Processing and characterization of aluminium alloys or composites exhibiting low-temperature or high-rate superplasticity

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Wide applications of superplastic forming still face several problems, one is the high temperature that promotes grain growth, another is the low forming rate that makes economically inefficient. The current study is intended to develop a series of fabrication and thermomechanical processing, so as to result in materials possessing either low temperature superplasicity (LTSP) or high rate superplasticity (HRSP). The former has been achieved in the cast Al alloys, while the latter was accomplished in powder-metallurgy aluminium matrix composites. The aluminium alloys, after special thermomechanical processes, exhibited LTSP from 300 to 450 °C with elongations varying from 300 to 700%. The LTSP sheets after 700% elongation at 350 °C still possessed fine grains 3.7 μm size and narrow surface solutedepletion zones 11 μm in whith, resulting in a post-SP T6 strength of 500 MPa, significantly higher than that of the HTSP superplasticity alloys tested at 525 °C or above. Meanwhile, it was found that LTSP materials may be transferred into HTSP materials simply by adding a preloading at 300-400 °C for a small amount of work. As for the endeavor in making HRSP materials, 2024Al/SiC, 6061Al/SiC and Al/Al_3Ti systems processed by powder metallurgy or mechanical alloying methods are under investigation. The average sizes of the reinforeing SiC or Al3Ti particles, as well as the grain size are all around 1 μm. The aluminium composites have exhibited HRSP at 525-620 °C and 10^{-2}-10^{1} s^{-1}, with elongations varying from 150 to 350%. This ultimate goal is to produce an alloy or composite exhibiting low temperature and high strain rate superplasticity (LT&HRSP).

INTRODUCTION

1. Superplasticity:

Superplasticity is referred to the capability of certain polycrystalline materials to undergo extensive elongation, often preventing from continuous necking, prior to failure when tested at appropriate temperature and strain rates.

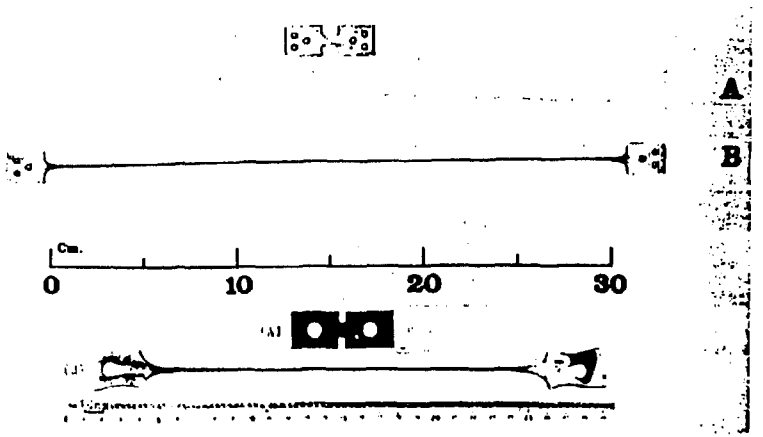

FIG. 1. A superplastic elongation of 4,850% is demonstrated in a Pb–62 wt% Sn alloy[10] by the two top samples and 5,500% is demonstrated by the two bottom samples in a commercial aluminum bronze.[10] The current world record is about 8,000% in the commercial aluminum bronze.[10]

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Records of superplasticity

Materials classification	Material	Elongation
Metals	Zn-Al	8800%
Metal matrix composites	6061Al/SiC	1400%
Intermetallic compounds	Super $\alpha 2$ Ti_3Al	1500%
Supercooled amorphous mater.	$La_{55}Al_{25}Ni_{20}$	15000%
Structure ceramics	Y-TZP	800%
High T_c ceramics	Y-Ba-Cu-O	110%
Ceramic matrix composites	Al_2O_3/Y-TZ	625%
High rate superplastic mater.	MA-Al	1250%

HISTORICAL DATA ON SUPERPLASTICITY

1912 Bengough, $\alpha + \beta$ Brass, approx. 200% elong.

1920 Resenheim et al., Zn-Al-Cu in near-eutectic composition

1928 Jenkins, Cd-Zn and Sn-Pb alloys, approx. 400% elong.

1934 Pearson, Pb-Sn and Bi-Sn eutectic alloys, 1950% elong.

1945 Sviderskaya, Zn-Al alloys, observed large elongations 'sverkhplastichnost' (ultra-high plasticity) was coined and led the term superplasticity became public

