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A new grain-refinement process for superplasticity of high-strength 7075 aluminium alloy

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The superplasticity of high strength 7075 aluminium alloy has been improved to a great extent by the new thermomechanical treatment proposed. This treatment (TMPA) includes solution treatment, overageing, warm-rolling deformation, recrystallization and an artificial ageing process. The maximum elongation may be up to 2100% under deformation at an initial strain rate of $8.33\times10^{-4}\ s^{-1}$ and a temperature of 510 °C, which is much higher than reported before. Observation of the microstructure changes revealed that the excellent superplastic elongation of the alloy seems mainly to be due to a decrease in the grain growth rate of the alloy and a reduction in the number of cavities nucleated during superplastic deformation.

1. Introduction

In recent years, various thermomechanical processes have been developed to produce a fine grain size in high-strength aluminium alloys [1–6]. Paton *et al.* [7] have applied their TMP method and processed 7075 and 7475 aluminium alloys. These materials exhibited optimum superplastic behaviour at 789 K (516 °C) and a strain rate of $2\times10^{-4}\ s^{-1}$. However, Bampton *et al.* [8] reported that, because the high-strength 7075 aluminium alloy contains more iron and silicon impurities than the 7475 aluminium alloy, its maximum elongation is lower than that of 1200% of 7475 aluminium alloy [8, 9].

In this paper, we propose a new thermomechanical process (TMPA) to produce a stable fine grain structure in high-strength aluminium alloy 7075. After grain refinement by this new method, the superplasticity of 7075 aluminium alloy will be improved greatly.

2. Experimental procedure

The composition of the 7075 aluminium alloy examined in this research is given in Table I. The alloy was in the form of a 10 mm thick sheet.

The parameters for the new thermomechanical treatment are presented in Table II. Solution heat treatment was carried out in furnace baths where the temperatures were controlled to within ± 5 K of the desired temperature. Deformation was carried out by rolling at temperatures between 200 and 220 °C. Between passes through the rolling mill, specimens were replaced in the furnace for periods ranging from 2–5 min while the roll gap was being reset. The duration of the reheating periods over the range of 2–5 min had no effect on the recrystallized grain size.

Tensile samples of 10 mm gauge length, 5 mm width, and thickness of 1.5 mm, were machined from the sheet after grain refinement by the new method. The tensile axis of the samples was always parallel to the rolling direction. Tensile testing was done on a 100 kN capacity Instron utilizing a three-zone furnace and chromel–alumel thermocouples for temperature control and measurement. Temperature was maintained constant to within ± 3 K both with respect to time and position along the gauge length of the sample. Testing was accomplished utilizing constant crosshead speeds throughout each test. Grain size was measured by the linear intercept method on micrographs [4].

Transmission electron microscopy (TEM) was accomplished on specimens removed from bulk material such that foil normals were parallel to the sheet normal direction. Thinning was accomplished in a solution of 25% nitric acid in methyl alcohol at 243 K (− 30 °C) and the specimens were examined in a EM-420 TEM operated at 120 kV.

An EPM-180 electron probe was used to examine the segregation of solution atoms on the grain boundary during superplastic deformation.

3. Results and discussion

In Fig. 1a, it is shown that 7075 Al contains a dispersion of insoluble particles formed by chromium and manganese additions to the alloy. These particles are

