SUPERPLASTICITY IN AEROSPACE MATERIALS

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ABSTRACT

Superplastic flow property and microstructural evolution of aerospace materials, with the examples of AA8090 Al-Li alloy and IN718 superalloy, are presented. Thermo-mechanical treatment of quasi-single phase material like this is used for producing fine grains required for superplasticity. In as-processed stage the microstructures are noted to have banded structure, which changes to equiaxed grains with concurrent grain growth and cavitation. Under this state, the flow stress varies with strain but the pseudo-steady state is attained after suitable prestraining at elevated temperatures. The maximum values of strain rate sensitivity index for deformation of AA8090 and IN718 superalloy are determined to be 0.82 and 0.70, respectively, with the corresponding values of ductility to be 475% and 579%. The activation energy for deformation suggest the role of grain boundary diffusion as an accommodation process for grain boundary sliding but, in the case of IN718 superalloy, grain boundary sliding is accommodated by lattice diffusion above a critical temperature.

Keywords: Superplasticity, Al-Li alloys, Superalloy, Thermomechanical Treatment, Constitutive Relationship, Superplastic Forming

1. Introduction

Important consideration for aerospace materials is light weight and high strength, besides the other mechanical and environmental properties. Metals constitute to be the major materials with approximately 68% aluminum alloys, 9% steel, 4% titanium alloys for typical A340-300 aircraft [1]. The proportions of these metals vary for other aircrafts but nevertheless they remain the dominant materials, with fiber reinforced composites being only 15-20% and other miscellaneous materials being around 2%. While aluminum is ductile it does not have good strength. The knowledge of physical and mechanical metallurgy is then exploited to improve substantially upon this limitation, by making use of strengthening by precipitates and other means. Gaining in strength invariably leads to a loss of ductility, which introduces another kind of limitation viz. the formability of such material is now reduced much. However, thermo-mechanical processing leads to fine stable grain size in some of these materials to make them superplastic. Superplasticity is the ability of certain materials to exhibit exceptionally large tensile ductility of several hundred percent at elevated temperatures, above half (0.5 T_m) the absolute melting point, and at intermediate strain rates of nearly 10^{-5} to 10^{-2} s^{-1}. Thus 2XXX and 7XXX series aluminum alloys of high strength and used in aircrafts can also be formed superplastically. This process leads to unique advantages of reductions in weight of material and its manufacturing cost, owing to the ease in forming like glass blowing. Other advantages include design flexibility, reproducibility with fine details and dimensional tolerance etc. However, one gets concerned with the lower rate of productivity due to the strain rate limitation and high temperature related problems. Superplastic forming is used to produce a variety of components and it turns out to be the only way out for hard-to-work materials. Superplastic forming is a welcome option in manufacturing the high specific strength (ratio of yield strength to density of material) components. This has led to the development of thermo-mechanical processing of the promising existing materials and also to emergence of new materials, which could not have come into existence in the absence of the possibility of superplastic forming. The successful attempts in the direction of producing superplastic materials include Al-Li alloys, Mg-alloys, superalloys, Ti-alloys and many more materials as reviewed elsewhere [2,3].

Study of superplasticity involves: (i) production of fine grains, usually less than 10 μm grain size in metals, (ii) characterization of superplasticity and finally (iii) superplastic forming. However, the characterization of superplasticity appears to dominate the superplastic literature. Development of fine grains by thermo-mechanical processing is seen more in quasi-single phase materials that have

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potential for extending the use of existing or newly designed alloys. Superplastic forming is mostly reported in the materials which have got industrial applications or in the model materials that are easy to form and get useful data for understanding the process. All these aspects of superplasticity will be examined and reviewed with reference to the aerospace materials in the present paper.

2. Development of Fine Grains

Wert [4] showed in 7075 Al-alloy that thermo-mechanical treatment (TMT) of precipitating system can give rise to fine grains for superplasticity. Subsequent to this work, fine grains were obtained in several other materials using the same principle or some variations thereof. The TMT for this system consists of (a) solution treatment, (b) overaging, (c) warm rolling, and (d) recrystallization. The objectives of each step are as follows.

