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## Materials research at NPS

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# MATERIALS RESEARCH AT NPS

By Terry R. McNelley and Stephen J. Hales  
Department of Mechanical Engineering

## Introduction

Metals are useful partly because they can bend permanently before they break, i.e. they can deform plastically. Metal plasticity is usually evaluated by measurement of the percentage elongation during tensile testing and the result is referred to as the ductility of the material. Ductility of structural metals is typically 10-50% at ambient temperature and perhaps attains 100% at elevated temperatures.

Some metallic alloys are superplastic under certain processing and deformation conditions. Superplasticity is the ability of such alloys to exhibit very large, neck-free tensile elongation; an extreme example of such deformability, 5550% elongation, was reported by Higashi and co-workers.<sup>1</sup> More commonly, ductility in tension above 200% elongation to fracture is considered superplastic,<sup>2</sup> and many superplastic alloys exhibit maximum elongations of 500 to 1000%. Such extensive ductility is attained only in a narrow range of temperatures and deformation rates and then only with a suitably processed material. An example is illustrated in Figure 1, which shows a tension test specimen of an Aluminum-Magnesium alloy deformed ~600% to fracture; the test was done at a temperature of 300°C and strain rate equal to  $5 \times 10^{-3} \text{ sec}^{-1}$ .

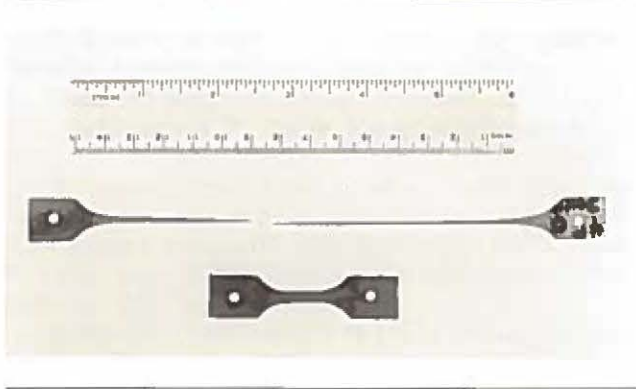
This phenomenon is much more than a laboratory curiosity. Several Titanium-based alloys are processed superplastically to form complicated components such as

landing gear doors for the Air Force's B-1 aircraft, and superplastic Nickel-base alloys facilitate the manufacture of numerous complex gas turbine engine components. A wide variety of structural forms may be fabricated from superplastic Al alloys. A recent example is the oil cooler inlet duct, indicated by the arrow in the photograph in Figure 2, for the Navy's P-3 anti-submarine aircraft.<sup>3</sup> Other commercial components range from door panels for automobiles to equipment enclosures for scientific and medical instrumentation and even to architectural panels.

The foregoing examples emphasize that superplasticity allows the designer to fabricate complex shapes from a single piece of metal. This in turn will usually lead to weight savings and improvements in system performance and maintainability. The superplastically formed inlet duct for the P-3 replaced an assembly of some 30 individual parts and fasteners, with substantial weight savings. Also, fatigue and corrosion often begin at fastener holes or other sites where components are joined, and thus superplastic forming of components will help extend service lives of Naval platforms and improve their maintainability. Some additional examples of Naval application of this technology include parachute boxes, backrests and equipment packs for aircraft ejection seats; helicopter engine air intakes; and stores carriers for attack aircraft. Not all applications are for aircraft: two superplastic aluminum parts have been used to replace a fifteen-part welded assembly for a Naval compass stabilizer unit for use aboard ship.

