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The effect of layer number on the superplasticity of laminate 7475/2091 Al alloy

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The layer number is one of the important structure parameters in laminate material. For laminate 7475/2091, the laminate with same thickness but different layer number from 2 to 20 were processed and studied. Experiment results show that the layer number of the laminate has a strong influence on the superplasticity of the laminate. Microscopy observations indicate that a metallurgical bonding at interface is obtained between two components in the laminates. There is a diffusion effect region on each side of a bonding interface. Cavities nucleate and grow there, especially at bonding interfaces in optimum superplastic deformation condition. The cavity evolution during superplastic deformation is an important factor that affects total elongation rate of the laminate. © *2000 Kluwer Academic Publishers*

1. Introduction

Laminates have been reported to have superior combined properties to its constituent materials which include improved ductility, toughness [1], fracture resistance [2], fatigue property [3], corrosion resistance etc. They have shown a strong potential in engineering service. However, care must be taken while choosing forming methods for the laminates. A critical restriction is to keep the entirety of the laminates during forming. Superplastic forming can fit for the requirement and is one of the promising hot forming methods for the materials. With the forming method, products with complicate shape can be produced in single deformation operation. It was reported that superplastic forming was employed for production of airframe primary structures and aeroengine components such as fan blades [4, 5].

A variety of materials from ceramics to metal have been proved to have superplasticity after proper pretreatment [6]. For monotonous materials, different material generally requires different optimum pretreatment procedure to obtain maximum superplasticity. However, for laminate materials, this could not be true. It is possible that different laminates use same superplastic pretreatment procedure to obtain a proper superplasticity if they have the same superplastic component. Sherby *et al.* [7] reported that non-superplastic (α-iron) could behave superplastically after bonding with superplastic high carbon steel into a laminate structure. Under iso-strain assumption, theoretical models on strain rate sensitivity m and active energy Q between the laminate and its constituent components were established by them [7]. With the models, superplastic component volume fraction of laminate has a positive effect on the strain rate sensitivity of the laminate, so a higher elongation rate could be expected for the laminate with higher superplastic component volume fraction. However, the effect of layer number on superplasticity of laminates is not clear from previous research work. The objective of this work is to investigate the effect in laminate 7475/2091 Al alloys.