Solution treatment gives uniform starting condition by dissolving the soluble precipitates. However, insoluble dispersoids of ~0.1 μm size remain in the material. Overaging creates precipitates of large size of about 0.75 μm that act as nucleation sites for recrystallization. Warm rolling develops intense dislocation structure around the non-deformable hard particles, extending substantially from the interface into the matrix. In some cases, cold rolling is employed to generate high dislocation density but the cell structure development gets delayed. In recrystallization, the dislocation structure is replaced by dislocation free new grains, whose size can be restricted by reducing the annealing time. Upon longer exposure to recrystallization temperature the new grains occupy the whole matrix, extending from the particle/matrix interface. The microstructures developed in certain category of materials are stable against static annealing. Instead of static recrystallization such materials are subjected to dynamic recrystallization for getting the required grain size prior to superplastic deformation.

Some of the thermo-mechanical treatments applied to get fine grain sizes in aerospace materials for superplasticity are listed in Table 1.

Recently IN718 superalloy, which is known to be important for high temperature aerospace turbine components and space shuttle applications, has been developed into superplastic grade by suitable thermo-mechanical processing. The widely used aerospace material Ti-6Al-4V and its modifications to lower the superplastic temperature are two-phase materials. The desired fine grained and equiaxed microstructures in these materials are obtained in a simpler way, as common to other two-phase materials, by mechanical working and annealing, with no consideration to precipitation used for quasi-single phase materials.

Table 1: Some aerospace materials with their thermo-mechanical treatments for grain refinement and superplasticity

Material	TMT steps	grain size	Ref.
7075/ 7475 Al-alloy	Solution treatment: 482°C/3 h/water quench; overaging: 400°C/8 h/water quench; rolling: 200°C/90% reduction; recrystallization: 482°C/0.5 h/water quench	~10 μm	[4]
Al-3Cu-2Li-1Mg-0.2Zr	Solution treatment: 538°C/1 h/water quench; overaging: 400°C/136 h/water quench; deformation: 300°C/$\varepsilon = 1$; recrystallization: 400-525°C	-	[5]
Al-2.4Li-1.15Cu-0.67Mg-0.11Zr	Solution treatment: 520°C/4 h/water quench; cold rolling: 0, 5, 10%; overaging: 350-400°C/4-20 h/water quench; warm rolling: 260-300°C/85-90% reduction	0.5 μm	[6]

3. Superplastic Characteristics of Aerospace Materials

Superplastic characterization consists of evaluating the strain rate sensitivity index (m) from log(stress, σ)-log(strain rate, $\dot{\varepsilon}$) data obtained either by constant true strain rate or constant cross head speed, or differential strain rate type tests. The value of m for superplasticity must be ≥ 0.3. The stress-strain rate data obtained from these tests are plotted in double logarithmic scale and the slope represents the value of m. There exist a number of other relations for obtaining the values of m from the two consecutives strain rates and corresponding stress values [7] of differential strain rate tests. Tensile ductility to failure increases with increasing value of m, and the two are commonly reported as superplastic mechanical property of materials. In addition to this, the dependence of σ-$\dot{\varepsilon}$ behavior on test temperature (T, in K) and grain size (d) is known to follow the constitutive relationship of type:

$$\dot{\varepsilon} = \frac{AD_0Eb}{kT}\left(\frac{b}{d}\right)^p\left(\frac{\sigma}{E}\right)^n \exp\left(-\frac{Q}{RT}\right) \qquad (1)$$

Here $D_0 \exp\left(-\frac{Q}{RT}\right)$ is diffusion coefficient, and often it is grain boundary diffusion coefficient, E is Young's modulus, k is Boltzmann's constant, A is a mechanism and material dependent dimensionless constant, which is determined experimentally when the magnitudes of stress exponent, n (n=1/m), the exponent of inverse grain size, p, and the activation energy for deformation, Q, are determined from experimental results. In the section to follow these superplastic properties and the nature of stress-strain curves of some of the aerospace materials will be described.