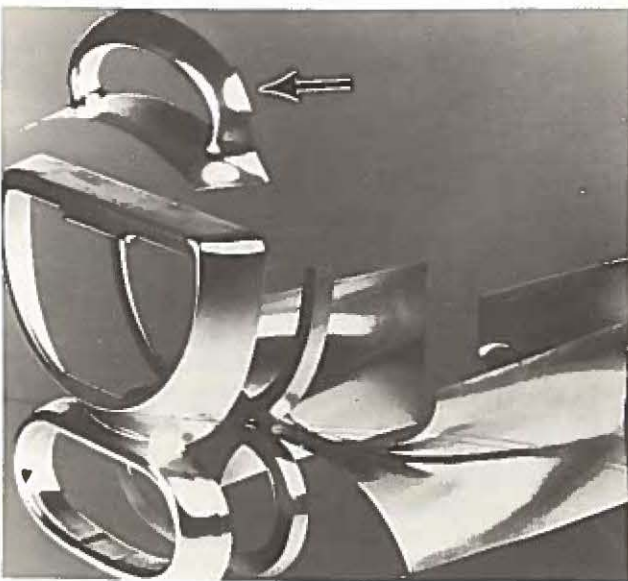
**Figure 1**

Undeformed (top) and superplastically deformed (bottom) test samples of an Al-10 wt.% Mg-0.5 wt.% Mn alloy. The material was thermomechanically processed by warm rolling at 300°C and then tested in tension at 300°C using a strain rate of  $5 \times 10^{-3} \text{ S}^{-1}$ . Elongation attained was ~ 600%.



**Figure 2**

Oil cooler inlet duct, indicated by the arrow, is superplastically formed from an Aluminum alloy. This one-piece component replaced an assembly of numerous parts and fasteners and is fabricated for the Lockheed P-3 Orion aircraft (photo courtesy of Lockheed Corp.).



Under what conditions may a metal behave superplastically? From the perspective of applied mechanics, all superplastic metals have one important feature in common, namely a highly strain-rate sensitive flow stress.<sup>(2,4-6)</sup> This is often described by the relation

$$\sigma = K\dot{\epsilon}^m$$

where  $\sigma$  is the flow stress of the metal at a strain rate  $\dot{\epsilon}$ ,  $K$  is a material constant and  $m$  is the strain rate sensitivity coefficient. It is observed experimentally<sup>7</sup> and readily demonstrated analytically<sup>8</sup> that elongation to failure increases as  $m$  increases. When  $m = 1$ , behavior would be Newtonian viscous and materials such as hot glass, which exhibit a linear stress vs. strain-rate relationship, are ideally superplastic. Superplastic metals generally have  $m$  values nearer to 0.5.

The strong stress dependence of the strain rate, as described by the coefficient  $m$ , arises from deformation mechanisms that depend in turn upon microstructural considerations.<sup>2,4-6</sup> These microstructural prerequisites are:

- A fine grain size. Typical superplastic metals exhibit grain sizes below  $10 \mu\text{m}$ ; models for behavior generally presume that the superplastic strain rate  $\dot{\epsilon} \propto d^{-p}$  where  $d$  is the grain size and  $p = 2$  or  $3$ . Thus, finer grain size will enhance the superplastic response.
- A second phase. As superplastic forming is generally done at warm temperatures, where pure metals and single phase alloys would experience grain growth, a second phase is generally necessary to retard grain growth. This phase must deform with the matrix and generally must be uniformly distributed.
- Mobile high-angle grain boundaries and equiaxed grains. The predominant mode of deformation during superplastic flow is grain boundary sliding; this requires high-angle (disordered) boundaries and also that the boundaries be mobile to relieve stress concentrations that would otherwise result from grain boundary sliding. Equiaxed grains will accommodate better the grain rotation that accompanies grain boundary sliding.

## Superplastic Al Alloys

The discovery of superplasticity is usually attributed to Pearson in 1934.<sup>9</sup> Sherby and Wadsworth<sup>2</sup> have pointed out, however, that Bengough<sup>10</sup> first reported "enormous elongations" attained in specially processed brass as early as 1912. Even so, the phenomenon remained in the laboratory until Underwood's 1962 review of Soviet work in the field; then, superplasticity was thought to be attainable only in a few alloys of eutectic or eutectoid composition.<sup>6</sup> Indeed, the first Al-base alloy for which superplastic ductilities were reported was Al-33 wt.% Cu.<sup>11</sup>

