# SUPERPLASTICITY IN METALLIC MATERIALS

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## ABSTRACT

Superplasticity is the ability of a polycrystalline material to exhibit, in a generally isotropic manner, very high tensile elongations prior to failure. New progress has been made considerable in achieving high elongations under high strain rate conditions. Two maximum values in superplasticity are proposed, the highest superplastic *elongation* and the highest superplastic *strain rate*. The highest elongation is more than 8000 %, which is recorded in a commercial aluminum bronze deformed at a relatively high strain rate of 4 x $10^{-2}$ $s^{-1}$ at 1123 K. The highest superplastic strain rate is 300 $s^{-1}$, where an elongation of 1000 % is obtained in a mechanically alloyed IN9021 aluminum alloy with very fine grains of 500 nm.

## INTRODUCTION

It generally breaks after a small amount of plastic deformation, when polycrystalline materials are pulled in tension. Superplasticity refers to an ability of some materials to pull out to very high tensile elongations prior to failure. The highest elongations reported are 4850 % in a Pb-Sn eutectic alloy [1], and greater than 5500 % for an aluminum bronze [Cu-10wt%Al-4.5wt%Fe-6wt%Ni-2wt%Mn], which can be superplastically deformed at 1073 K and 6.3 x $10^{-3}$ $s^{-1}$ [2]. The ability to achieve large neck-free strains in tension is of interest not only scientifically but also because of the many potential applications for these types of materials in a very wide range of industrial forming operations. However, the rate of superplastic forming is relatively slow compared to many manufacturing processes and from a commercial viewpoint the development of materials that exhibit superplasticity at fast rates is desirable. From the microstructural aspect of the fact that almost superplastic materials contain fine grain sizes of less than about 10 μm, the most important requirement for superplastic flow is the refinement of grain size in materials. The structural feature can be modified to obtain such an enhancement in superplastic forming rates is to decrease the grain size. Very recently, superplasticity is found at very high strain rates of more than 1 $s^{-1}$ [labeled as positive exponent superplasticity] in mechanically alloyed aluminum alloys which consist of very fine grained structures of 500 nm [3~8]. A mechanically alloyed IN9021 alloy exhibited a maximum elongation of 1250 % at 50 $s^{-1}$ at 823 K [5]. These mechanically alloyed aluminum alloys contain a large amount of very fine oxide and carbide particles obtained by the mechanical alloying process. Due to the pinning effect of these fine particles, a fine grain or subgrain structure with near nano-scale is often obtained for superplastic deformation. The new generational superplasticity in metallic materials will be discussed in the present work.

## MATERIALS AND EXPERIMENTAL PROCEDURES

The compositions of Cu-Al-Fe-Ni-Mn alloys used in the present work are give in **TABLE 1**, which also includes typical superplastic properties. The main steps in the processing schedule used to develop superplastic fine grained microstructures are : homogenizing treatment at 1168 K for 8.4 ks, hot rolling at high temperatures between 1125 and 1168 K with a reduction of about 70 %, intermediate heat treatment at 913 K for 3.6 ks, warm rolling at 913 K with a large reduction of 80 % and cold rolling at room temperature with a small reduction of 20 %. Tensile samples with 5 or 10 mm gauge length and 5 or 8 mm width were machined from these rolled sheets of 2 or 3 mm thickness, with the gauge length parallel to the rolling direction. Tensile tests were carried out in air at high temperatures between 1023 and 1173 K and a strain rate range from $10^{-5}$ to 1 $s^{-1}$. The flow stresses for each sample were determined from the maximum engineering true stress.

TABLE 1 Compositions and typical superplastic properties of commercial aluminum bronzes.

| Alloy | Composition(wt%) | | | | | Superplastic performance | | | |
|---|---|---|---|---|---|---|---|---|---|
| code | Al | Fe | Ni | Mn | Cu | Temp.(K) | Strain rate ($s^{-1}$) | m value | E (%) |
| #X | 8.9 | 4.1 | 3.9 | 0.9 | Bal. | 1073 | $10^{-3}$ | 0.5 | 1800 |
| #Y | 10.0 | 4.5 | 6.0 | 1.7 | Bal. | 1073 | $6x10^{-3}$ | 0.6 | >5500 |
| #Z | 10.3 | 5.2 | 5.0 | 1.5 | Bal. | 1123 | $4x10^{-2}$ | 0.5 | >8000 |

The commercial mechanically alloyed IN9021 (Al-4.0wt%Cu-1.5wt%Mg-1.1wt%C-0.8wt%O) used in this work was obtained as extruded bars. The extruded bars were subsequently thermomechanically processed at our laboratory into thin rolled sheets (1 mm thick). Tensile samples with 5 mm gauge length and 4 mm width were machined from these rolled sheets, with the gauge length parallel to the rolling direction. Tensile tests were carried out in air at temperatures between 698 and 873 K and a wide range of strain rate from $10^{-3}$ to 2000 $s^{-1}$. The flow stresses for each sample were determined from the fixed true strain of 0.1.

Low strain rate tests (less than $10^{-1}$ $s^{-1}$) were performed with an Instron machine, and intermediate strain rate tests ($10^{-1}$ ~ 300 $s^{-1}$) were done with a hydraulic tensile testing machine, and the dynamic tensile tests (400 ~ 2000 $s^{-1}$) were done using a split Hopkinson pressure bar system which incorporates a specific attachment.

