Superplasticity in Very Fine Grained Al-Based Alloys Produced by Mechanical Alloying

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Very high strain rate (also known as positive exponent) superplasticity in three mechanically alloyed IN9021, IN9052 and IN905XL aluminum alloys has been characterized over a wide range of strain rates between $10^{-3}$ to 300 s$^{-1}$ in air at temperatures from 698 to 873 K. The temperature dependence of flow stress, elongation and strain rate sensitivity exponent (m value) reveals that optimum superplasticity might occur at temperatures close to or above the melting point of each alloy. The presence of a liquid phase, resulting from the low melting point regions, as a result of solute segregation by mechanical alloying, is responsible for the observed positive exponent superplasticity. It is proposed that superplastic flow at high strain rates is controlled by a grain boundary sliding mechanism accommodated with relaxing the stress concentration by isolated liquid phases at grain boundaries. Mechanically alloyed processing is a powerful method to produce the desired microstructures with not only fine grain size but also optimizing segregation in solute along boundaries, required for positive exponent superplasticity in aluminum alloys.

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I. Introduction

Recently, a number of aluminum alloys produced by mechanical alloying(1)-(8) were reported to behave in a superplastic manner at very high strain rates (i.e., over 1 s$^{-1}$, denoted as positive exponent superplasticity(9)). For example, extremely large superplastic elongations (~1250%) were recorded in mechanically alloyed (MA) IN9021 deformed at very high strain rates from 10 to 100 s$^{-1}$ and 823 K(8)(9). A similar phenomenon was also observed in ultra-fine grained MA IN905XL(9)(10)(7) and IN9052(9)(8), in which large elongations were found at very high strain rates of from 10 to 20 s$^{-1}$. This strain rate range is several orders of magnitude faster than that for conventional superplastic aluminum alloys, such as the SUPRAL and 7475 alloys. Microstructurally, MA aluminum alloys contain a large amount of very fine oxide and carbide particles introduced through mechanical alloying processes. As a result of grain-boundary pinning by these fine particles, a fine grain or subgrain structure is often obtained.

In this paper, the tensile behaviors of three MA aluminum alloys, i.e., Al-Cu-Mg (IN9021), Al-Mg (IN9052) and Al-Mg-Li (IN905XL), with ultra-fine grained structures (typically, grain size is about 0.5 μm) are characterized over a wide range of strain rates and temperatures. This is to explore the possible mechanisms of high strain rate superplasticity in MA aluminum alloys. On the basis of the new experimental results obtained from the temperature dependence of the flow stress, the elongation and the strain rate sensitivity exponent, it is proposed basically that the dominant deformation mechanism for all MA IN9021, IN9052 and IN905XL aluminum alloys is grain-boundary sliding, and the presence of liquid phases is suggested to be responsible for the observed positive exponent superplasticity in MA aluminum alloys.

II. Experimental Procedures

The three commercial MA aluminum alloys (IN9021, IN9052 and IN905XL) used in the present study were initially obtained as extruded bars. Their chemical compositions are shown in Table 1. These extruded bars were then, thermomechanically processed into thin rolled sheets (1 mm thick), from which tensile samples with a 5 mm gauge length and 4 mm width were machined. The gauge length was parallel to the rolling direction. Tensile tests were carried out over a wide range of strain rate

<table>
<thead>
<tr>
<th>Materials</th>
<th>Mg</th>
<th>Li</th>
<th>Cu</th>
<th>O</th>
<th>C</th>
<th>Al</th>
</tr>
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</table>
| IN9021      | 1.5| 4.0| 0.8| 0.8| 1.1| bal.
| IN905XL     | 4.0| 1.5| 0.4| 0.8| 1.2| bal.
| IN9052      | 4.0| 0.8| 0.8| 1.1| bal.|

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(10^{-3} to 300 s^{-1}) in air at temperatures between 698 and 873 K. Flow stresses at a fixed true strain of 0.1 were determined from each sample tested at a given temperature and strain rate. Microstructures of the samples were examined by transmission electron microscopy.

III. Results and Discussion

1. Microstructures

The microstructural features are very similar in all superplastic MA aluminum alloys. A typical microstructure of the superplastic IN9052, annealed at a superplastic temperature of 848 K for 9 \times 10^3 s, is given in Fig. 1. The annealed MA aluminum alloys consisted of very fine grained, equiaxed microstructures with an average grain size of about 0.5 \mu m. In fact, a number of dislocation structures were often observed within grains. Most grain boundaries were noted to be high-angled, but low-angled grain boundaries of less than 7 degree were also occasionally found. The microstructure also contained a large amount of fine oxide and carbide particles with sizes of less than 30 nm, which lead to a strong pinning effect on the migration of grain boundaries. The volume fraction of the fine particles was estimated to be almost 5 vol\% for each of the MA aluminum alloys.