3.1 Superplastic properties of AA8090 Al-Li alloy

AA8090 Al-Li alloy, subjected to thermo-mechanical treatment for superplasticity and annealed at 813 K for 5 min, exhibits banded structure as shown in figure 1 [8].

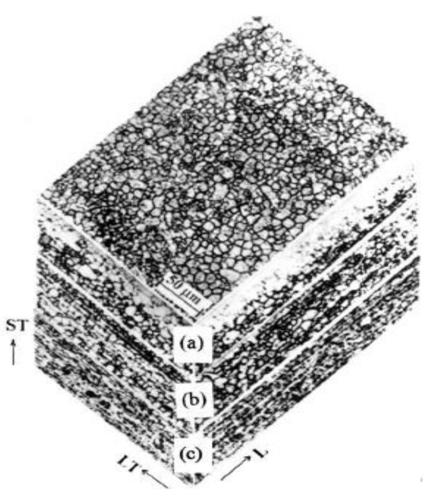


Fig 1 Microstructures of AA8090 Al-Li alloy in thermomechanical treated and short time annealed condition (813 K/5 min) [8]

This microstructure is seen to vary along the thickness of the sheet, with nearly equiaxed grains towards the surface and more elongated grains in the mid-thickness layer. These layers are highly textured with the dominance of (S):{123}<634> texture in the former layer and brass (Bs):{110}<112> in the later layer. Owing to such a structural variation the two layers exhibit different superplastic properties. There appear anisotropy in the flow property in both the layers as presented by the stress-strain curves for the tensile deformation of the center layer at the superplastic condition of $\dot{\varepsilon} = 1x10^{-3}s^{-1}$ and T = 803 K in figure 2 [9]. The values of m at this condition were found to be 0.82, 0.64 and 0.56 for the tensile specimens of full thickness, surface layer and center layer, respectively, with the corresponding values of tensile ductility as 475, 420 and 286%. The flow properties of surface and center layers suggest composite type contributions to the superplastic flow property of the full thickness sheet. During deformation the texture gets randomized and the microstructure changes to be equiaxed, leading to uniform structure and isotropic flow property at larger strains. There also occur grain growth and cavitation during superplastic deformation of this material.

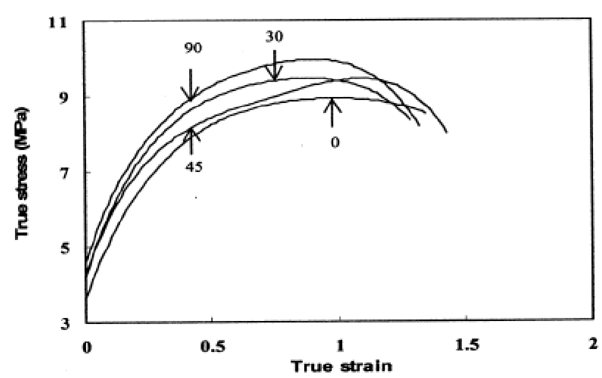
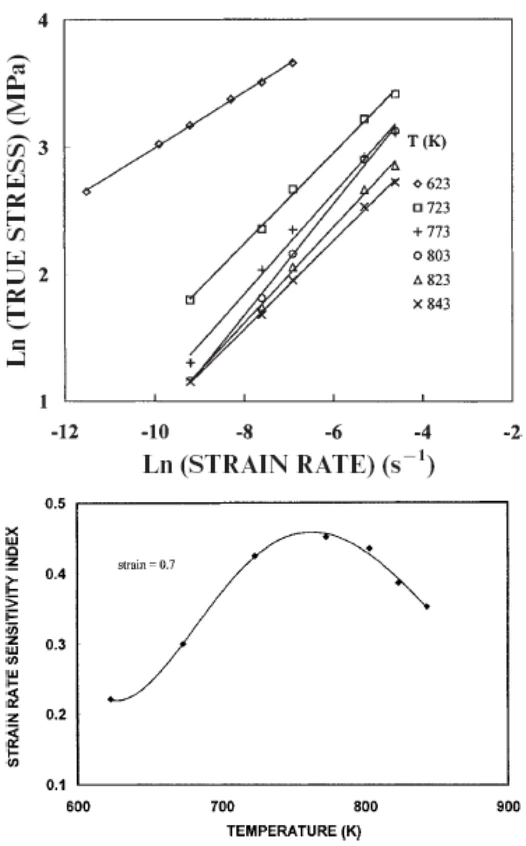