## RESULTS AND DISCUSSION

Commercial aluminum bronzes

From the approximate relationship between composition, temperature and phase field in Cu-Al-Fe-Ni alloys for the compositions in TABLE 1, the aluminum bronzes used in the present work have complex microstructures consisting of $\alpha+\kappa$ or $\alpha+\beta+\kappa$ phases at the range of temperatures from 873 to 1173 K [9]. The $\alpha$-phase, in fact, consists of two aluminides based on $Fe_3Al$ and NiAl, respectively. Typical superplastic microstructures obtained in the commercial aluminum bronzes #X, #Y and #Z are shown in **Figure 1** after annealing for 1.8 ks at 1073 K for both alloys #X and #Y, and at 1123 K for alloy #Z. In alloy #X the microstructure consists of f.c.c. $\alpha$-phase grains of mean diameter 2 μm, with about 15 % volume fraction of fine uniformly distributed $\kappa$ particles of less than 1 μm in sizes. Alloy #Y consists of equal volume fractions of $\alpha$ and $\kappa$ phases each about 2 μm in size, while alloy #Z consists of $\alpha+\beta+\kappa$ phases having respective volume fractions of 30, 40 and 30 %, and corresponding phase sizes of about 2, 4 and 2 μm.

The changes in flow stress and m value with strain rate at a typical superplastic temperature of 1073 K for three commercial aluminum bronzes #X, #Y and #Z are shown in **Figure 2**. The flow stress for alloy #Z is lower and maximum m value is attained at a higher strain rate than for the other alloys. These observations are due to the presence of a small amount in $\beta$-phases at 1073 K, which is a soft accommodating constituent at high temperatures [10]. In the case of alloys showing $\alpha+\kappa$ microstructures at 1073 K, alloy #Y has a lower flow stress, and maximum m value at higher strain rates than for alloy #X. This reflects the stabilizing effect of the large volume fraction of $\kappa$-phase in alloy #Y.

**Figure 3** shows the change of mean size of $\alpha$-grains and cavitation behavior of alloys #X, #Y and #Z with strain at 1073 K for the strain rates given in TABLE 1. In all alloys dynamic growth of both grains and cavities is observed during superplastic flow. Grain growth is least in alloy #Y which contained approximately equal volume fractions of $\alpha$ and $\kappa$ phases, whereas alloys #X and #Z have a higher and similar rate of $\alpha$-grain growth. However, the rate of cavitation with strain for alloys #Y and #Z which contain large volume fractions of second and/or third phases are smaller than those of alloy #X with a smaller volume fraction of second phase particles. The lower rate of cavitation in alloy #Y is attributed to its high microstructural stability and in alloy #Z to the presence of a significant volume fraction of the highly accommodating $\beta$-phase.

The superplastic elongations of three aluminum bronzes #X, #Y and #Z are shown in **Figure 4** as a function of strain rate for different temperatures between 1023 and 1173 K, at which these alloys have complex microstructures consisting of $\alpha+\kappa$ or $\alpha+\beta+\kappa$ phases. It can be seen for alloys #Y and #Z that very high elongations of more than 4000 % can be obtained at relatively high strain rates from $10^{-3}$ to $10^{-1}$ $s^{-1}$, and this is consistent with the location of maximum m value at higher strain rates in Figure 2. As shown in **Figure 5**, the highest elongation of more than 8000 % is obtained in alloy #Z deformed at a high strain rate of $4x10^{-2}$ $s^{-1}$ at 1123 K at which its microstructure consists of $\alpha+\beta+\kappa$ phases. Aluminum bronze containing $\beta$-phases have the greater superplastic deformation potential. However, it is difficult commercially to develop a uniform microstructure, and also there are problems associated with the post-deformation properties of the material. It is easier to develop and control the