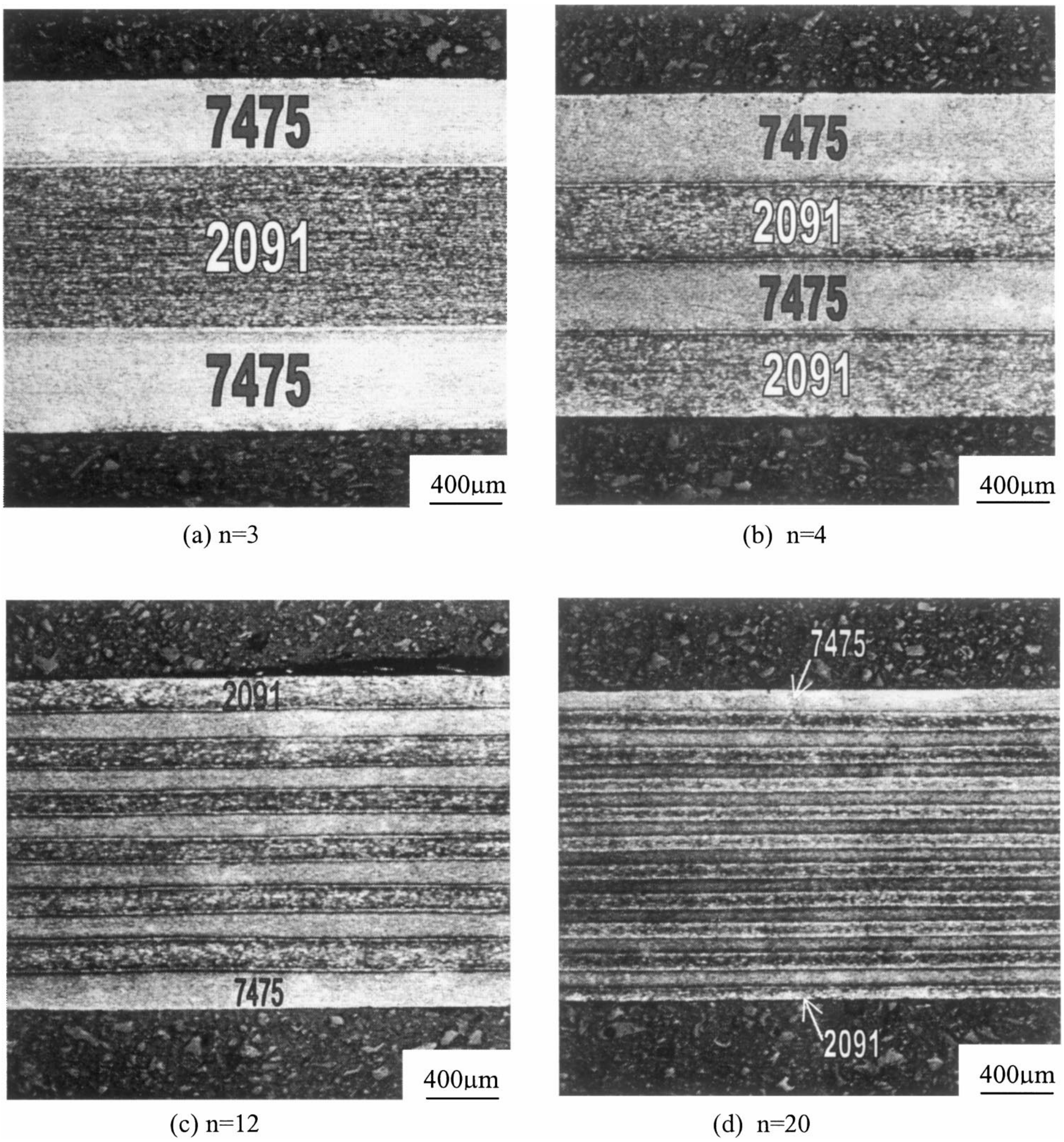
2. Experimental

The compositions of 2091 and 7475 Al alloys used in this study are shown in Table I. The original thickness of 2091 and 7475 Al alloys plates is 10 mm. By hot rolling and cold rolling, the plates were reduced to sheets with different thickness. These sheets of 2091 and 7475 Al alloy were fully recrystalized at 530 °C and 490 °C respectively for one hour. Three steps surface cleaning were used. First, the sheets were cleaned with an alkali water solution at concentration of 15%; then they were cleaned with water; after drying, their surface was finally cleaned with steel brush. The clean sheets of 2091 and 7475 Al alloy were then alternatively stacked into sandwich piles of 12 mm in thickness. After holding 15 mins at 400 °C, each sandwich pile was isothermal pressed into a laminate of 8 mm in thickness to obtain metallurgical bonding between different constituents. With above procedures, 7 kinds of laminates with different layer number i.e. 2, 3, 4, 5, 10, 19 and 20 were produced. The volume fraction of 7475 component in each laminate is about 50%, see Fig. 1.

A simplified pre-treatment procedure of 7475 Al alloy [8] were used to treat the 7475/2091 laminates, which consists of homogenizing, warm rolling,

TABLE I Chemical compositions of 7475 and 2091 Al alloy in weight percent

Al alloy	Zn	Li	Mg	Cu	Zr	Cr	Fe	Si	Ti	Al
7475	5.96	no	2.25	0.06	no	0.22	0.064	0.28	0.06	Bal.
2091	no	2.2	1.2	2.6	0.15	no	0.1	0.1	no	Bal.



(a) n=3 (b) n=4

(c) n=12 (d) n=20

Figure 1 Laminates 7475/2091 with a layer number *n* and volume fraction 50% (a) $n = 3$, (b) $n = 4$, (c) $n = 12$, (d) $n = 20$.

recrystalization and two stage artificial aging. The homogenizing was at 490 °C, warm rolling started at a temperature of 410 °C and ended at a temperature about 150 °C. By warm rolling, the 8 mm thick laminate plates were reduced into 1.2 mm thick sheets with total reduction rate of 85%.