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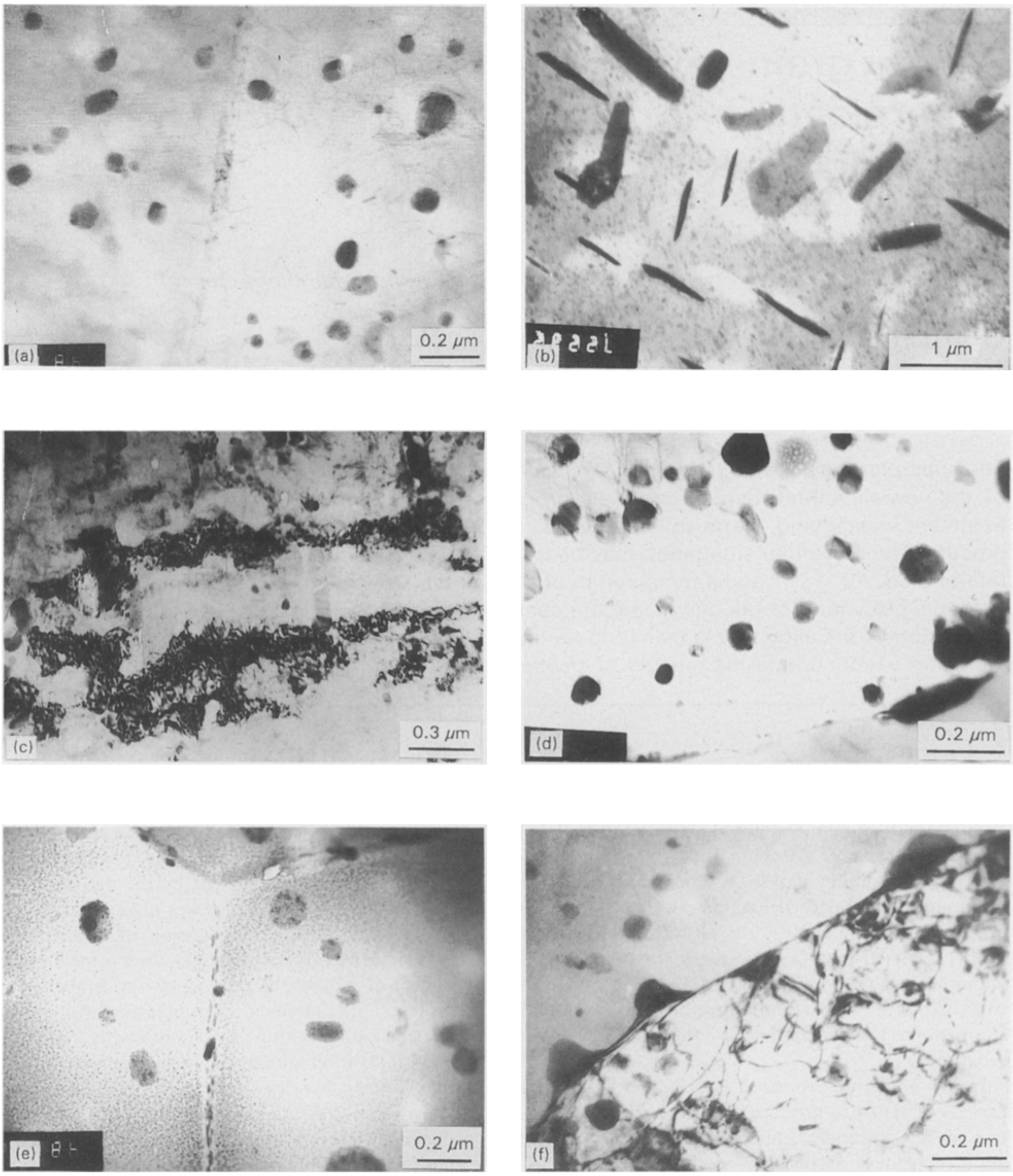

Figure 1 Transmission electron micrographs of structure produced during each step of the TMPA treatment and during superplastic deformation. (a) Solution treated 3 h/490 °C; (b) solution treated, aged 2 h/350 °C; (c) solution treated, aged 2 h/350 °C; deformed 85% at 220 °C; (d) treated as (c), recrystallized 30 min at 490 °C; (e) treatment (d), artificial ageing 1 h/100 °C → 5 h/180 °C; (f) treatment (e), deformed to a true strain of 1.0 at a temperature of 510 °C and strain rate of $8.33 \times 10^{-4}\ s^{-1}$.

approximately 0.1 μm diameter and fairly homogeneously dispersed [4].

