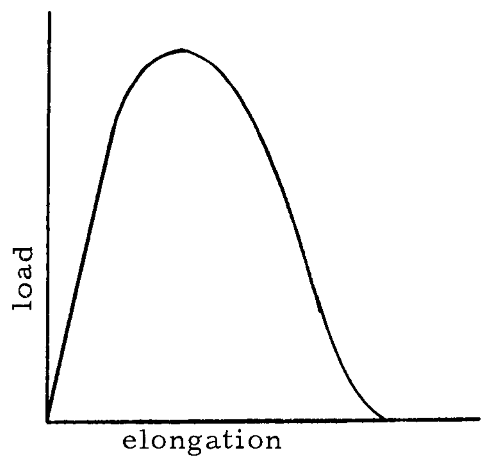


Figure 9. Representative load-elongation curve for the specimens tested in this investigation.

The last column indicates results found upon metallographic examination of samples taken from selected specimens. At least one sample was taken from each alloy composition, metallographically polished, and etched with a ten percent solution of NaOH in water. This "metallographic observation" column lists the structure of the alloy. A typical photomicrograph showing the finely-divided Al-Zn eutectoid alloy structure is shown in Figure 10. The sample was taken from the undeformed grip section of the specimen. Figure 11 shows the typical undeformed grip structure of the alloy containing one percent magnesium. A third phase has now been distributed throughout the finely divided matrix. The amount of this phase present increased with increasing magnesium content and observations made under this heading are relative value assignments to this amount.

Figure 12 shows the comparison of a set of specimens after testing at 0.02 in/min strain rate at 250°C. The specimen containing no magnesium failed after superplastic deformation while those specimens containing magnesium failed by intercrystalline fracture showing no superplastic elongation. Although quantitatively different, a comparison of the other two sets of test specimens at the different strain rates show the same trend.

Figure 13 illustrates the change in flow stress as a function of the amount of magnesium present in the Al-Zn base alloy for each strain rate tested at 250°C. Figure 14 illustrates the trend in elongation as a function of the amount of magnesium present for each strain rate.

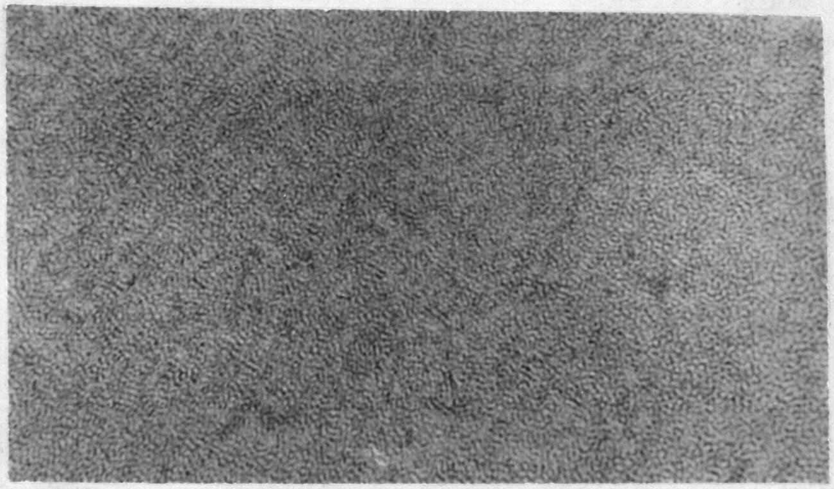


Figure 10. Photomicrograph of typical Al-Zn eutectoid microstructure. Undeformed grip section, 500x. The dark phase is the Al-rich phase.