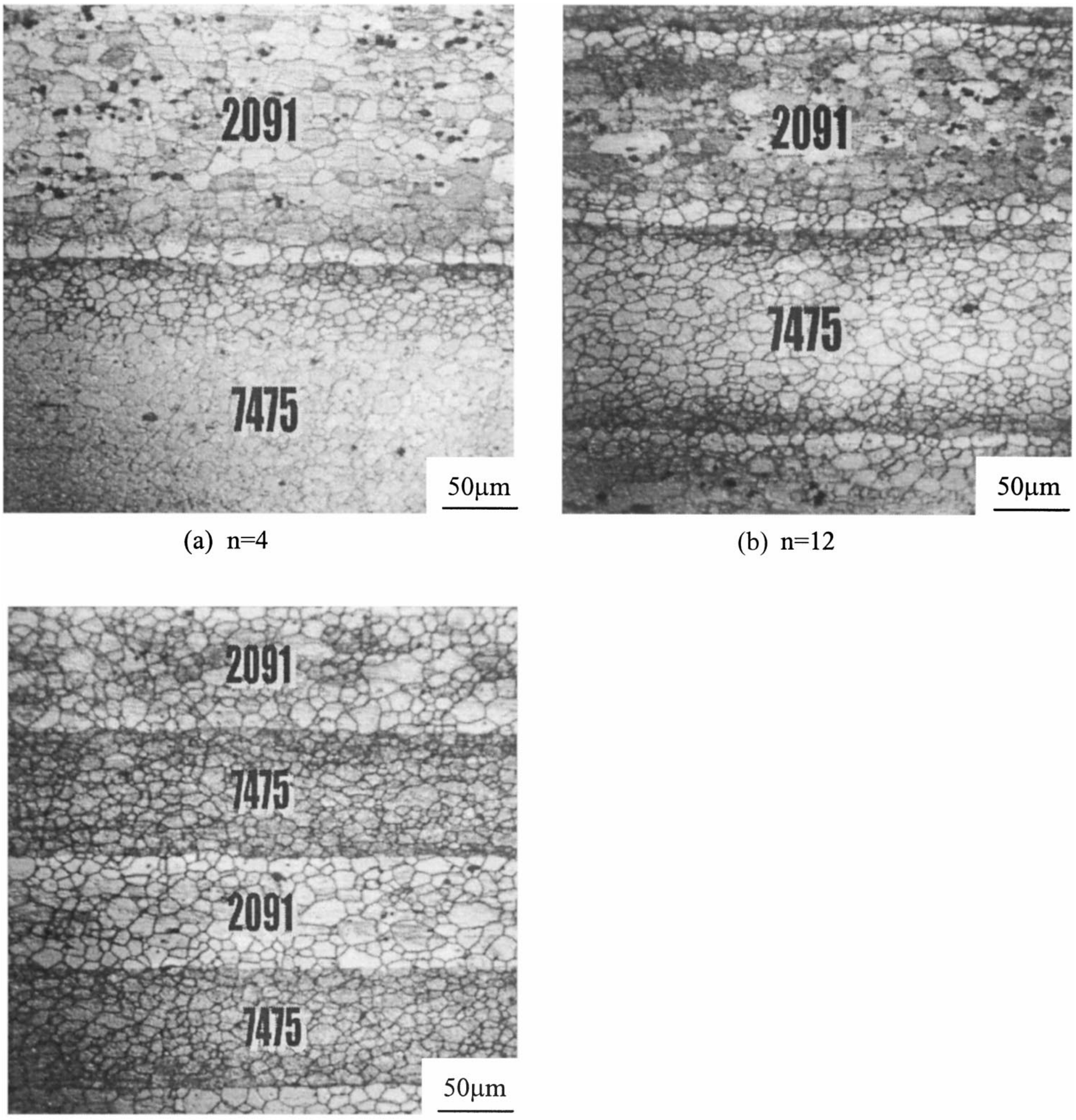
Superplasticity test was done on AG-100 electronic tensile test machine. Deformation temperature was in the range of 470–540 °C. The initial strain rate was 8.33×10^{-4}, 1.66×10^{-3}, 3.32×10^{-3} and 8.33×10^{-3} s^{-1} respectively. Keller reagent was used to etch microstructure.

3. Experiment results and discussions

3.1. Bonding interface

After bonding and superplastic pre-heat-treatment of the laminates, the metallurgical bonding at interface was obtained, see Fig. 2. Some combined grains consisting of two components of the laminate were observed at bonding interface. Scanning electron microscope (SEM) observations show that some impurity intermetallic phases with the size of 0.5–2 micron existed at bonding interface, see Fig. 3. The size and the volume fraction of the intermetallic phases at interface do not show much difference from that in inner part of 2091 component. After etching, small intermetallic phases could loss from matrix and result in etching pits, see Fig. 4. Energy dispersive X-ray spectrum (EDX) analysis do not shown obvious difference in chemical composition between most intermetallic phases at bonding interface and these intermetallic phases inside each component of the laminate, see Fig. 5.

During superplastic pre-heat-treatment, the laminates have been warm and cold rolled with total



(a) n=4

(b) n=12

(c) n=20

Figure 2 The microstructure of the laminates of 7475/2091 with layer number n and volume fraction 50% (a) $n = 4$, (b) $n = 12$, (c) $n = 20$.