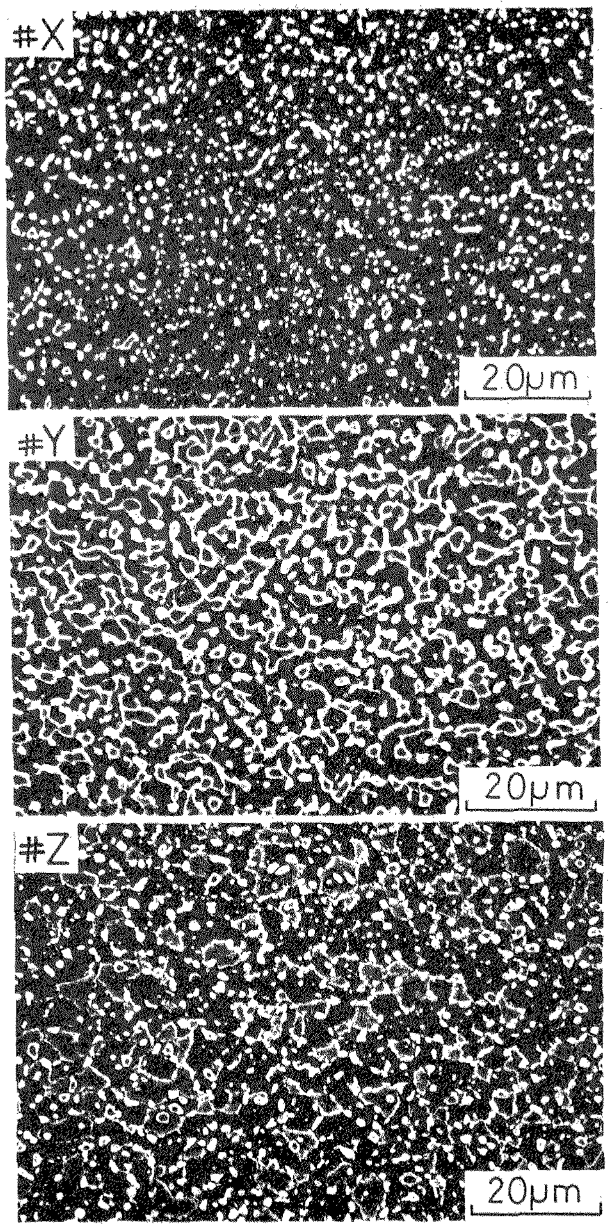


**Figure 1.** Typical superplastic microstructures obtained in the commercial aluminum bronzes #X, #Y and #Z after annealing for 1.8 ks at 1073 K for both alloys #X and #Y, and at 1123 K for alloy #Z.

$\alpha$+$\kappa$ microstructures in commercial aluminum bronze. From the observations for volume fraction effect of $\kappa$-phase on the superplastic elongation of $\alpha$+$\kappa$ alloys for the range of superplastic temperatures from 873 to 1173 K, it is concluded that optimum superplastic properties are obtained for microstructures with intermediate volume fractions of each of the two $\alpha$ and $\kappa$ phases.

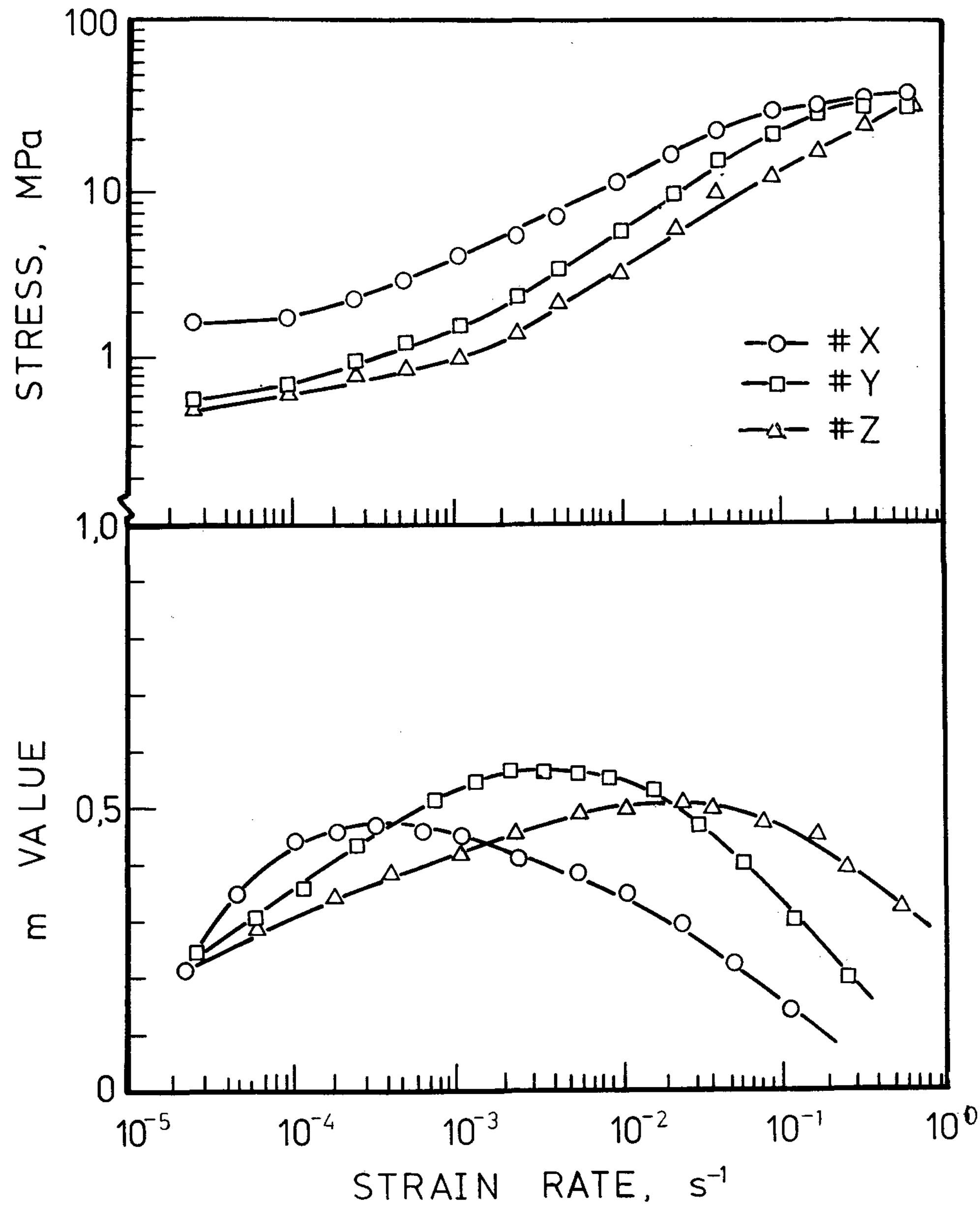


Figure 2. The changes in flow stress and m value with strain rate at a typical superplastic temperature of 1073 K for commercial aluminum bronzes #X, #Y and #Z.