Fig 2 Stress-strain curves obtained from centre layer specimens having different orientation (degrees marked along the curves) with respect to rolling direction ($\dot{\varepsilon} = 1x10^{-3}s^{-1}$ and T = 803 K) [9]

The presence of unstable structure and the non-steady state condition of deformation in the beginning lead to no meaningful values of the parameters (m, p, Q) of the constitutive relationship, as they vary with strain. At larger strain, the stress-strain curves approach pseudo-steady state upon stabilization of structure. Under this condition, m is noted to be reasonably stable between 0.41 and 0.45 over the strains of 0.30 and 1.50 but it is distinctly dependent on temperature. This is illustrated by ln(σ) vs ln($\dot{\varepsilon}$) plot in figure 3(a) at various test temperatures and by the variation of m as a function of temperature in figure 3(b), where the maximum value of m = 0.45 is noted at T = 773 K [10]. Superplastic deformation of AA8090, in annealed condition (813 K/1h), also exhibited similar value of m (m = 0.43) with maximum ductility of 402%. The activation energy for superplastic deformation was found to be 93 kJ/mol. While the early part of superplastic deformation was dominated by dislocation based mechanism, as evident from the parameters of constitutive relationship and substructure examined, the later stage of deformation was similar to the widely known mechanism for superplastic deformation. This mechanism for superplastic deformation is dominated by grain boundary sliding and its accommodation by dislocation slip and/or diffusional process.



(a)

(b)

Fig 3 (a) Ln (stress) vs ln(strain rate) plots for AA 8090 Al-Li alloy at various test temperature at strain of 0.70; (b) Variation in strain rate sensitivity index as a function of test temperature [10]

3.2 Superplastic properties of IN718 superalloy

Superalloy IN718 was obtained in the form of superplastic grade sheet. In as-received condition, the microstructure is noted to be banded as shown in figure 4. Upon annealing at 1198 K, the microstructure changed into fine equiaxed grains whereas annealing at 1273 K led to a large grain growth, even within a short period of 15 min. Superplastic characterization was done by constant strain rate and differential strain rate tests of this material in both as-received and annealed conditions. The same are summarized below.

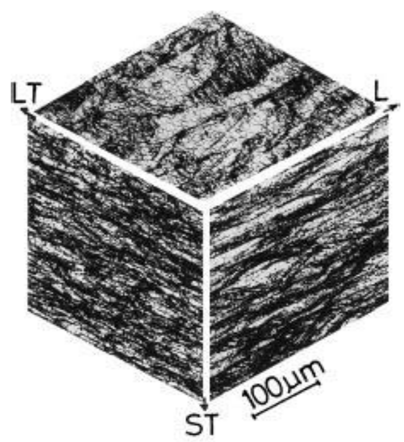


Fig 4 Initial microstructures of superplastic forming grade IN718 superalloy in as received condition

Log (stress)-log (strain rate) plot of the data, obtained by differential strain rate tests of as-received sheet over the strain rate range of ~$5x10^{-6}$ – $3x10^{-2}$ s^{-1} and the temperature range of 1173-1273 K, is presented in figure 5 [11]. The slopes of the curves, representing the strain rate sensitivity index, are large of superplastic characteristic with m = 0.62 at lower strain rates and higher temperatures. True constant strain rate tests were conducted over the strain rate range of $1x10^{-4}$ to $1x10^{-1}$ s^{-1} and the temperature range of 1173-1273 K. Stress-strain curves were found to exhibit flow hardening followed by flow softening and pseudo-steady state. Flow hardening is ascribed to grain growth, as revealed in figure 6(a), whereas flow softening is ascribed to the concurrent grain growth as shown by the micrograph in figure 6(b), under the superplastic condition of $\dot{\varepsilon}$ = $1x10^{-4}$ s^{-1} and T = 1248 K. Within superplastic region, grain growth and cavitation both were noted to increase with temperature. The maximum tensile ductility of 485% was obtained at the strain rate of $1x10^{-4}$ s^{-1} and T = 1198 K.