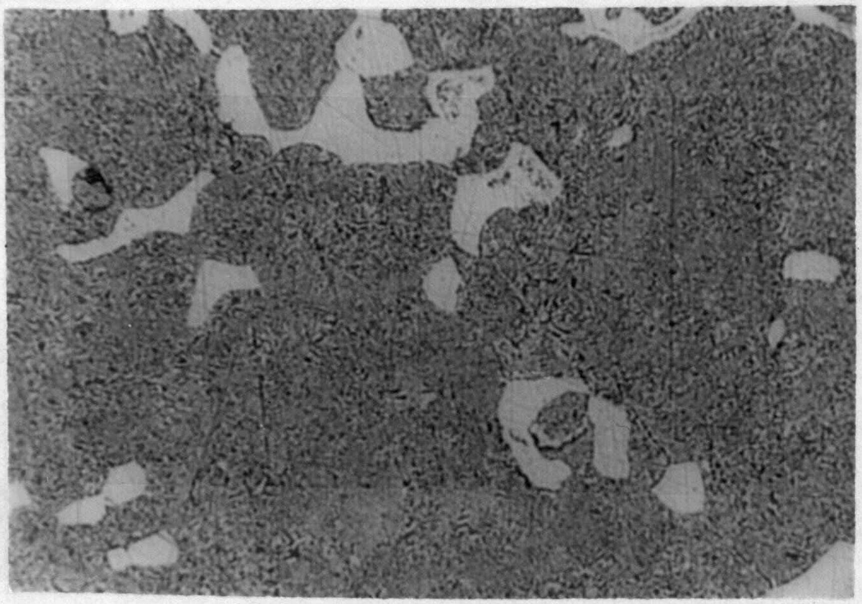


Figure 11. Photomicrograph of typical microstructure of the Al-Zn base alloy containing one percent magnesium. Undeformed grip section, 500x.

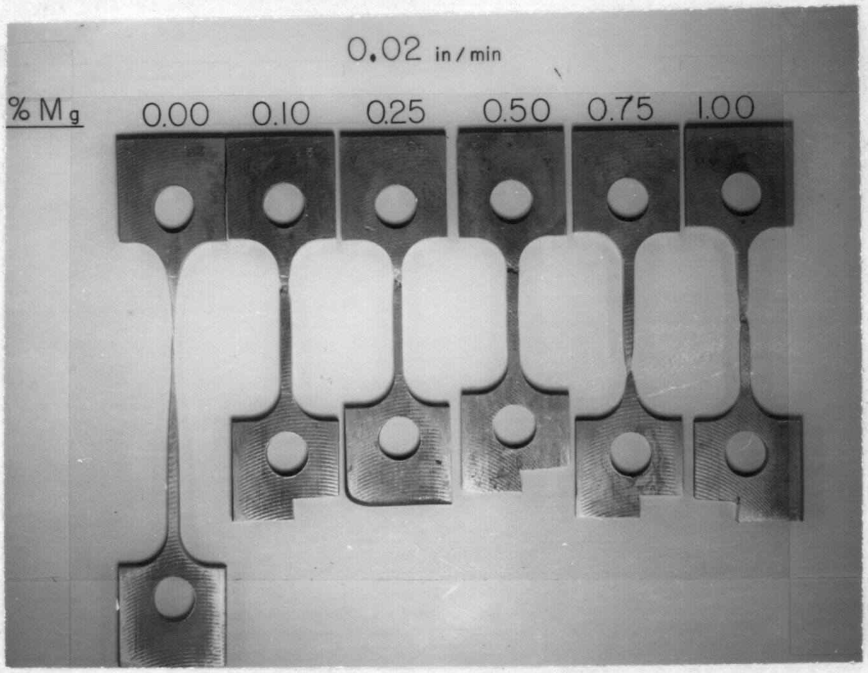


Figure 12. Comparison of fractures for the set of specimens tested at 0.02 in/min at 250°C.

Table 2. Experimental results for the alloys tested.[1]

Percent Magnesium Content	Flow Stress lb/in.^{2}	Percent Elongation	Metallographic Observation
		Strain Rate--0.02 in./min	
0.00	860	262	
0.10	3040	44	no third phase visible
0.25	5260	26	
0.50	3550	20	large amount third phase present
0.75	8150	52	
1.00	5810	54	large amount third phase present
		Strain Rate--0.20 in./min	
0.00	1960	175	
0.10	5690	26	
0.25	2100	52	no third phase visible
0.50	10620	24	
0.75	11000	25	
1.00	7250	5	
		Strain Rate--2.00 in./min	
0.00	5460	125	no third phase visible
0.10	8400	12	
0.25	11170	5	
0.50	10320	45	small amount third phase present
0.75	20100	20	large amount third phase present
1.00	20400	6	large amount third phase present

[1] Testing temperature--250°C.