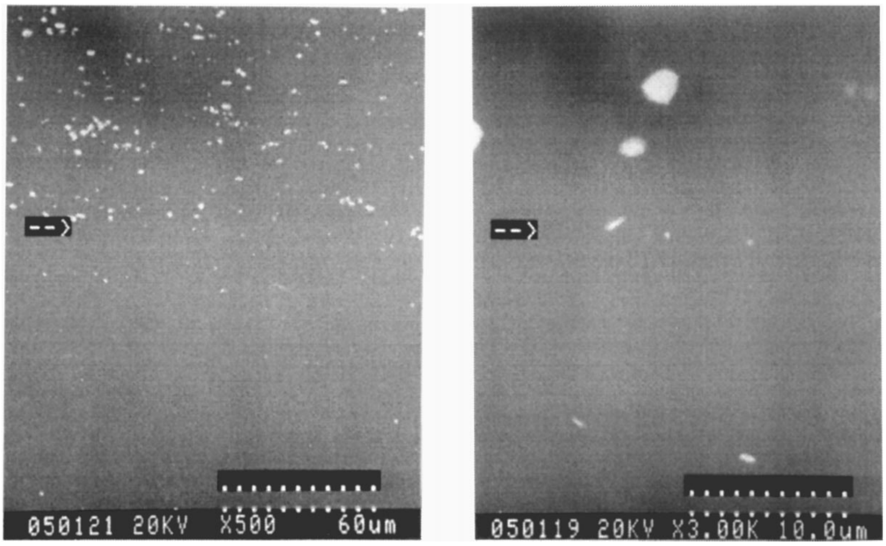


Figure 3 Back scattered electron (BSE) images of the polished longitudinal section of the laminate with layer number 5. The arrows show bonding interface position. The upper part above the arrow is 2091 component and the lower part below the arrow is 7475 Al alloy component.

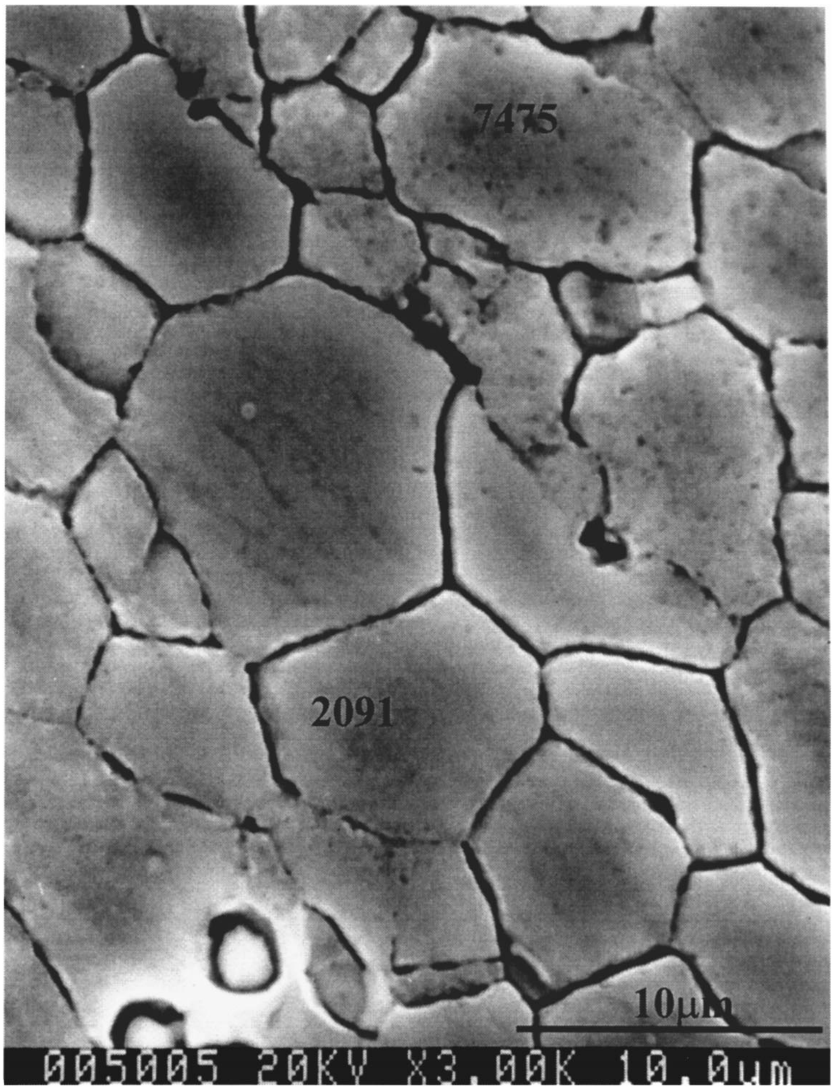


Figure 4 SEM image to show the bonding interface and impurity intermetallic phases in the laminate with a layer number of 5.