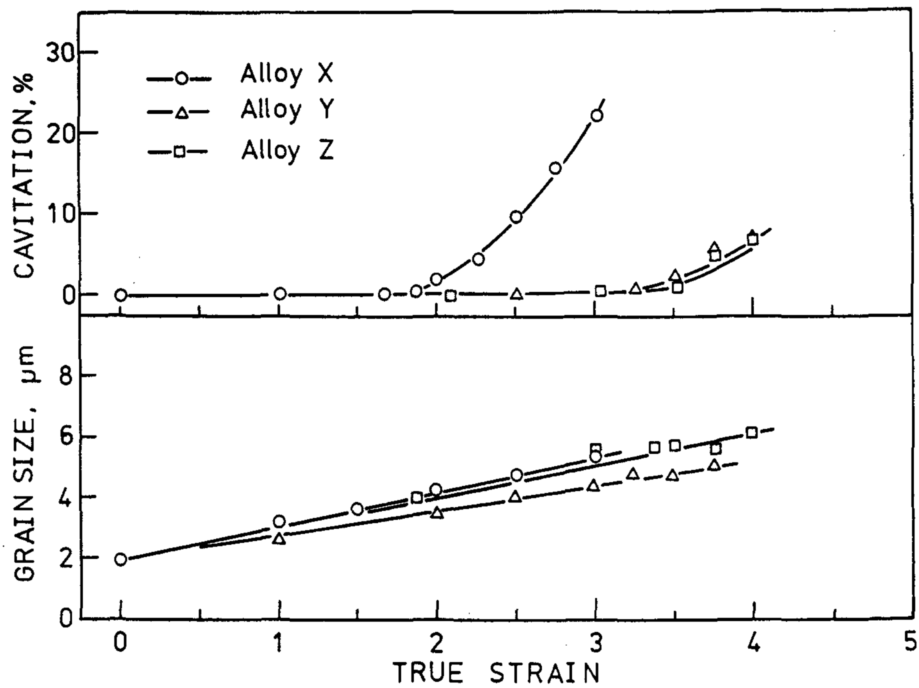


Figure 3. The change of mean size of α-grains and cavitation behavior of alloys #X, #Y and #Z with strain at 1073 K for the strain rates given in TABLE 1.

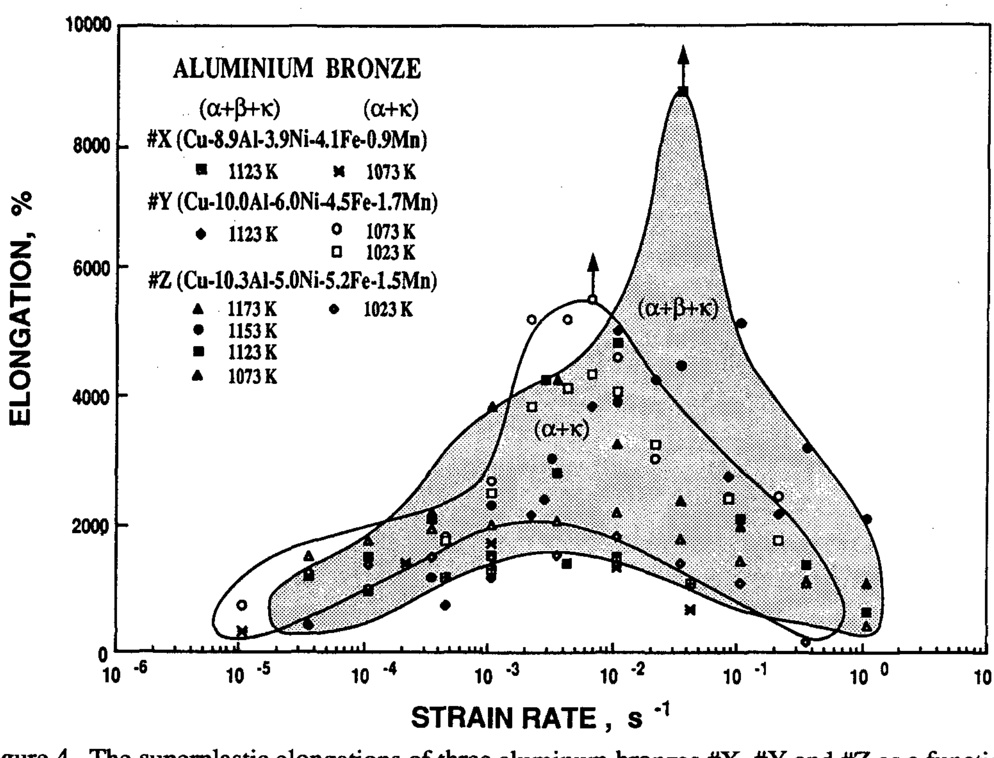


Figure 4. The superplastic elongations of three aluminum bronzes #X, #Y and #Z as a function of strain rate for different temperatures between 1023 and 1173 K, at which these alloys have complex microstructures consisting of α+κ or α+β+κ phases.