This is the eutectic composition at the Aluminum end of the Al-Cu phase diagram, and as late as 1970, the opinion continued to be expressed that no useful superplastic Al-base alloys were likely, as the various possible eutectic systems, like Al-Cu, involved phases that would render the alloys too brittle at ambient temperature.<sup>5</sup> Some time thereafter however, research in Britain resulted in the commercialization of Supral alloys; these are based on the Al-Cu system, but have Cu content of only 5-6 wt. %, well below the eutectic.<sup>12</sup> In order to maintain the fine structure necessary for superplasticity, Zr is added to form fine Al<sub>3</sub>Zr particles; these pin boundaries of recrystallized grains and prevent them from growing during superplastic forming.

Subsequent work using similar approaches has led to superplastic versions of existing alloys such as the high-strength Al alloy designated 7475 and also to superplastic Al-Li alloys.<sup>3,13,14</sup> There are, however, some significant problems with all of the Al-base superplastic alloys. The limited volume fractions of second phase attainable with additions of elements such as Zr results in rather coarser grain sizes, 10-20 μm, than attainable in other superplastic alloy systems. This, in turn, results in relatively higher temperatures, ~500°C, i.e. 80% of aluminum's melting temperature, being required for superplastic forming, as well as lower strain rates being required to sustain high ductility. Low strain rates present a particular problem in that production rates in superplastic forming generally are low and this tends to offset reduced tooling costs associated with the process. Finally, cavitation resulting from tensile separation of grain boundaries is an acknowledged<sup>16,17</sup> problem in superplastic aluminum alloys, and this results in degraded ambient temperature properties of components formed by the process. Current research in many laboratories is addressing these problems and solutions are being developed. For example, cavitation may be limited by use of back pressure during forming.<sup>16</sup>

It has been noted that the processing used to induce elevated temperature superplasticity results in highly refined grain structures, and it is generally perceived that such structural refinement may have application beyond superplastic forming. For instance, grain-refined Al alloys have stress-corrosion resistance superior to conventional alloys. Stress-corrosion cracking is a severe limiting factor in use of high-strength Al alloys in marine environments; indeed, the conventional 7475 Al alloy is heat-treated in such a way as to give up considerable strength to gain resistance to stress corrosion, and application of processing studies such as that at NPS may facilitate use of many alloys at higher strength levels than is now possible.

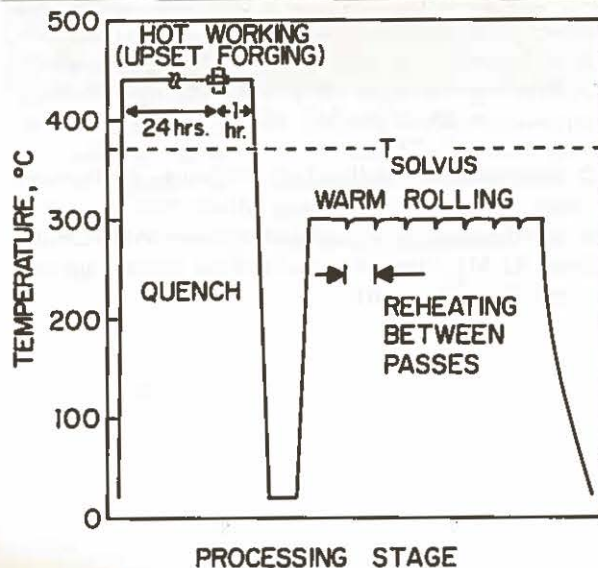
## Research at NPS Thermomechanical Processing

The program at NPS has focused upon the Al-Mg system and has addressed various issues in addition to superplasticity. The Al-Mg system is the basis for several moderate-strength alloys, many of which are used in marine applications. The first research at NPS considered various methods of processing to refine microstructures and strengthen these alloys;<sup>18</sup> the thermomechanical process (TMP) most generally utilized in the research is illustrated in Figure 3 and has as its central feature mechanical working at warm temperature by rolling. By warm is meant a temperature below the Mg-solvus, but above 200°C. As such, the processing is most applicable to relatively high Mg-content alloys, i.e. 6-10 wt. % Mg.