2. Stress-strain behavior

Typical true stress-true strain curves at constant true strain rates from 10^{-3} to 300 s^{-1} are shown in Figs. 2–4 for IN9021, IN9052 and IN905XL, respectively. The curves in the low strain rates range are shown in the bottom part and those in the high strain rates range in the top part of each figure. The testing temperature is 823 K for IN9021 and IN905XL, but it is 863 K for IN9052. Typical superplastic materials exhibit a classical, well-behaved flat stress-strain curve, i.e., the flow stress remains almost constant during superplastic flow. The curves in the present study, however, indicate basically that strain hardening prevailed over the entire stress-strain curve of all MA aluminum alloys. However, significant strain softening is noted to occur in IN9021 after large deformation at strain rates faster than 20 s^{-1}. This relates to the fact that it is difficult to control tests performed at such a high constant true strain rate. Such hardening, which has been reported in a superplastic I/M

Fig. 1 A typical superplastic microstructure annealed at 848 K for 9 \times 10^3 s for IN9052.

Fig. 2 Typical true stress-true strain curves of IN9021 at a testing temperature of 823 K for constant true strain rates from 10^{-3} to 300 s^{-1}.

Fig. 3 Typical true stress-true strain curves of IN9052 at a testing temperature of 863 K for constant true strain rates from 10^{-3} to 300 s^{-1}.
7475 alloy\(^{(9)}\), is a typical manner related to dynamic grain growth. An average grain size after deformation of more than 1000\% under optimum superplastic condition for IN9021 was found to be similar to the initial undeformed grain size. This relates to the structural stability at high temperatures in MA aluminum alloys resulting from two effects; the first is the strong pinning provided by the fine oxide and carbide particles, the second is an effect arising from the instantaneous time for diffusion transportation from extremely high strain rates. An explanation for the strain hardening based on dynamic grain growth is not consistent with the present result. The strain hardening behavior in MA aluminum alloys may relate to the nature of initial boundaries present before hot deformation and the following evolution of boundary structures with straining. Further study such a measurement of grain boundary misorientation with elongation will be required to understand the origin of hardening in flow stress for MA aluminum alloys exhibiting superplasticity at high strain rates.

3. Strain rate dependence of flow stress and elongation

The superplastic behavior of the MA aluminum alloys against strain rate are noted to be quite similar: the flow stresses at all testing temperatures increase with strain rate, and the curves are of a sigmoidal shape, as has been observed in many other superplastic materials. In the equation \(\sigma = k\dot{\varepsilon}^m\), where \(\sigma\) is the flow stress, \(\dot{\varepsilon}\) is the strain rate, \(k\) is a constant incorporating structure and temperature dependencies, the strain rate sensitivity exponent, \(m\), is the slope of this curve \((d\ln \sigma / d\ln \dot{\varepsilon})\).

In general, large elongations are obtained when high \(m\) values are found, i.e., small elongations are obtained at low strain rates, with a corresponding low \(m\) value, but it increases with strain rate. Large elongations are obtained at extremely high strain rates (generally over 1 s\(^{-1}\)), with a corresponding high \(m\) value of greater than 0.3.

A typical variation in flow stress and elongation of IN9021 at four different testing temperatures from 723 to 848 K is represented in the top or the bottom part of Fig. 5 respectively as a function of strain rate\(^{(10)}\). It is noted that the using strain rates in this figure are higher than 1 s\(^{-1}\), i.e., positive exponent strain rates. In the low strain rate regime, low \(m\) values less than 0.3 \((n\geq3)\) and smaller elongations are found. In the superplastic range at very fast strain rates of more than 10 s\(^{-1}\), however, relatively high \(m\) values from 0.3 to 0.5 \((n=2−3)\) and corresponding large elongations are obtained. Also, the values of total elongation appear to remain high at yet higher strain rates. It has been reported previously that the maximum elongation for IN9021 or IN9021 alloys tested at temperatures below 773 K and at strain rates of about 1 s\(^{-1}\) is 500\%\(^{(12)}\). In contrast, in the present work, IN9021 tested at nearly 823 K, and higher elongations of more than 1000\% are obtained at extremely high strain rates between 10 and 300 s\(^{-1}\). Three specimens superplastically deformed to fracture at different testing conditions can clearly reveal that both of the specimens deformed at 823 K are fairly uniform with no visible necking, whereas the sample, exhibiting a smaller elongation of 750\% at a strain rate of 2 s\(^{-1}\) and at a lower temperature of 748 K, microscopically shows gradual and diffuse necking\(^{(10)}\). In contrast to the observed results obtained from these frac-