Tensile specimens annealed for 15 min to 5 h in the temperature range of 1198 to 1273 K, and having the grain sizes of 2.9 to 16.9 μm, were subjected to constant strain rate and differential strain rate tests. The condition for the former type of test was $\dot{\varepsilon}$ = $5x10^{-5}$ s^{-1} at T = 1198 K and for the latter the test temperature was the same but the strain rate ranged from $\dot{\varepsilon} \approx 2.5x10^{-6}$ to $3x10^{-2}$ s^{-1}. While the nature of the stress-strain curves, figure 7 [12], is similar to that noted in the as-received condition, viz. flow hardening in the beginning followed by flow softening and pseudo-steady state in some cases, both the strain rate sensitivity index and ductility were found to have improved for small grained samples. Under superplastic conditions the maximum values of the same were 0.7 and 579%, respectively.

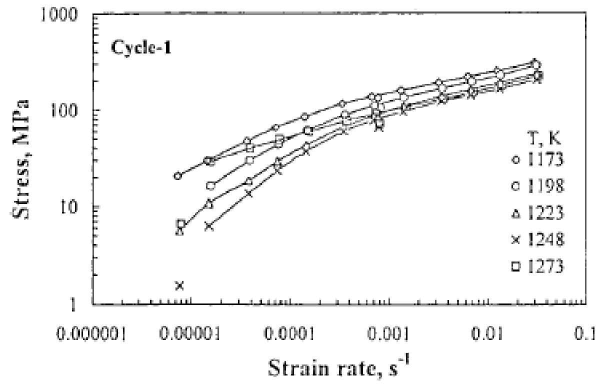


Fig 5 Log (stress) vs log (strain rate) plot for IN718 superalloy at various test temperatures [11]

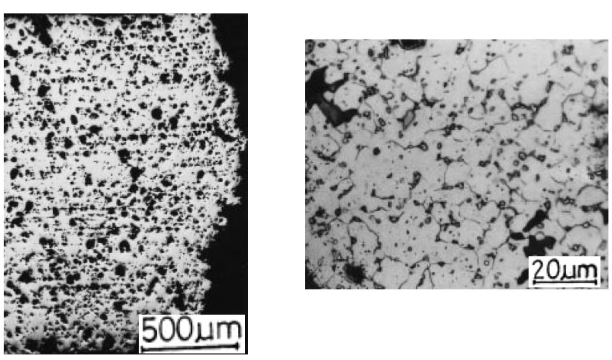


Fig 6 Cavitation (a) and grain growth (b) observed during superplastic deformation of IN718 superalloy ($\dot{\varepsilon}$ = 1x10^{-4} s^{-1} and T = 1248 K) [11]

As seen in figure 7, there appears upward trend in the stress-strain curves prior to failure in few cases. This increase in stress with strain can be understood by the micro-tensile type flow behavior of cavitating high strain rate sensitive superplastic material. The ligament between two large cavities behaves like a micro-tensile specimen, whereby the strain rate is raised to very high level for the same external strain rate, necessitating much higher flow stress ($\sigma \propto \dot{\varepsilon}^m$ and $\dot{\varepsilon} = \frac{\Delta L}{L}$, where ΔL is the change in gage length L) for deformation in the view of large value of m.