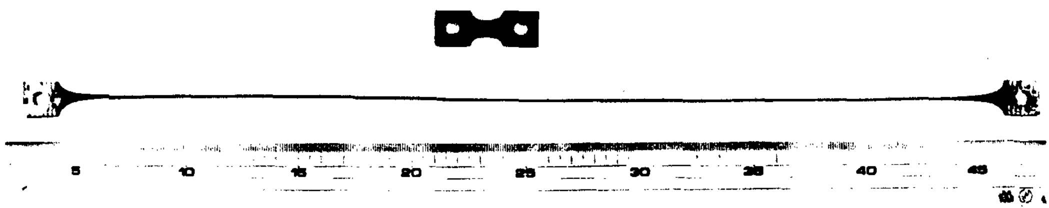

Figure 5. Exceptional superplasticity in an aluminum bronze pulled in tension at 1123 K to an elongation of 8000 %.

In these superplastic aluminum bronze cavitation does occur during superplastic flow, as shown in **Figure 6**, including previously reported results of other superplastic alloys. It can be seen that there is a linear relationship between the volume of cavities plotted logarithmically and superplastic true strain, that is consistent with plasticity controlled cavity growth [11]. It is obvious that the levels in cavitation of these superplastic aluminum bronzes are smaller than those of other superplastic alloys and are very low for commercially significant strains.

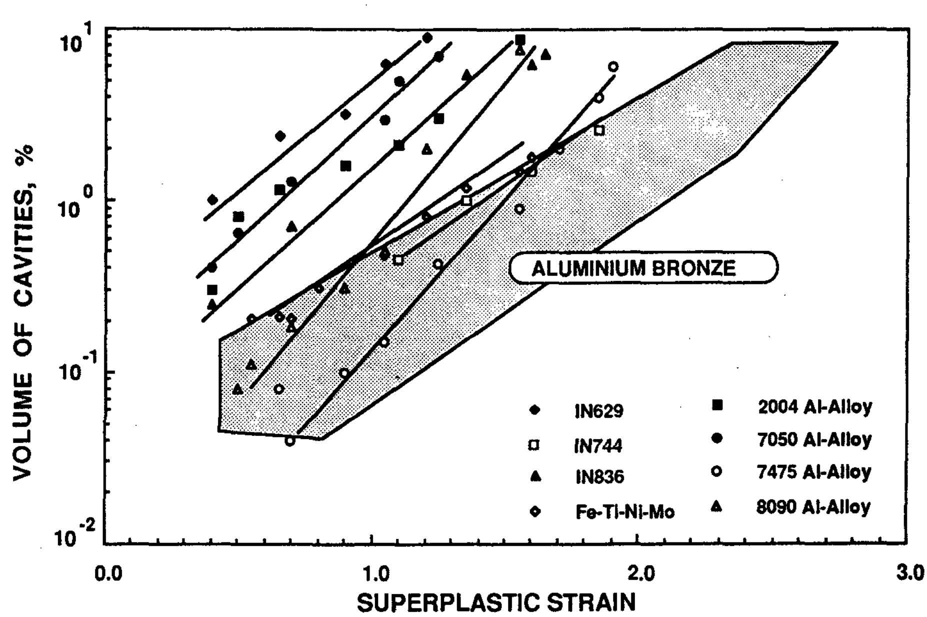


Figure 6. Cavitation behavior of commercial aluminum bronzes, including previously reported results of other superplastic alloys.

Commercial mechanically alloyed IN9021 aluminum alloy

The comparative superplasticity data for three mechanically alloyed aluminum alloys and a mechanically alloyed aluminum composite are summarized in **TABLE 2**. The microstructure consists of very fine grains of about 500 nm in all mechanically alloyed materials [7,8]. Most grain boundaries are observed to be high angled, and occasionally grains with low-angle boundaries of less than 7 degree are found. Also, a large number of very fine carbides and oxides with particle sizes less than 30 nm are found in a very fine-grained matrix of all alloys. The volume fraction of the fine particles is almost 5 vol% for all alloys. One of the reasons that IN905XL did not show a large elongation over 300 % is related to some coarse grains (3 to 10 μm in sizes) which resulted from a poor control during mechanical alloying processes [7].

TABLE 2 Superplastic properties of mechanically alloyed aluminum alloys and composite.

| Materials | Temperature (K) | Strain rate ($s^{-1}$) | Stress* (MPa) | m value | Elongation (%) | Grain size (nm) |
|---|---|---|---|---|---|---|
| IN9052 | 863 | 10 | 15 | 0.6 | 330 | 500 |
| IN905XL | 848 | 20 | 12 | 0.6 | 190 | 400 |
| IN9021 | 823 | 50 | 18 | 0.5 | 1250 | 500 |
| SiCp/IN9021 | 823 | 5 | 5 | 0.5 | 600 | 500 |

* True stress at $\varepsilon$=0.1

Variation in flow stress (top) and elongation (bottom) for a mechanically alloyed IN9021 aluminum alloy is plotted in **Figure 7** as a function of strain rate for testing temperatures from 698 to 873 K. There are some similarities in superplastic behavior in all mechanically alloyed materials : the flow stress at all testing temperatures increased with strain rate, and the curves are of typical sigmoid shape as has been observed for superplastic materials. In the low strain rate regime, the strain-rate sensitivities, less than 0.05 ($n \geq 20$), are indicative of an apparent threshold stress, and in the superplastic range, a relatively high strain rate sensitivity of more than 0.5 ($n=2$) was obtained at higher temperatures, with corresponding relatively large elongations. It is evident for all mechanically alloyed materials that the m value increases from about 0.3 to more than 0.5 with the test temperature increases.