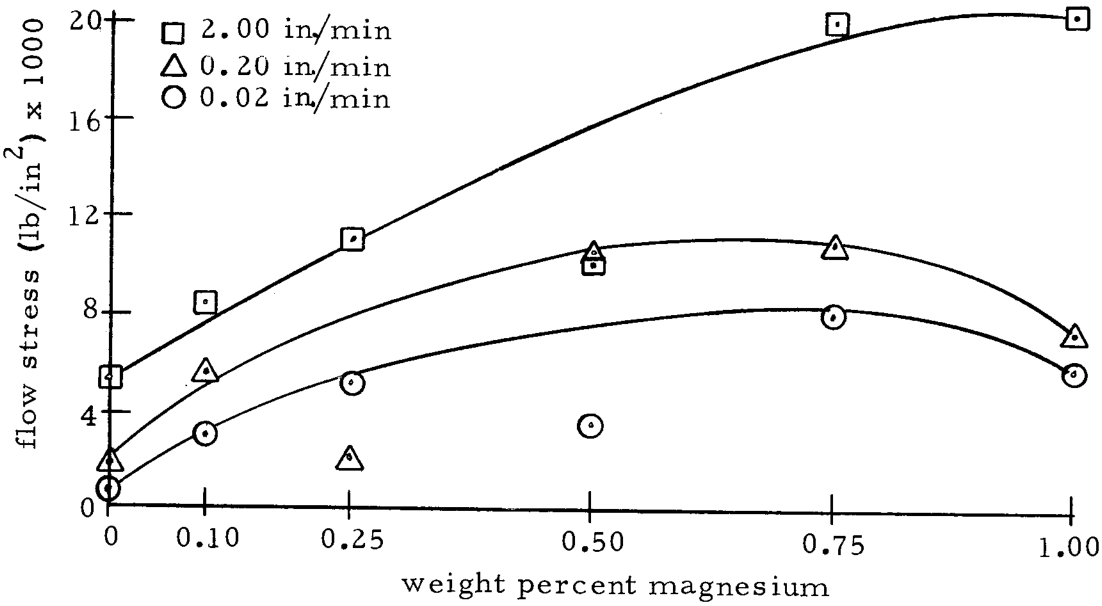


Figure 13. Change in flow stress as a function of the amount of magnesium present in the Al-Zn alloy.