![Fig. 4 Typical true stress-true strain curves of IN905XL at a testing temperature of 823 K for constant true strain rates from 10\(^{-1}\) to 200 s\(^{-1}\).](image)

![Fig. 5 The variation in flow stress (top) and elongation (bottom) of IN9021 at four testing temperatures from 723 to 848 K as a function of strain rate.](image)
tured samples after tensile testing at 823 and 748 K respectively, the degree of diffuse and gradual necking in the former is larger than that in the latter, resulting from its lower $m$ value of 0.3. This also generally corresponds to the introduction of larger elongations in the former samples cause to the more stability in deformation by higher $m$ value.

The comparative superplasticity data for IN9021, IN9052 and IN905XL are summarized as follows; a maximum value of 1250% elongation is obtained at a strain rate of 50 s$^{-1}$ and at 823 K in IN9021. In comparison, a maximum elongation of 330% is obtained for IN9052 at a strain rate of 10 s$^{-1}$ at 863 K, and 190% is for IN905XL at a strain rate of 20 s$^{-1}$ and 848 K. All MA aluminum alloys showed superplasticity at extremely high strain rates of more than 1 s$^{-1}$. It clearly shows that mechanical alloying processing is a powerful method to produce the desired microstructures of less than 0.5 $\mu$m in grain size, required for very high strain rate (positive exponent) superplasticity in these aluminum alloys. The major reason that IN905XL does not show a large elongation over 300% is related to the fact that some coarse grains (3 to 10 $\mu$m in sizes) are present in the microstructure. The presence of coarse grains is resulted from poor control during mechanical alloying processes$^{(9)}$. A comparison of the elongation data from IN9021 obtained in the present study with those from other aluminum-based superplastic alloys produced by other processing routes reveals obviously that the maximum superplastic strain rate, at which an elongation over 1000% is found, increases with decreasing grain size. In fact, the optimum strain rate for MA aluminum alloys is almost one million times faster than that of the conventionally processed superplastic aluminum alloys.

4. Temperature dependence of flow stress, elongation and strain rate sensitivity

The variation in elongation to failure (top) and flow stress (bottom) of two different MA aluminum alloys tested at optimum superplastic strain rates as a function of temperature is shown in Fig. 6 for IN9021 and Fig. 7 for IN9052 respectively. Basically the dependence of the superplastic behavior in both MA aluminum alloys is noted to be quite similar: the elongations increase with temperature and reach a maximum value, then drop rapidly. The increment in elongation for IN9021 is significantly larger than that for IN9052. Whereas the flow stresses decrease with increasing temperature. It is noted that a discontinuity in flow stress is clearly found in both MA aluminum alloys, i.e., the significant drop in stress at temperatures between 730 and 750 K for IN9021 and at about 860 K for IN9052. Particularly in IN9021 the fall of the flow stress from 120 to 60 MPa is very significant to note. This strongly suggests that the dominant deformation mechanism in both MA aluminum alloys should change near the temperatures where the significant drops in stress are found.

The changes of the mean values of strain rate sensitivity exponent, $m$ value, in the superplastic strain rate range are shown in Fig. 8 as a function of testing temperature for all MA aluminum alloys. It is evident that the $m$ values of all MA aluminum alloys increase with temperature. The $m$ values at lower testing temperatures are in the range from 0.25 to 0.33 ($3 < n < 4$) for all MA aluminum alloys, are obviously lower than those of more than 0.5 ($n < 2$) obtained at higher testing temperatures. For example, for IN9021 tested at temperatures below 748 K,
the $m$ values are about 0.3. However the $m$ value increases with temperature and becomes higher than 0.5 at temperatures over 773 K. This high value of $m=0.5$ indicates that grain boundary sliding is the dominant deformation mode in MA aluminum alloys.