Because the rolling takes place below the Mg-solvus temperature, the intermetallic β phase (Al<sub>3</sub>Mg<sub>5</sub>) will precipitate during the rolling in conjunction with introduction of dislocations. The microstructure of such a warm rolled Al-10 wt. % Mg - 0.1 wt. % Zr alloy is shown in Figure 4, a micrograph obtained by transmission electron microscopy. Owing to the extreme distortion experienced by the material during the rolling process, it is hard to differentiate the features. The high dislocation density obscures the grain structure and the β phase is only occasionally discernable as highly faulted precipitates ~ 0.5 μm in size.

**Figure 3**

A schematic of the thermomechanical processing method showing initial solution treating and hot working as well as the warm working. Strains attained during the warm rolling are > 2.0, i.e. more than 80% reduction.



## Superplasticity

The structure shown in Figure 4 does not satisfy the microstructural prerequisites outlined previously, and yet as shown in Figure 5, the ductility exceeds 500% during the tension testing at 300°C at a strain rate of about  $5 \times 10^{-3} \text{ S}^{-1}$ . More recent research<sup>19,21</sup> has examined the behavior of such alloys and concluded that these warm-rolled alloys undergo a type of recrystallization often termed continuous.<sup>22</sup> In these Al-Mg alloys, this is seen as a more or less gradual conversion of the heavily dislocated structure of Figure 4 into one consisting of fine, recrystallized grains 1–5  $\mu\text{m}$  in size. Indeed, during deformation, it was observed<sup>21</sup> that such recrystallized grains coarsened gradually during superplastic deformation. The effect of this is also seen in the lower plot of Figure 5. There, the stress for a given strain rate is seen to increase with strain during a mechanical test. As the superplastic strain rate is directly related to the stress and inversely related to the grain size, grain growth during deformation at constant strain rate will be accompanied by such apparent hardening.

The alloy characterized in Figures 4 and 5 possesses an ambient temperature (25°C) tensile strength of  $\sim 70$  Ksi ( $\sim 500$  MPa), with 15 percent elongation to fracture during ambient temperature testing. Thus, this processing results in a relatively high-strength alloy of good ductility which is also superplastic at lower temperature, 300°C, when compared to other superplastic Al alloys. Further, the strain rate at peak ductility,  $5 \times 10^{-3} \text{ S}^{-1}$ , is higher by a factor of 25 when compared to that reported for the 7475 Al alloy,<sup>13,14</sup> and this would be of considerable importance in enhancing production rates when using superplastic forming. Especially noteworthy is the observation<sup>20,21</sup> that these warm-rolled alloys do not cavitate during superplastic flow at warm temperature. It is believed that this is the result of the highly refined grain structure retained during the superplastic deformation, and the relatively low temperature (300°C) at which the deformation is occurring. It was noted<sup>19,20</sup> that these alloys will undergo normal rapid recrystallization upon heating to a temperature above the Mg solvus and that resultant grain sizes are 10–20  $\mu\text{m}$ . At such temperatures, e.g. 450°C, these now-recrystallized alloys cavitate as observed with other superplastic Al-base alloys,<sup>16,17</sup> thus suggesting that the absence of cavitation at lower temperatures with these Al-Mg alloys is a result of the processing and not simply the alloy itself.

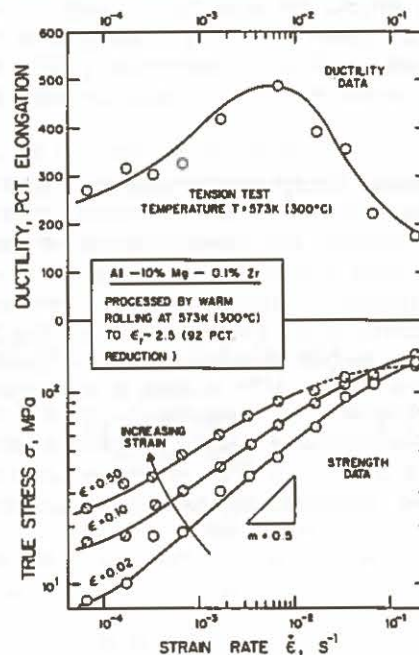
**Figure 4**

A transmission electron micrograph illustrating the high dislocation density in the warm-rolled condition of an Al-10 wt.% Mg-0.1 wt.% Zr alloy. A faulted  $\beta$  ( $\text{Al}_3\text{Mg}_5$ ) precipitate is indicated by the arrow at the right-center of the micrograph.