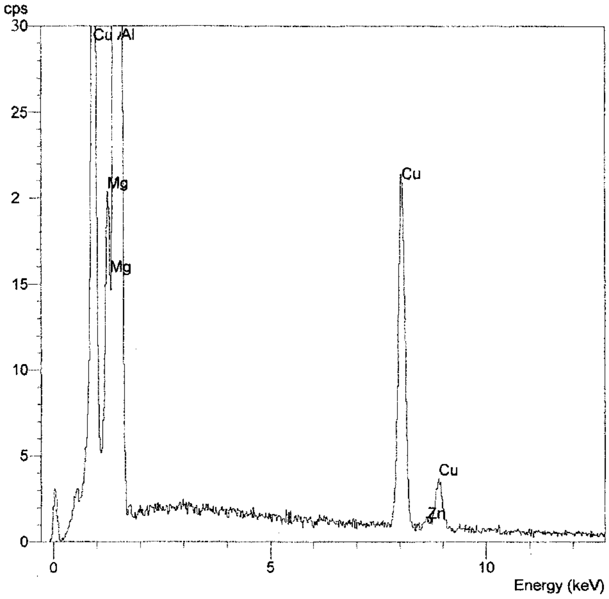
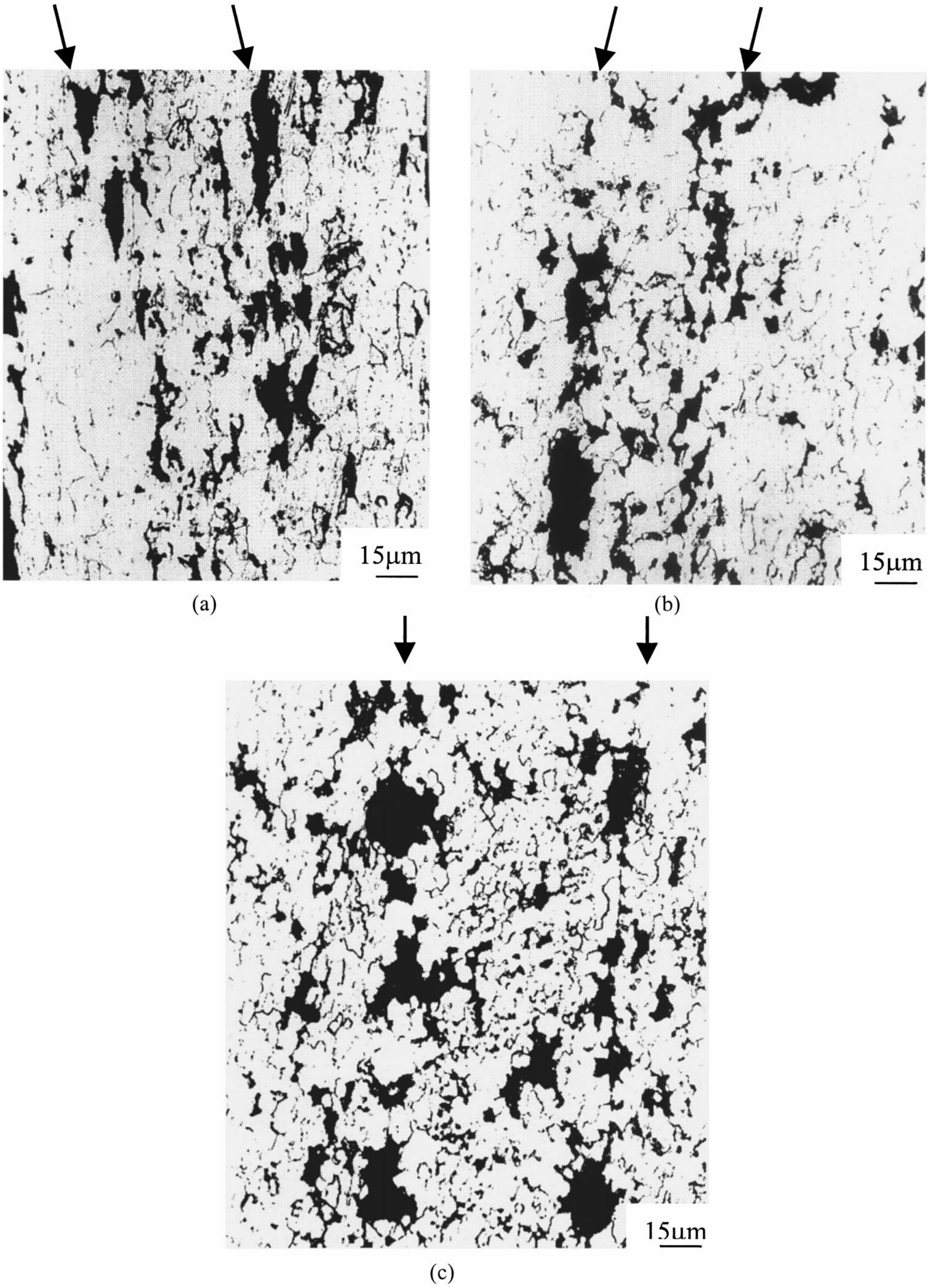


Figure 5 Energy disperse X-ray (EDX) spectrum of a intermetallic phase at bonding interface of laminate 7475/2091.

reduction rate of 85% and then held at 490 °C for 30 mins for recrystalisation. A fully recrystalised fine microstructure formed in 7475 Al alloys layers of all laminates, the average grain size is about 4.5 micron, see Fig. 2. For 2091 alloys, the full recrystalisation temperature is 530 °C. Partial recrystalised microstructure should form in 2091 component of the laminates. However, because of element diffusion between two components through bonding interface, fully recrystalised microstructure with average grain size about 9 micron was observed in 2091 Al alloy component in the laminate with layer number of 20, see Fig. 2c. For the laminates with layer number no more than 12, partial recrystalized microstructure was observed in inner part of each 2091 component and fully recrystalised microstructure with average grain size about 9 micron was observed near bonding interface, see Fig. 2a and b. By measuring the thickness of fully recrystalised part in each 2091 component in all laminates, supposing diffusion affected ranges in either side of bonding interface are same, the thickness of the diffusion effect zone at interface is roughly estimated to be about 50 micron.