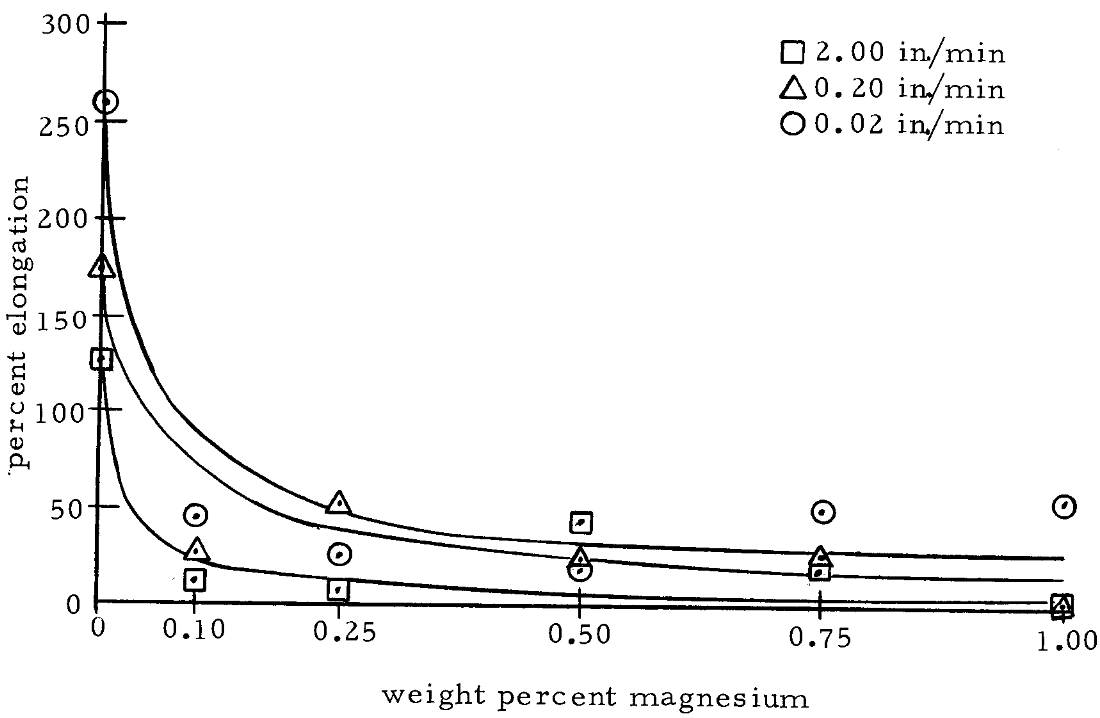


Figure 14. Change in elongation as a function of the amount of magnesium present in the Al-Zn alloy.

DISCUSSION AND INTERPRETATION OF RESULTS

Superplastic Deformation

Superplastic elongation occurred in the specimens containing no magnesium at all three strain rates tested at $250^{o}C$. Table 2 shows that the maximum amount of superplastic deformation was 262 percent and obtained at the lowest strain rate tested, 0.02 in/min. The amount of superplastic elongation then decreased as the strain rate was successively increased. At the same time the opposite trend was found for the flow stress. At the lowest strain rate tested and the greatest amount of elongation, the flow stress was at the minimum value, 860 lb/in^2. This increased as the strain rate was successively increased in the range tested.

Figure 12 shows a comparison between the specimen fractures for the lowest strain rate, 0.02 in/min. Note that in the 0.00 percent magnesium specimen that superplastic deformation has led to a large amount of neck free extension. The resulting needle point fracture has a reduction in area of nearly 100 percent and the cross section at fracture still retains the rectangular appearance of the original gage cross section. This last point makes clear that in superplastic deformation, any surface irregularities and effects are preserved upon testing. The other two superplastic specimens are similar to the one shown in Figure 12 in having the needle point fractures and nearly 100 percent reduction in area. The amount of extension is, however, dependent on strain rate and differs for each specimen.

The Effect of Magnesium on Superplastic Deformation and Flow Stress

For the strain rate range tested at 250°C, any amount of magnesium investigated destroyed any significant superplastic deformation and led to intercrystalline fracture. Table 2 shows that some elongations of about 50 percent were obtained at different strain rates, but a close look at Figure 12 shows why superplasticity is destroyed. The intercrystalline fractures of the specimens containing magnesium display little reduction in area and show no evidence of superplastic extension. Superplasticity cannot be significant deformation mode under these circumstances.

The flow stress values shown in Table 2 are plotted for each strain rate as a function of magnesium content in Figure 13 to illustrate the change in flow stress. Similar to the result found with the superplastic specimens, this figure shows, that for any given magnesium content, a higher strain rate will increase the flow stress. For any one of the three strain rates, the flow stress increases rapidly through a maximum as the magnesium content increases to about 0.75 percent, then decreases with larger magnesium contents. This indicates the maximum to which such small amounts of magnesium can harden the base alloy.

The elongation values shown in Table 2 are plotted for each strain rate as a function of magnesium content in Figure 14. Although these data are very inconsistent, it appears that once the intercrystalline fracture mode has become dominant--here at less than the 0.10 percent magnesium level--the amount of elongation decreases slightly as the magnesium content is increased. Another trend shown in Figure 14 is that the greatest elongation was found at the lowest strain rate and that successive increases in strain rate decreased the amount of elongation obtained--again similar to the results found with the superplastic alloys.