**Figure 5**

Tension test data for the material of Figure 4 showing superplastic ductility of  $\sim 500\%$  at strain rates near  $10^{-2} \text{ S}^{-1}$  (top plot). Stress versus strain-rate data is at the bottom where it is seen that the material hardens during deformation with a decrease in the coefficient  $m$ .



## Microstructural Evolution

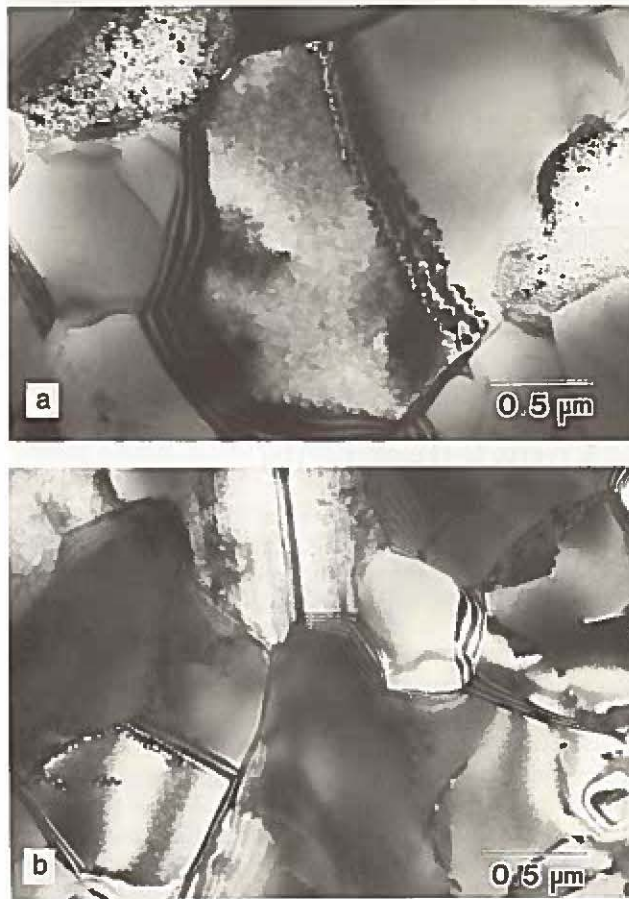
Superplasticity, and associated difficulties such as cavitation, are very sensitive to microstructure and to changes in microstructure during processing and deformation. More extensive application of superplastic forming to Al alloys will require better understanding of microstructural evolution and the potential problems associated with it. The research program at NPS is currently focusing on study of microstructural evolution during processing and superplastic deformation of these higher Mg, Al-Mg alloys. In addition, the program is examining the effects of lithium additions to these alloys and also the possibility of adapting the processing methodology to other Al-base alloys.

For example, Figure 6a illustrates the effect of static annealing at 300°C of the rolled structure of Figure 4. The structure following superplastic deformation at this same temperature (300°C) and at a strain rate near that of peak ductility,  $2 \times 10^{-3} \text{ S}^{-1}$ , is shown in Figure 6b. After annealing only, the as-rolled structure has been replaced by a recrystallized microstructure of grain size 1-5  $\mu\text{m}$ . The grains are essentially dislocation free, although some grains contain subgrain boundaries in which individual dislocations are discernible. Such a subboundary is seen in Figure 6a, running roughly vertically and just to the right of center of the micrograph. The grain boundaries are free of precipitates except for the  $\beta$  which is found to form preferentially at triple points. Here, the  $\beta$  is seen as the mottled-appearing regions at the upper left and also on the right side of the micrograph. In comparing the  $\beta$  to that present in the as-rolled condition, coarsening to a size of 1-2  $\mu\text{m}$  is evident, although no faulting is seen within the particles.