Small elongations are obtained at low strain rates, however, the elongation increases with strain rate and large elongations are found in the extremely high strain rate range over 1 $s^{-1}$, that is, at a positive exponent strain rate. Especially, for the IN9021 alloy tested at nearly 823 K, very large elongations of more than 1000 % are obtained at the extremely high strain rates between 10 to 300 $s^{-1}$. Also about 200 % was obtained at an impact strain rate of 2000 $s^{-1}$. An example of the fractured specimens was shown in **Figure 8** for the IN9021 alloy. It is clear, by comparison with an untested specimen, that the specimens deformed at very high strain rates of more than 1 $s^{-1}$ are relatively uniform with little or no necking. Furthermore, the maximum value in elongation is 1250 %, which is obtained at a strain rate of 50 $s^{-1}$ and at 823 K. In addition, a large elongation of 1000 %, which is accepted reasonably as an enough value for the superplastic elongation, was recorded at 300 $s^{-1}$, also, an elongation of about 200 % was obtained at an impact strain rate of 2000 $s^{-1}$. So, it is 300 $s^{-1}$ for the highest superplastic strain rate where a large elongation of 1000 % was obtained in superplastic materials.

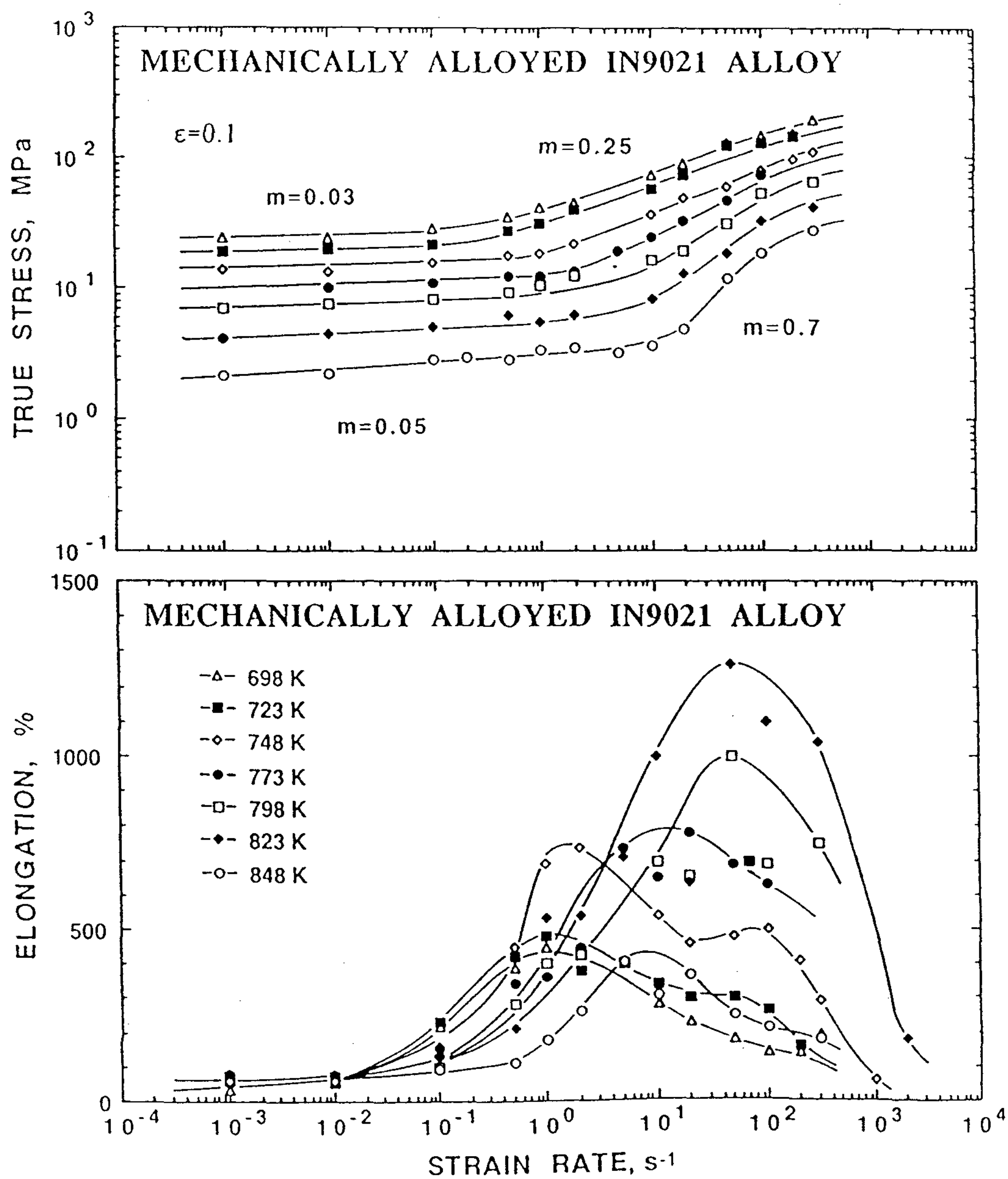


Figure 7. Variation in flow stress (top) and elongation (bottom) for a mechanically alloyed IN9021 aluminum alloy as a function of strain rate for testing temperatures from 698 to 873 K.