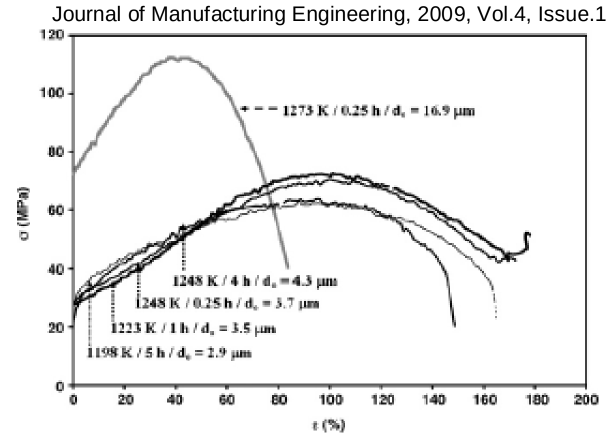


Fig 7 Stress-strain curves of annealed IN718 superalloy of different grain sizes ($\dot{\varepsilon}$ = 5x10^{-5} s^{-1} at T = 1198 K) [12]

In order to overcome the effect of non-steady flow and to evaluate the parameters of the constitutive relationship for steady-state condition of deformation, a tensile specimen was subjected to high temperature straining to true strain of 0.30 at $\dot{\varepsilon}$ = 1x10^{-4} s^{-1} and T = 1248 K. This pre-straining was followed by differential strain rate tests of the same single specimen over the strain rates ~ 1x10^{-5} to 3x10^{-4} s^{-1} and T = 1248 -1173 K, at intervals of ~ 10 K. The strain rate change was in an increasing sequence whereas the temperature change was in the decreasing sequence. The values of m were found to increase with temperature from 0.37 at 1173 K to 0.58 at 1248 K. The magnitudes of activation energy for superplastic deformation were determined to be 179.3 and 345.4 kJ/mol in the temperature ranges of 1173-1218 K and 1248-1248 K, respectively. These values of activation energy below the transition temperature of 1218 K (0.79 T_m, where T_m is absolute melting point) and above it are comparable with the activation energies for grain boundary and lattice diffusion, respectively. The mechanism for superplastic deformation is identified to be grain boundary sliding and its accommodation by diffusion or dislocation process. As a function of temperature, grain boundary diffusion is known to be dominant below ~ 0.8 T_m whereas closer to the melting point, i.e. above this critical temperature, the lattice diffusion dominates. Therefore, the mechanism for superplastic deformation is suggested to be grain boundary sliding and its accommodation by grain boundary diffusion below 1218 K and by lattice diffusion above this critical temperature.

3.3 Superplastic properties of other aerospace materials

Presented in this section, from the literature, is a summary of the superplastic characteristics of other aerospace materials, viz. other Al-alloys and Ti-alloys. The nature of stress-strain curves reported for the various materials exhibit flow hardening in the early part of deformation due to grain growth. While Al-alloys are susceptible to cavitation the Ti-alloys do not cavitate under optimum superplastic condition. Many of these materials possess elongated grains in as-processed condition, which change towards equiaxed grains with concurrent grain growth during the early part of deformation. The change from elongated grains to equiaxed grains causes flow softening whereas grain coarsening is responsible for flow hardening. In the presence of dispersoids in Al-alloys, there also occurs dislocation activity and a part of flow hardening arises by the conventional work hardening process. The presence of particles also becomes the source of stress concentration during deformation, which can lead to cavity nucleation. The development of cavities and randomization of texture in Al-alloys contribute to flow softening, but the flow hardening appears to be the dominant phenomena in many Al-alloys. Pseudo-steady state behavior in terms of a constant flow stress, with no variation in stress as a function of strain, can occur also by the balance between flow hardening and flow softening.

Superplastic characteristics of some Al- and Ti-alloys are listed in Table 2.

Table 2 Typical values of the parameters of the constitutive relationship and tensile ductility

Alloys	Constitutive parameters			% Elongation	Ref.
	m	Q (kJ/mol)	p		
IM Al 7475	0.67	141	-	2000	[13-14]
Al 7075	0.5	142	-	200	[15]
PM 8090 AL-Li	0.4	93	-	210	[5]
Ti-6Al-4V	0.6	282	2-3	650	[16]
Mg AZ31	0.5	124	2	580	[17]
Ti-10V-2Fe-3Al	0.33	133-157	-	1422	[18-19]

4. Superplastic Forming of AA8090 Al-Li Alloy

Pancholi and Kashyap [20] used the argon gas pressure bulging method for examining the superplastic formability of AA8090 aluminum sheets. The initial microstructure is shown in figure 1. In view of the variation in microstructure along the thickness direction, blanks of 1 mm thickness were machined to represent the surface, mid-thickness and composite layers, as also employed for tensile tests [9]. Three constant forming pressures were employed, viz., low, intermediate and high which corresponded to the initial strain rates of $4x10^{-4}$, $1x10^{-3}$ and $5x10^{-3}$ s^{-1}, respectively. The forming temperature was 803 K. The bulges thus produced are shown in figure 8.