The purpose of air cooling after solution treatment was to shorten the overageing time (8 h) as defined by Paton *et al.* [4].

As shown in Fig. 1b, after overageing for 2 h, not only large precipitate particles but also fine dispersoid particles were formed in alloy. These large precipitate particles may be the M phase, a mixture of the isomorphous phases $MgZn_2$ and MgAlCu [10]. The small particles are either dispersoid particles formed from chromium and manganese in solution after solution treatment or small M-phase equilibrium precipitates. There were also some large particles containing aluminium, iron and silicon impurities. They were not dissolved during solution treatment. These particles play a small role in grain refinement of the alloy [4].

Fig. 1c is a transmission electron micrograph of structures produced after 85% reduction of thickness. The heavily dislocated microstructure was created in this process.

Fig. 1d shows a transmission electron micrograph in which the grain growth has been retarded by the

TABLE I Chemical composition (wt %) of 7075 Al used

Zn	Mg	Cu	Mn	Cr	Fe	Si	Ti	Al
5.83	2.16	1.39	0.25	0.14	0.38	0.17	0.03	Balance

TABLE II Optimum thermomechanical treatment parameters for 7075 Al

Step	Temperature (°C)	Time (h)	Conditions
Solution treatment	490	3	Air cooling
Overageing	350	2	Water quenching
Deformation by rolling	220	85%	Reduction of thickness
Recrystallization	490	0.5	Water quenching
Artificial ageing	100 °C×1 h → 180 °C×5 h		Water quenching

small dispersoid particles during the recrystallization process. In Fig. 1e, narrow precipitate-denuded zones, formed by adding the artificial ageing process may be seen. There are also many M-phase precipitate particles formed in this process.

Fig. 1f shows the microstructure during superplastic deformation. It is seen that grain growth was retarded by the small dispersoid particles formed either by chromium or manganese or small M-phase particles which have not already resolved.

Fig. 2 shows the elongation obtained in testing at 510 °C as a function of strain rate under ageing and no ageing conditions. It is seen that, after the artificial ageing process, the superplastic elongation of the alloy is increased greatly; when the alloy is deformed at 510 °C and at an initial strain rate of $8.33 \times 10^{-4}\ s^{-1}$, the maximum elongation is increased from 850% to 2100% by adding the artificial ageing process.

Fig. 3 illustrates the grain-size changes during superplastic deformation of the aged and unaged specimens. It is shown that the grain-size stability of the aged specimen is increased. This is the important reason why the aged specimen undergoes large elongation.

Plots of true stress versus true strain during deformation of the aged and unaged specimens are shown in Fig. 4. It is seen that the strain hardening for the unaged specimen is stronger than that of the aged specimen. This may affect the cavity nucleation of specimens during superplastic deformation.

Fig. 5 shows the macroscopic appearance of some of the specimens after superplastic tension. It is seen that the aged specimen has a more homogeneous deformation than the unaged one.

In order to understand further the role of the artificial ageing process, cavity distributions of the aged and unaged specimen are compared in Fig. 6 when the 7075 Al alloy was deformed to a same strain level of 2.1. It can be seen that after the ageing process, the number of cavities formed during deformation was much less than that with no ageing. It is well known that cavitation damage is responsible for the premature failure of many superplastic aluminium alloys