Microstructural Effects

Since the mechanical properties of a metal are closely dependent on its microstructure, it is necessary to find out why the flow stress and elongation change as magnesium is added to the base Al-Zn alloy. Figure 10 shows the typical superplastic microstructure of the Al-Zn alloy. This finely divided microstructure consists of the dark Al-rich α phase and the light Zn-rich β phase (8). Figure 11 shows a typical microstructure for the base alloy containing one percent magnesium. Now the addition of a third phase is evident which at lower magnification can be seen to be uniformly dispersed throughout the microstructure. The ternary diagrams in Figure 4 show this phase to be the intermetallic compound Mg_2Zn_{11} with a small amount of Al in solid solution. The amount of magnesium which can be added to the Al-Zn eutectoid alloy without forming this third phase is very small as can be seen in Figure 4c. Although Figure 4c is at the $100^{\circ}C$ isotherm, this solid solution line would not be expected to change much below the invariant temperature. Evidently even as little as 0.10 percent magnesium is enough to precipitate this intermetallic compound.

The amount of this third phase present depends on the magnesium content. In general it was found that the more the magnesium, the more the intermetallic compound visible. Table 2 shows the metallographic observations made noting the amount of third phase present in at least one sample at each magnesium composition tested. At the 0.10 and 0.25 percent levels of magnesium composition, the third phase was unresolved with a light microscope. The aid of an electron microscope here would have been useful in determining the extent and distribution of this third phase in these alloys.

Superplasticity is destroyed by this third phase which forms as a network blocking any significant superplastic deformation of the fine-grained matrix around it. As the amount of magnesium present increases so does the amount of the third phase present, making the network larger and thicker. The amount of compatible deformation between the matrix and the third phase then decreases and fracture is preferred over plastic deformation. Elongation then becomes less as the amount of third phase increases.

The flow stress initially rises as more magnesium is added because although superplastic deformation is restricted due to the rigid third phase, plastic deformation is possible and fracture is not preferred till greater applied loads. As the amount of third phase increases, it severely restricts any plastic deformation and fracture is then preferred at lower applied loads.

An interesting answer was found for Garwood's point that the addition of magnesium to the Al-Zn alloy delayed the heat evolution that takes place when the high temperature α' phase decomposes after quenching (9). Figure 4b shows that little, if any, Mg_2Zn_{11} is in equilibrium at the solution heat treating temperature. Upon quenching to room temperature some third phase should be present. As the α' phase decomposes into equilibrium α and β phases, heat is evolved which is used in assisting formation of the third phase. Heat can then be absorbed by this phase when formed and the overall effect is to slow the apparent heat evolution process and also to decrease the magnitude of the effect. Such a difference was observed with specimens for this investigation. Although the specimens containing no magnesium showed a marked heat evolution starting about two minutes after quenching which raised the specimen temperature high enough so it could not be hand held, many specimens containing magnesium showed only a slight warmness which lasted a somewhat longer time.

Another effect was noted during machining. The Al-Zn alloy easily welded to the milling cutter and gummed the surface of the tool. As magnesium content was increased this effect decreased considerably. Machinability improved with greater magnesium content throughout the composition range.

Comparison with Previous Work

The trends obtained with the superplastic specimens in this investigation support those previously found by Backofen (7) and Holt (15). Although grain size measurements were not taken in this study, the annealing time of one hour at 250°C was used to obtain a uniform grain size in all specimens. This annealing time has been previously used by Holt (15) and although grain size may differ slightly, the general trend is the same. Most importantly, the flow stress was found to increase with increasing strain rate and the elongation was found to decrease with increasing strain rate at 250°C.

Backofen (7) however, obtained superplastic elongations of over 500 percent during his investigation. The most superplastic specimen tested during this study showed an elongation of 262 percent (Table 2). To understand why these specimens did not achieve the larger elongations previously recorded, a closer look at Figure

12 is in order. Note that the superplastic deformation has occurred only at one end of the gage length; the whole gage length did not exhibit superplastic deformation. The same situation occurred with the other two superplastic specimens.