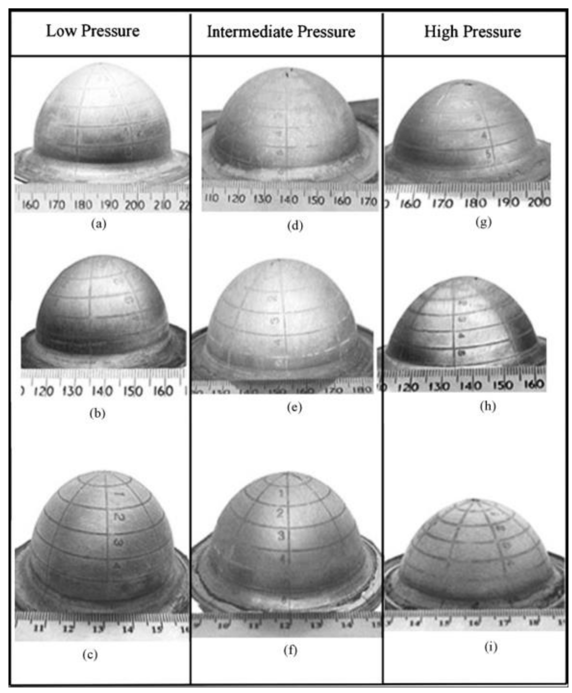


Fig 8 Argon gas bulging of surface (a, d, g), mid-thickness (b, e, h) and composite (c, f, i) layers at three pressure levels [20]

It is seen that the formability is superior for the surface layer material, formed at intermediate pressure, and it is most inferior for the mid-thickness layer. The reason for the best formability exhibited by the surface layer is the pre-existing nearly equiaxed grains that are necessary for superplasticity. The middle layer blank contained only elongated grains and so superplastic formability is very poor in this case. Since the composite layer contains composite type microstructure of elongated grains and equiaxed grains of equal sheet thickness layers within the 1 mm thick blank, the superplastic forming behavior also is noted to be intermediate between the other blanks.

5. Conclusions

Based on the work done in the authors' groups and the relevant literature on superplasticity of aerospace materials the following conclusions and comments can be drawn.

1. Commercial grade materials that undergo precipitation can be suitably thermo-mechanically processed to produce fine microstructures, which lead to fine equiaxed grains for superplasticity by static or dynamic recrystallization.

2. AA8090 Al-Li alloy exhibits microstructural and textural variation along the thickness direction of the sheet, with nearly equiaxed grains and texture (s): {123}<634> towards surface, and elongated grains and texture (Bs): {110}<112> in the mid-thickness region. The superplastic properties and superplastic formability of these layers are different but the two layers, when present together, contribute to composite like behavior in both flow properties and superplastic formability. With the progress in deformation the microstructure tends to be equiaxed and texture gets eliminated. Also, the initially low angle boundaries change into high angle grain boundaries to promote superplasticity by grain boundary sliding and its accommodation by grain boundary diffusion.

3. There occur flow hardening in the early part of deformation in many aerospace superplastic materials, which can be party attributed to grain growth but the other part is suggested to be due to conventional work hardening. During later stage of deformation, quasi-single phase alloys exhibit extensive cavitation but so is not the case in the two-phase Ti-6Al-4V alloy.

4. The varying stress with strain and changing microstructure result in a large variation in the magnitudes of the parameters of the constitutive relationship. However, suitable deformation at elevated temperature prior to superplastic deformation overcomes the non-steady flow state and then reasonable values of the parameters for superplastic deformation can be obtained.

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