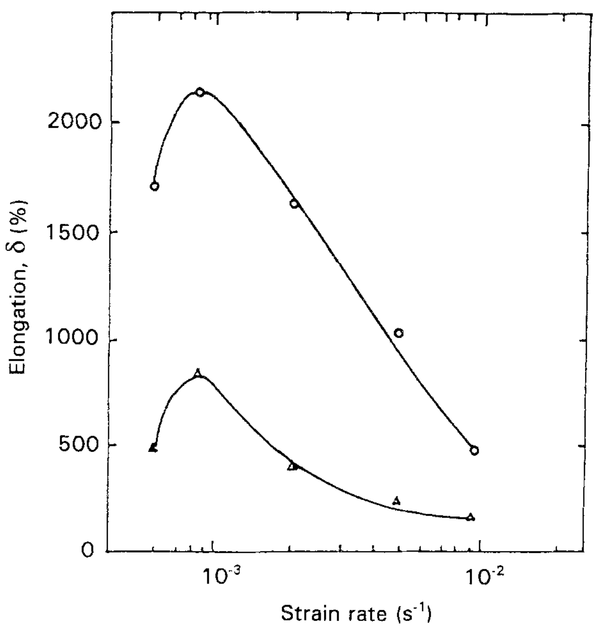

Figure 2 Elongation as a function of log strain for 7075 aluminium alloy: (○) aged; (△) unaged.

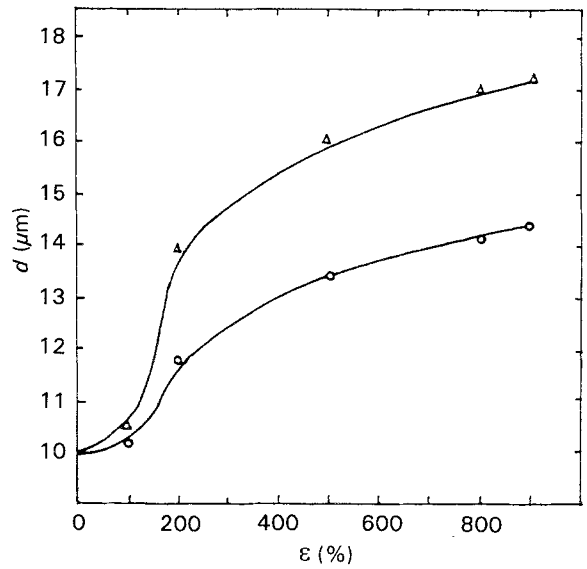

Figure 3 Grain size, *d* (μm), as a function of strain: (○) aged, (△) unaged.

[11]. So if the number of cavities is reduced during superplastic deformation, the superplastic behaviour will certainly be improved.

Fig. 7 is the electron probe test which shows the concentration of copper, magnesium and zinc elements on grain boundaries for the aged specimen during superplastic deformation. Although the M-phase particles which precipitate during artificial ageing will be resolved at high temperature, it is difficult for all of them to be resolved during the early stages of superplastic deformation. Furthermore, the dissolved M phase can induce the concentration of copper, magnesium or zinc elements on grain boundaries, as shown in Fig. 7. This may also be of benefit for stablizing the grain size of the alloy during deformation.

The principle of grain refinement of the thermomechanical treatment in this paper is that, after the

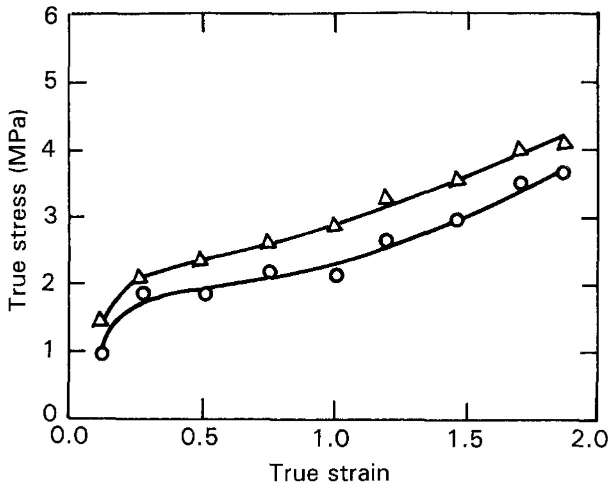

Figure 4 Plots of true stress versus true strain for the (○) aged and (△) unaged specimens during superplastic deformation.