In checking the machining accuracy of other specimens, it was found that errors of up to 0.01 inches in width were present along the one inch long gage section. This error can mean about an eight percent difference in cross sectional area between the two ends. Since yielding occurs first at the smallest cross sectional area, this section would start to superplastically elongate first. Upon further straining, this section tends to be preserved but since it is located next to the shoulder of the grip section, elongation can only significantly occur in one direction. Hence any superplastic elongation is reduced considerably. It is to be noted that accurate machining is a necessary requirement throughout the gage length if the total gage length is to uniformly extend.

This investigation has supported previous studies performed by Backofen (7) and Holt (15) on the Al-Zn eutectoid composition alloy concerning the superplastic behavior under different strain rates at 250°C. By adding magnesium to this base alloy, it was found that the flow stress did increase markedly. Superplastic deformation was, however, virtually destroyed by the appearance of a third phase, Mg_2Zn_{11}, in the microstructure. This third phase formed a uniform dispersion throughout the finely-divided microstructure and led to intercrystalline fracture and relatively low elongations.

CONCLUSIONS

1. The Al-Zn eutectoid alloy deformed superplastically when tested in tension at 250°C. Any addition of the five levels of magnesium investigated destroyed any significant indication of superplastic deformation and led to intercrystalline fracture due to the intermetallic compound Mg_2Zn_{11} present in the microstructure.
2. Magnesium increases the flow stress of the Al-Zn alloy significantly, passing through a maximum at about 0.75 percent magnesium content. Once past the change in fracture mode from superplastic to intercrystalline, the trend in elongation is for a slight decrease with increasing magnesium content.
3. Due to the network of Mg_2Zn_{11} present in the Al-Zn alloys containing magnesium, fabrication of machine parts cannot be achieved by superplastic deformation at 250°C.

RECOMMENDATIONS FOR FUTURE RESEARCH

1. The temperature distribution throughout the testing furnace should be as uniform as possible. This can be better accomplished by improving the controlling devices that distribute the current to different sections of the furnace, taking a longer time to bring the specimen to test temperature, or by allowing a longer holding time at temperature prior to testing to allow the temperature distribution to even out.
2. Specimens should be more accurately machined in future investigations. Attention should be given to making the entire gage section as nearly uniform in cross section as possible.
3. Since magnesium significantly lowers the solidus boundaries of the Al-Zn eutectoid system, careful attention should be paid to solution heat treating temperatures and to a lesser extent, annealing temperatures. It is all too easy to exceed the solidus temperatures and partially melt the alloy.
4. In an effort to achieve the most homogeneous specimens possible, a more effective casting method is desirable. Casting under inert gas using an induction furnace with a bottom pouring device would be suggested to eliminate the awkward handling of a crucible containing the molten metal, the mechanical stirring, and oxygen contamination of the alloy.

5. Heat treating, annealing, and testing could also be performed under inert gas atmosphere to completely eliminate oxygen contamination from the investigation.

BIBLIOGRAPHY

1. Alden, T.H. Discussion of "Superplasticity in the Al-Cu eutectic alloy," by David L. Holt and Walter A. Backofen. Transactions of the American Society for Metals 60:274-275. 1967.

2. Alden, T.H. The origin of superplasticity in the Sn-5% Bi alloy. Acta Metallurgica 15:469-480. 1967.

3. American Society for Metals. Metals handbook. 7th ed. Cleveland, Ohio, American Society for Metals, 1948. 1332 p.

4. American Society for Metals. Metals handbook, 8th ed. Vol. 1. Metals Park, Ohio, American Society for Metals, 1961. 1300 p.

5. Avery, D.H. and W.A. Backofen. A structural basis for superplasticity. Transactions of the American Society for Metals 58:551-562. 1965.