3.2. Cavity evolution

Many research works have observed cavitation during superplastic deformation in 2091 and 7475 alloys [9]. The cavity nucleation, growth and linkage finally result in superplastic deformation failure. In monotonous materials, cavities are situated on grain boundaries and distribute uniformly in a specimen. However, in laminates,



(a) (b) (c)

Figure 6 Cavities in microstructure of laminate 7475/2091 with layer number 3 after superplastic tensile deformation at 510 °C and different initial strain rates. The middle layer is 2091 and outside layers are 7475. The arrow in the diagram show bonding interface positions, (a) at an initial strain rate of 8.33×10^{-4} s^{-1}, (b) at an initial strain rate of 1.67×10^{-3} s^{-1}, (c) at an initial strain rate of 4.01×10^{-3} s^{-1}.

there are bonding interfaces in their microstructure, the cavity evolution is different from that in each individual component alloy. Cavitation in the laminate of 7475/2091 after superplastic tensile deformation is shown in Fig. 6. Most cavities were observed in 2091 component rather than 7475 component of the laminate. They are situated mainly on grain boundaries and especially much more in bonding interface region. The cavity size on bonding interface is much larger than one in inner part of 2091, which result from cavity nucleation and growth primarily on bonding interface during tensile deformation. With increase of strain rate, the more cavities formed inside 2091 component and their morphologies tend to be more equal-axis, see Fig. 6. At a lower strain rate, 8.33×10^{-4} s^{-1}, corresponding to optimum superplastic deformation condition, the cavities have an elongated morphology, they grew and connected mainly along tensile axis parallel to the elongation direction during superplastic deformation, see Fig. 6a. At a higher strain rate,

3.32×10^{-3} s^{-1}, the cavity growth and connection take place both in transverse and longitudinal direction of the specimen, the cavities have a more equa-axised morphology, which was often observed in superplastic Al alloys [10, 14–18].

The cavity nucleation induced by brittle intermetallic phase precipitation was reported in a laminate consisting of stainless steel 26Cr-1Mo and ultrahigh carbon steel UHCS-3Si [11]. In 2091/7475 laminate, intermetallic particles were observed on bonding interface. However, many coarse intermetallic particles were also observed in inner part of 2091component, as stated in prior section. It is therefore implied that in addition to the brittle intermetallic phase precipitation mechanism, other mechanisms should involve and result in cavitation in interface region of the laminates. Experimental observations shown that the cross section of a specimen laterally bent toward 7475 Al alloy side during superplastic tensile deformation in a laminate with an even layer number such as 2, 4 and 6 etc, schematically shown in Fig. 7. The bending was caused by the elongation ability difference between 2091 component and 7475 component. During tensile deformation, the 7475 component forced the connected 2091component to elongate in longitudinal direction and reduce its cross section area at a same rate. Hence, in longitudinal section of a tensile specimen, see Fig. 7b, an additional tensile stress concentrates at bonding interface in 2091 component and results in cavity nucleation there. In transverse section of the tensile specimen, see Fig. 7a, an additional compress stress concentrated at the 2091 component connecting with 7475 component, a nonuniform deformation in cross-section, e.g. bending, is caused.

Next possible mechanism of cavity nucleation may come from mismatch grain sliding. It is known that in addition to grain sliding, the dominant mechanism of superplastic deformation, other mechanisms such as diffusion creep and dislocation motion also operated during sperplastic deformation [8]. Each mechanism's contribution to total deformation depends on material, its microstructure, deformation temperature and deformation rate [12, 13]. In laminate of 7475/2091, there was obvious microstructure difference between 2091 and 7475 component, the contributions of each operating deformation mechanisms to total deformation in the two components could be different. The mismatch gain boundary sliding at bonding interface could also cause stress concentration and cavitation there.

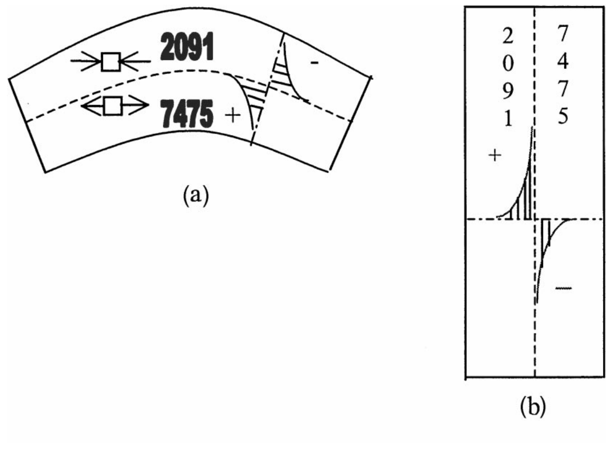


Figure 7 Schematic diagram of cross section bending and internal additional stress distribution in transverse section and longitudinal section of a two-layer laminate, (a) cross section bending after tensile deformation and additional stress distribution, (b) internal additional stress distribution in each component, the dash line represent bonding interface.