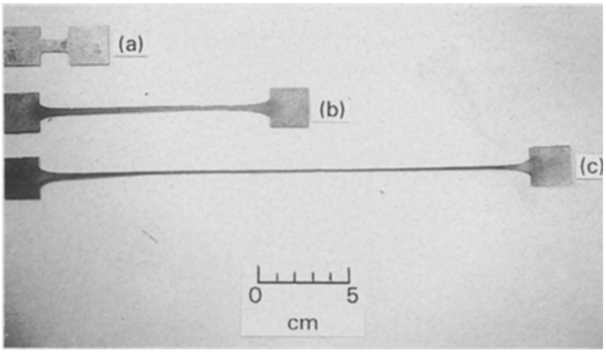

Figure 5 Tensile specimens of 7075 Al alloy. (a) The original specimen, (b) the unaged specimen tested at 510 °C and initial strain rate $8.33\times10^{-4}\ s^{-1}$, (c) the aged specimen tested at 510 °C and initial strain rate $8.33\times10^{-4}\ s^{-1}$.

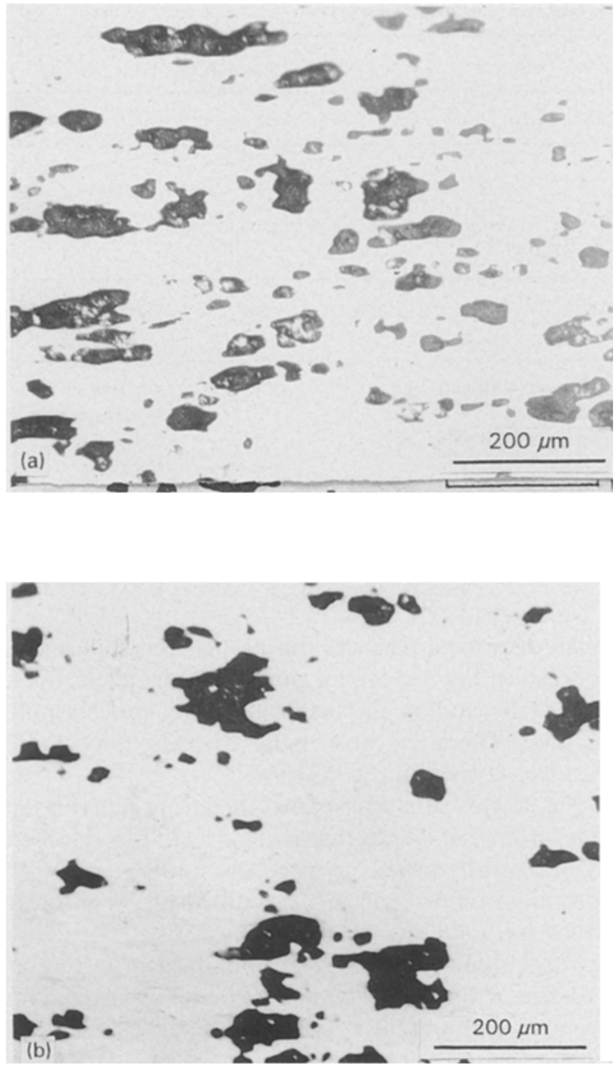

Figure 6 Cavity distributions of 7075 aluminium alloys when deformed at a temperature of 510 °C and initial strain rate of $8.33\times10^{-4}\ s^{-1}$ to the same strain level of 2.1: (a) unaged specimen, (b) aged specimen.

solution treatment and overageing process, dispersions of large particles recrystallizing grains and of small particles decreasing grain growth are formed. During the warm-rolling process, strong deformation zones are formed around the undeformed particles and the dislocation cells near larger particles have a lattice orientation quite different from the general orientation away from particles. During recrystallization, high-angle boundaries are formed around the highly misoriented cells near large particles, and these cells become nuclei for recrystallizing grains. After recrystallization, the growth of new grains is restricted by small dispersed particles. Finally, a fine grain size is obtained.

The role of artificial ageing after recrystallization is to make M phase [12] precipitate in order to improve the distribution of the second-phase particles. During superplastic deformation, both the M-phase particles which have not been dissolved and the dispersion of insoluble particles which contain chromium and manganese, retard the growth of grains. The dissolved M phase can produce some element concentration on grain boundaries which may also be useful for reducing the rate of grain-boundary movement. As shown in Fig. 3, the magnitude of the grain growth of aged specimen during superplastic deformation is obviously lower than that of the unaged specimen.