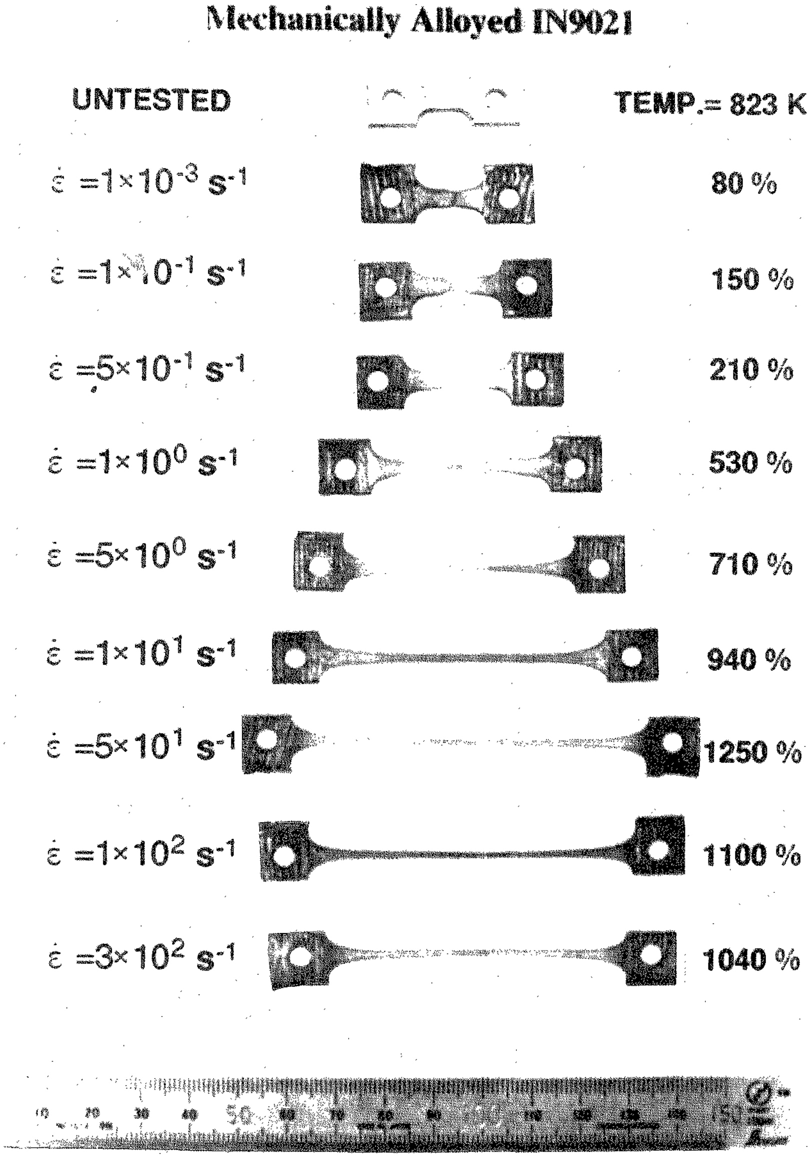


**Figure 8.** An example of the fractured specimens for the mechanically alloyed IN9021 alloy.

The phenomenological equation relates the superplastic strain rate to grain size, flow stress and diffusion, is given as follows:

$$\frac{\dot{\varepsilon}}{D}=K\left(\frac{\sigma}{E}\right)^{n}\left(\frac{b}{d}\right)^{p} \qquad (1)$$

where $\dot{\varepsilon}$ is the strain rate, $D$ is the diffusion coefficient, $K$ is a constant that is principally a function of the deformation mechanism, $d$ is the grain size, $b$ is the Burgers vector, $p$ is the grain size exponent, $n$ is the stress exponent, $\sigma$ is the stress and $E$ is the dynamic unrelaxed average Young's modulus. The knowledge of parameters of $n$ and $p$ allows the elucidation of the rate-controlling mechanism. In general, $n$ is nearly 2 for superplastic region when grain boundary sliding is dominate deformation mechanism and $p$ often takes on values of 2~3, which depends on the diffusion-controlled mechanism.