3.3. Superplastic elongation rate

For 7475/2091 laminates, the deformation parameters, such as superplastic deformation temperature and initial strain rate, have a strong influence on a superplastic parameter, the elongation rate. At an initial strain rate of 8.33×10^{-4} s^{-1}, the optimum superplastic deformation temperature of the laminates is about 510 °C, see Fig. 8. At temperature of 510 °C , the maximum elongation rate can be obtained at the strain rate of 8.33×10^{-4} s^{-1}, see Fig. 9. The effect of laminate layer

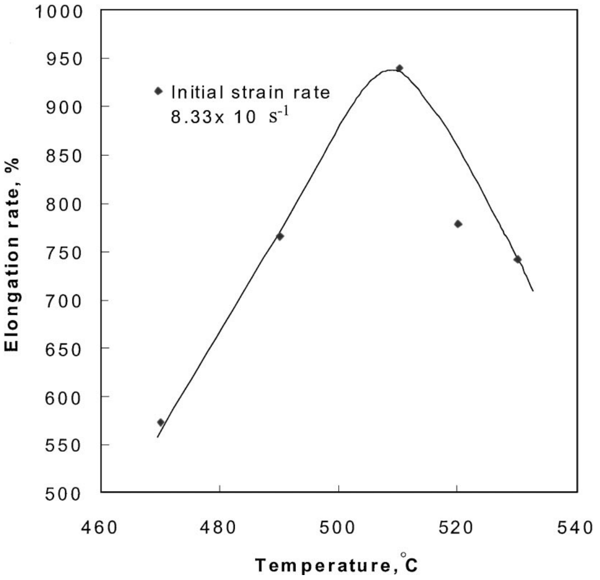


Figure 8 The effect of deformation temperature on the elongation rate in the laminate 7475/2091 with a layer number of 3 at an initial strain rate 8.33×10^{-4} s^{-1}.

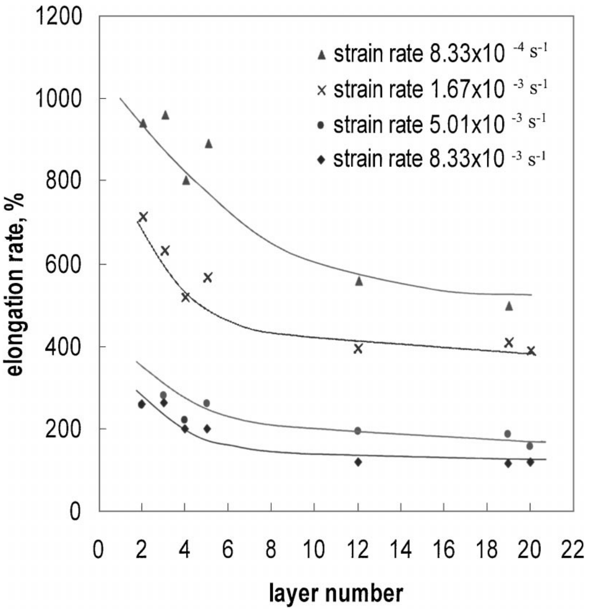


Figure 9 The effect of layer number on elongation rate in laminate 7475/2091 at deformation temperature 510°C and different initial strain rates.

number on the superplastic elongation rate of the laminates could also be observed in Fig. 9. With the increase of layer number, the elongation rate decreases. The decrease was rapid when layer number was relatively low and slow when the layer number was high, e.g. $n = 19$. This phenomenon can be understood with cavity growth models.

Cavity growth in superplastic materials may occur by diffusional growth processes or the power-law growth mechanism [14, 18]. Diffusional growth processes result in cavities with a round appearance whereas the power-law growth mechanism leads to cavities that tend to be elongated along the tensile axis. The diffusional growth is cavity spacing related growth mechanism. Experimental results [15–17] have shown that the diffusional cavity growth mechanism is dominant only when the cavities are small, typically less than 1 micron. Related with cavity growth processes, the total elongation of specimen η_Σ could be divided into three parts. The first, η_0, is the elongation before initial cavity nucleation. The second, η_D, is the elongation during initial cavity nucleation and growth to the size of 1 μm by diffusion. The third, η_f, is the elongation with respect to cavity growth from 1 μm to failure of elongation deformation by power law or superplastic diffusion. They have following relationship.