Obtaining a fine and stable grain size is necessary for the alloy to exhibit superplasticity before deformation. After grain refinement by the TMPA treatment proposed in this paper, not only the grain size of 7075 alloy is refined but also the stability of grain size is reinforced, which is beneficial for reducing the amount of cavity nucleation. So the 7075 alloy exhibits excellent superplasticity. It should be pointed out that the maximum elongation of 7075 alloy obtained in this paper is much higher than that reported in the literature and the optimum strain rate is $8.33\times10^{-4}\ s^{-1}$ which is about four times higher than $2\times10^{-4}\ s^{-1}$ reported by Hamilton *et al.* [8]. This may be beneficial for the superplastic forming of 7075 Al alloy.

Although the thermomechanical treatment proposed in this paper includes the artificial ageing process, the time for finishing the whole treatment is no longer than the TMP proposed by Wert *et al.* [4]. In their treatment, the overageing time is 8 h, but in the TMPA treatment proposed in this paper, the overageing time is only 2 h, which is beneficial for practical application.

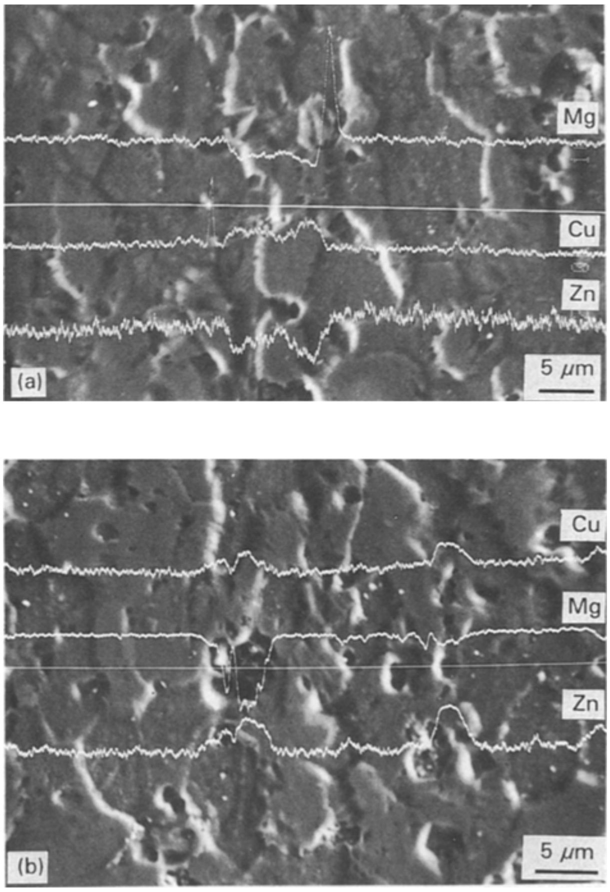

Figure 7 Electron probe showing the concentration of copper, magnesium and zinc on grain boundaries for the aged specimen when deformed at a temperature of 510 °C and a strain rate of $8.33 \times 10^{-4}\ s^{-1}$. (a) Magnesium, copper and zinc linear distribution, from the top line to the bottom line. (b) Copper, magnesium and zinc linear distribution, from the top line to the bottom line.

4. Conclusions

1. Adding artificial ageing to the modified thermomechanical treatment can greatly improve the superplasticity of 7075 aluminium alloy. After grain refinement by the TMPA process proposed in this paper, the maximum elongation of the alloy can be increased to 2100% when 7075 Al alloy is deformed at 510 °C and an initial strain rate of $8.33 \times 10^{-4}\ s^{-1}$.

2. The improvement of superplasticity of the 7075 aluminium alloy by the TMPA procedure is due to a decrease in the grain-growth rate of the alloy and a reduction in the number of cavities nucleated during superplastic deformation.

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