The optimum strain rate for a constant superplastic flow increases with refinement of grain size in materials according to eqn. (1). The strain rates where the maximum elongations were obtained in superplastic alloys in the present work, are shown in **Figure 9** as a function of the reciprocal of grain size. Included in this figure are other previously reported results for powder metallurgically processed aluminum alloys and a typical superplastic I/M 7475 aluminum alloy. Regardless of the processing route, it is clear that the optimum superplastic strain rate increases roughly with decreasing grain size. There is a linear relationship between strain rate and grain size; the grain size exponent $p$ which is the slop of this line is about 3, which is equal to the value when plastic deformation by grain boundary sliding accommodated by slip is characterized by dominate grain boundary diffusion [12]. It is premature to draw the conclusion, however, from the apparent relationship between optimum superplastic strain rates and grain sizes of various materials, that the grain size exponent is 3 for these superplastic materials. The values of the optimum strain rates for maximum superplastic elongations were not determined under the constant superplastic flow stress of $(\sigma/E)^n$. Further experimental evidence incorporating the grain size dependence of both superplastic elongation and flow stress is needed to determine the exact value of the grain size exponent $p$ in the superplastic strain rate range.

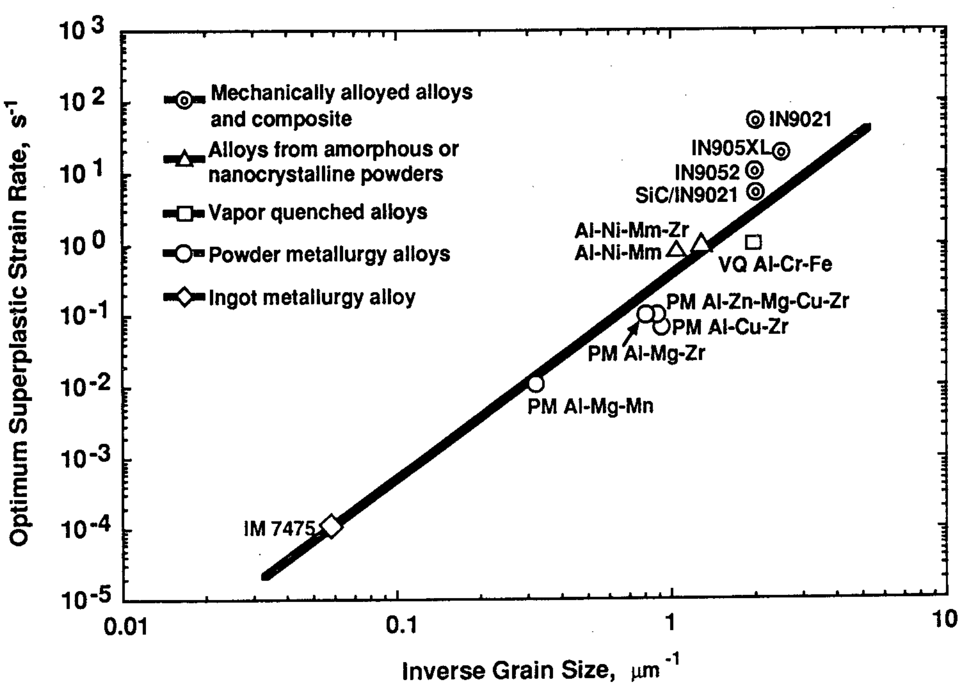


Figure 9. The change of the superplastic strain rates as a function of the reciprocal of grain size.

## CONCLUSIONS

Two maximum values for superplasticity in metallic materials are proposed, *i.e,* the highest superplastic elongation and the highest superplastic strain rate. The highest elongation is more than 8000 %, which is recorded in a commercial aluminum bronze deformed at a relatively high strain rate of 4 x $10^{-2}$ $s^{-1}$ at 1123 K. The highest superplastic strain rate is 300 $s^{-1}$, where an elongation of 1000 %, is a reasonably accepted value for the superplastic elongation, is obtained in a mechanically alloyed IN9021 aluminum alloy. Positive exponent superplasticity is found in near-nano scale materials produced by advanced processing. It is important for near-nano scale materials that the microstructural stability at high temperatures would be achieved with optimum microstructural control. The new generational superplasticity will be achieved under the most appropriate deformation condition in nano scale materials produced by optimized microstructural control.

## REFERENCES


1 M.M.I.Ahmed and T.G.Langdon, Metall. Trans., *8A*(1977)1832.
2 K.Higashi, T.Ohnishi and Y.Nakatani, Scripta. Metall.,*19*(1985)821.
3 T.G.Nieh, P.S.Gilman and J.Wadsworth, *ibid*, *19*(1985)1375.
4 T.R.Bieler, T.G.Nieh, J.Wadsworth and A.K.Mukherjee, *ibid*, *22*(1988)81.
5 K.Higashi, T.Okada, T.Mukai and S.Tanimura, *ibid*, *25*(1991)2053.
6 K.Higashi, T.Okada, T.Mukai, S.Tanimura, T.G.Nieh and J.Wadsworth, *ibid*, *26*(1992)185.
7 K.Higashi, T.Okada, T.Mukai and S.Tanimura, *ibid*, *26*(1992)761.
8 K.Higashi, T.Okada, T.Mukai and S.Tanimura, Mater. Sci. Eng., *A159*(1992)L1.
9 M.Cook, W.P.Fentiman and E.Davis, J.Inst.Metals, *80*(1952)419.
10 W.J.D.Patterson and N.Ridley, J.Mater. Sci., *16*(1981)457.
11 J.Pilling and N.Ridley, Res. Mechan., *23*(1988)31.
12 O.D.Sherby and J.Wadsworth, "Deformation Processing and Structure", ed by G.Krauss, ASM, Metals Park, (1984)355.