After superplastic deformation (Figure 6b) under conditions where the sample experienced the same time at temperatures as that in Figure 6a, the grain size is still 1-5  $\mu\text{m}$ , suggesting time at temperature is the primary factor controlling coarsening. The grains in the deformed material are somewhat less equiaxed than those in material experiencing only annealing and show some local distortion near the  $\beta$  precipitates. The  $\beta$  is slightly finer,  $\sim 1 \mu\text{m}$  in size, and is again substantially faulted. Some dislocations within grains are also apparent, and these may have been generated in conjunction with accommodation processes during grain boundary sliding. The highly refined structures documented here should have application in other areas as well as superplasticity; these structures should have much improved resistance to stress-corrosion cracking in comparison to structures normally found in high-Mg alloys. Existing alloys such as Al 5083 alloy, widely used in ship superstructures and related marine application, may benefit from such grain refining processing methods.

**Figure 6**

Transmission electron micrographs of an Al-10 wt.% Mg-0.1 wt.% Zr alloy (a) warm rolled and then recrystallized by static annealing at 300°C. Note the subgrain boundary dividing the center grain. In (b) is shown this same material superplastically deformed to a strain of  $\sim 200\%$  at a strain rate of  $2 \times 10^{-3} \text{ S}^{-1}$  and temperature 300°C.



## Future Directions

The fineness of microstructure demonstrated in Figure 6 is noteworthy. Other wrought superplastic Al-base alloys will exhibit grain structures at least four or five times coarser than those of this research. The fine structures result in relatively low deformation temperatures (300°C here versus 450-500°C for other Al-base alloys) and high strain rates for optimum ductility. The low deformation temperatures are thought especially important in the avoidance of the cavitation problem so often noted in such alloys. This all suggests potentially excellent ambient temperature properties as noted above, and current research is evaluating this area.

Finally, the mechanisms involved in the recrystallization process noted here are not well understood. Improved understanding of the modes of recrystallization in Al is a current topic of research at NPS. This understanding would have relevance to the development of refined grain structures in Al alloys and potential widespread application in areas demanding high performance materials for use in Naval aircraft and ships.

## Acknowledgements

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## Biographies

Dr. Terry R. McNelley is Professor of Materials, Department of Mechanical Engineering, Naval Postgraduate School. He teaches in the areas of materials science, welding, and physical and mechanical metallurgy. His research interests are in processing and microstructural control in high-carbon steels and in Aluminum alloys as well as in superplasticity.

Dr. Stephen J. Hales is Adjunct Research Professor of Materials, Department of Mechanical Engineering, Naval Postgraduate School. He has conducted research in powder metallurgy Ni-base alloys including dynamic compaction of superalloy materials and his current interests are in thermomechanical processing, recrystallization and superplasticity in Al alloys.

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Students at work stations



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An aerial photograph of a tropical atoll, showing several circular islands with white sandy beaches and turquoise water. The islands are arranged in a roughly circular pattern, with a central lagoon. The water is a deep blue, and the sky is a lighter blue. The overall scene is a beautiful, serene view of a tropical island.

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### About the Cover

Growth and motion of vortices generated by a plate at an angle of attack of 60 degrees. The picture is taken in a recirculating water table and the vortices are visualized by means of aluminum dust. The alternate shedding of vortices takes place practically about all bluff bodies (cylinders, cables, missiles, etc.) and gives rise to large drag, oscillating lift force, and hydro- or aero-elastic oscillations. The flow field may be simulated numerically through the use of the fundamental equations of motion. The visualization of flow helps to our physical understanding of the phenomenon and provides data for comparison with those obtained in numerical experiments. (See article beginning on page 3.)

Photograph is the courtesy of Professor Turgut Sarpkaya (NPS).

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