The optimum superplastic temperature of 823 K for IN9021 is noted to be much higher than the previously reported incipient melting point of the alloy (\(~773\) K$^{11}$). In fact, the incipient melting point of IN9021, used in the present study, was roughly determined to be only 740 to 750 K. The difference between the previous and the present value may be a result of entirely different thermo-mechanical treatments, which led to different solute distribution. Solute distribution can strongly affect the local melting points (e.g., near or at grain boundaries) in powder-metallurgy alloys, including metal-matrix composites. For the composites, segregation of alloying elements such as Mg and Cu was reported around reinforcements$^{10(12)}$. Also microchemical analysis by a field-emission TEM indicated very recently that the boundaries are segregated mostly by Si, O, and N. On the other hand, the interfaces are segregated mostly by Mg and O$^{13(14)}$.

Nonetheless, it is interesting to point out that the measured incipient melting point in IN9021 corresponds to the temperature at which the significant drop in stress is found and the $m$ value also starts to increase from a low value of 0.3 to a higher value of more than 0.5. It is pointed out again that the transient temperature of 860 K in IN9052, where the significant drop in stress is found and the $m$ value also increases from 0.3 to more than 0.5, corresponds to the published matrix solidus temperature of about 860 – 870 K for the Al-Mg alloys$^{15(16)}$. Nieh and Wadsworth$^{16}$ reported that there was no apparent discontinuity in superplastic properties of IN9021 and IN90211 across the melting point. However, an occurrence of both the drop in flow stress and an increment in $m$ value near or above the melting point is found in both MA aluminum alloys of IN9021 and IN9052. Large elongations are obtained also near or above the melting point. Therefore it is suggested that a liquid phase plays an important role in superplastic deformation for MA aluminum alloys. As recently suggested by Nieh and Wadsworth, the presence of a liquid phase give rise to superplasticity at high strain rates in the composites$^{16}$. However, the larger volume of a liquid, or the continuous liquid layer, can not support normal tractions, then not contribute to large elongations. It may be associated with intergranular decohesion at a liquid grain boundary leading to limited intergranular fractures. This can explain the drop in elongation observed at the highest temperature above the melting point in both MA aluminum alloys, as shown in Figs. 6 and 7.

A similar manner in the temperature dependence of the flow stress and elongation was reported in the Si$_3$N$_4$ reinforced aluminum alloy matrix composites$^{17}$. The possibility of partial melting along the interfaces and/or grain boundaries of the composites experimentally confirmed by differential scanning calorimetry$^{17(18)}$ and in-situ transmission electron microscopy$^{13(14)}$. An excellent agreement between partial melting temperature and optimum superplastic temperature suggested that a liquid phase plays an essential role in the superplastic flow of the composites at high strain rates. It was postulated that superplastic flow is controlled by a grain boundary sliding mechanism accommodated with relaxing the stress concentration by isolated liquid phases at interfaces between the matrix and reinforcements of the composites$^{19}$.

The results in this work reveal clearly that a liquid phase is related to mechanisms of superplastic deformation for MA aluminum alloys. Therefore, the presence of a liquid phase resulting from the low melting point region along grain boundaries, as a result of solute segregation during mechanically alloyed processing, appears to contribute significantly to the deformation mechanisms of high strain rate (positive exponent) superplasticity in MA aluminum alloys. Superplastic flow in MA aluminum alloys might be controlled by a grain boundary sliding mechanism accommodated with relaxing the stress concentration by isolated liquid phases at grain boundaries. A fundamental understanding of the deformation in the solid, liquid, and solid-liquid regions is necessary, in order to understand the superplastic deformation mechanism in MA aluminum alloys. The role of liquid phases in superplastic deformation is not sufficiently clear at present. Further detailed research is required to understand the role of liquid phases in superplastic deformation of MA aluminum alloys.

IV. Summary

(1) Very high strain rate (positive exponent) superplasticity in mechanically alloyed (MA) aluminum alloys, including IN9021, IN9052 and IN905XL, is characterized over a wide range of strain rates and temperatures.

(2) Experimental data revealed that superplasticity in three ultra-fine grained MA aluminum alloys occurred at temperatures close to or above the melting point of each alloy.

(3) The presence of a liquid phase, resulting from the low melting point regions, as a result of solute segrega-
tion by mechanical alloying, is proposed to be responsible for the observed positive exponent superplasticity in MA aluminum alloys.

(4) Mechanically-alloyed processing is a powerful method to produce the desired microstructures with not only fine grain size, but also in optimizing the segregation in solute at boundaries, required for positive exponent superplasticity in aluminum alloys.

REFERENCES