$$\eta_\Sigma = \eta_0 + \eta_D + \eta_f \tag{1}$$

According to the Beere' and Speight [18] model, the diffusional cavity growth rate is

$$\frac{dr}{d\varepsilon} = \frac{2\Omega\delta D_{gb}}{r^2 kT}\left(\frac{\sigma - 2\gamma/r}{\varepsilon}\right)\alpha \tag{2}$$

where r is the cavity radius, ε is the true strain, $dr/d\varepsilon$ is the cavity growth rate, Ω is the atomic volume, δ is the grain boundary width, D_{gb} is the grain-boundary diffusivity, k is Boltzman's constant, T is the absolute temperature, σ is the flow stress, γ is the surface energy, ε is the strain rate and α is the cavity size-spacing parameter. The cavity size-spacing parameter is given by the following expression:

$$\alpha = \frac{1}{4\ln(\lambda/2r) - [1 - (2r/\lambda)^2][3 - (2r/\lambda)^2]} \tag{3}$$

where λ is the inter-cavity spacing. In laminate 7475/2091, as stated before, cavity nucleates primarily on bonding interface. The inter-cavity spacing, λ, is related with layer number of the laminate, n, by the equation of $\lambda = \Delta h/n$, where Δh is thickness of the laminate. In Equation 2, cavity size spacing parameter α is directly proportion to cavity growth rate $dr/d\varepsilon$. In case of the cross-heads move at an constant velocity, the elongation corresponding to diffusional cavity growth, η_D, should be directly proportion to the time of cavity growing from nuclei to 1 micron in size. Hence, the elongation should be proportion to reciprocal of cavity growth rate as well as reciprocal of cavity spacing parameter. On the basis of Equation 3, the effect of layer number, n, on the reciprocal of the cavity size spacing parameter, $1/\alpha$, is shown in Fig. 10. The $1/\alpha$ decrease more rapidly when layer number is lower and slowly when layer number higher than 12. It is interested to note that the effect of layer number on $1/\alpha$ follows the same trend as the effect of layer number on elongation rate in the laminates, see Fig. 9.

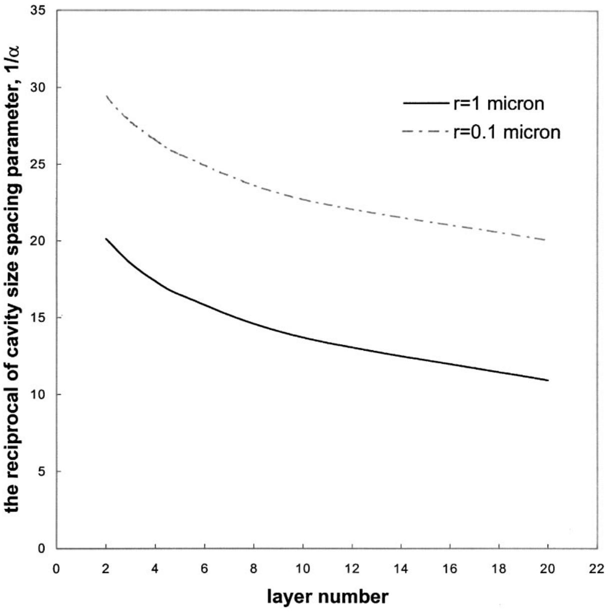


Figure 10 The effect of layer number on reciprocal of cavity size spacing paprameter.

When cavity size is more than 1 micron, the power law cavity growth or superplastic diffusional cavity growth will dominate the growth process, which is not cavity inter-spacing related and takes place in the direction of specimen elongation. The cavity growth rate can be calculated with following equation [9].

$$\frac{dr}{d\varepsilon} = r - 3\gamma/2\sigma \tag{4}$$

Supposing cavity growth along transverse direction to elongation is at a constant speed during elongating η_f, we get Equation 5,

$$\eta_f = C\frac{vh}{v_c n l_0} \tag{5}$$

where C is coefficient constant, h is the thickness of the laminates, n is layer number, v_c is cavity transverse growth velocity, v is cross head moving velocity, l_0 is initial specimen length. Equation 5 shows that layer number n has a negative effect on the elongation rate, η_f. On the basis of cavity growth models, above theoretical analysis results indicate that the cavity growth related elongation rate, η_D or η_f, closely depends on the laminate structure parameter, the layer number n. Cavitation during superplastic deformation in the laminate is an important factor that results in the effect of layer number on its elongation rate.

4. Conclusion

1. With the procedures used in the experiment, the laminates of 7475/2091 were successfully produced with

metallurgical bonding between 2091 Al alloy and 7475 Al alloy component. There is obvious microstructure difference between 2091 and 7475 component. The diffusion effect region near bonding interface of 50 micron in thickness was observed in microstructure of the laminates.

2. Cavitation takes place mainly in interface region during superplastic deformation, which is likely caused by the brittle intermetallic phases on bonding interface as well as deformation mismatch between two components.

3. The layer number is an important parameter affecting superplastic elongation rate of the laminates. Cavity growth models show that the cavity evolution in the laminate is an important factor resulting in the effect of layer number on superplastic elongation rate of the laminate.

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