6. Azzarto, F.J. and W.A. Backofen. Superplasticity in pellet- and ingot-extrusions of the magnesium alloy ZK 60. July 1966. 4 numb. leaves. (Massachusetts Institute of Technology. Metals Processing Laboratory. Deformation Processing of Anisotropic Metals Final Report on Naval Air Systems Command Contract NOw 66-0068-d.)

7. Backofen, W.A., I.R. Turner and D.H. Avery. Superplasticity in an Al-Zn alloy. Transactions of the American Society for Metals 57:980-989. 1964.

8. Chaudhari, P. Deformation behavior of superplastic Zn-Al alloy. Yorktown Heights, IBM Watson Research Center, New York. [1967] 20 numb. leaves.

9. Garwood, R.D. and A.D. Hopkins. The kinetics of the eutectoid transformation in zinc-aluminium alloys. Journal of the Institute of Metals 81:407-416. 1952-1953.

10. Guy, A.G. and J.E. Pavlick. Acceleration of deformation by concurrent phase change. Transactions of the Metallurgical Society of AIME 221:802-807. 1961.

11. Hansen, Max and K. Andreko. Constitution of binary alloys. 2d ed. New York, McGraw-Hill, 1958. 1305 p.

12. Hayden, H. W. *et al.* Superplasticity in the Ni-Fe-Cr system. Transactions of the American Society for Metals 60:3-14. 1967.

13. Herring, Conyers. Diffusional viscosity of a polycrystalline solid. Journal of Applied Physics 21:437-445. 1950.

14. Holt, David L. and Walter A. Backofen. Superplasticity in the Al-Cu eutectic alloy. Transactions of the American Society for Metals 59:755-768. 1966.

15. Holt, D. L. The relation between superplasticity and grain-boundary shear in the Al-Zn eutectoid alloy. General Motors Corporation Defense Research Laboratory, Santa Barbara, California. [1966] 8 numb. leaves.

16. Jones, R.B. and R.H. Johnson. Discussion of "A structural basis for superplasticity," by D.H. Avery and W.A. Backofen. Transactions of the American Society for Metals 59:356-359. 1966.

17. Lee, D. and W.A. Backofen. Superplasticity in some Titanium and Zirconium alloys. July 1966. 17 numb. leaves. (Massachusetts Institute of Technology. Metals Processing Laboratory. Deformation Processing of Anisotropic Metals Final Report on Naval Air Systems Command Contract NOw 66-0068-d.)

18. Martin, P.J. and W.A. Backofen. Superplasticity in electroplated composites of lead and tin. July 1966. 14 numb. leaves. (Massachusetts Institute of Technology. Metals Processing Laboratory. Deformation Processing of Anisotropic Metals Final Report on Naval Air Systems Command Contract NOw 66-0068-d.)

19. Packer, Charles M. and Oleg D. Sherby. An interpretation of the superplasticity phenomenon in two-phase alloys. Transactions of the American Society for Metals 60:21-28. 1967.

20. Pearson, C.E. The viscous properties of extruded eutectic alloys of Lead-Tin and Bismuth-Tin. Journal of the Institute of Metals 54:11-124. 1934.

21. Presnyakov, A.A. and V.V. Chervyakova. On the subject of "super-ductility" of alloys. Fizika Metallov i Metallovedenie 8(1):114-122. 1959. (Translated in Physics of Metals and Metallography 8(1):96-102. 1959.)

22. Starikova, G.V. and A.A. Presnyakov. Metastability of cast eutectics assessed in connexion with the "super-ductility" effect. Fizika Metallov i Metallovedenie 13(5):769-771. 1962. (Translated in Physics of Metals and Metallography 13(5):125-127. 1962.)

23. Stephens, Joseph R. and William D. Klopp. Superplasticity and low-temperature ductility in a Cr-30 atomic percent Co alloy. Transactions of the Metallurgical Society of AIME 236: 1637-1639. 1966.

24. Underwood, Ervin E. A review of superplasticity and related phenomena. Journal of Metals 14:914-919. 1962.

25. Van Horn, Kent R. (ed.). Aluminum. Vol. 1. Metals Park, Ohio, American Society for Metals, 1967. 435 p.