1962 Underwood, review paper, led to interest in Western world

1970 Commercial applications, non-structural

1974 Ti SPF applications, B-1 and Space Shuttle

1981 Guiness Book of Word Records, 4850% eong. in Sn-38Pb (Ahmed and Langdon, USC)

1985 Higashi, ~8000% elong. in Cu-Sn

1994 Langdon, ~8800% elong. in Zn-Al

GBS (Grain Boundary Sliding)

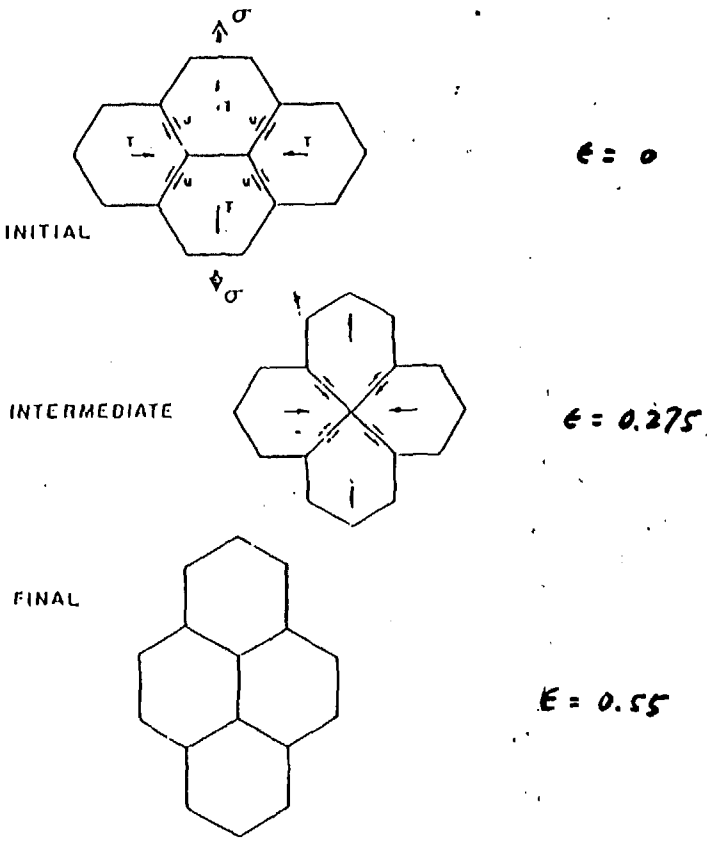


FIG. 2 A schematic illustration of the grain switching mechanism, involving grain boubdary sliding, proposed by Ashby and Verrall.

Types of Superplasticity

* Micrograin superplasticity
  - Fully (or statically) recrystallized
  - Continuously (or dynamically) recrystallized
  - Requirement:
    a. Fine and stable grain size (typically <10 μm)
    b. Temperature > $0.4T_m$
    c. Controlled strain rate
    d. Low flow stress
    e. Low tendency for cavitation
    f. High angle grain boundaries
    g. grain size distribution
    h. grain aspect ratio

* Transformation superplasticity

* Internal strain-induced superplasticity

formed Superform Metals Ltd and British Aluminium Company (now British Alcan Aluminium plc) began to produce the worlds first commercial superplastic aluminum alloy, Supral 100 (Registered Composition: Al, 6% Cu, 0.4% Zr; Designation: AA2004 produced per AMS4208). In 1974 Superform Metals Ltd introduced unique forming techniques and equipment (Reference 1) which enabled deep male formed parts to be produced. This male forming equipment is shown in Figure 2.

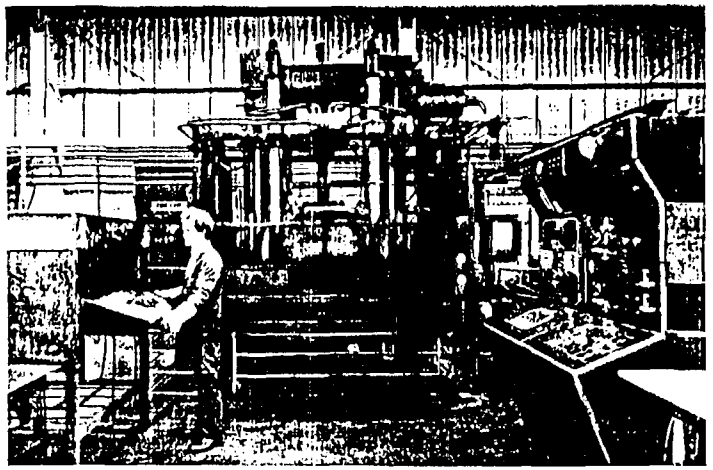


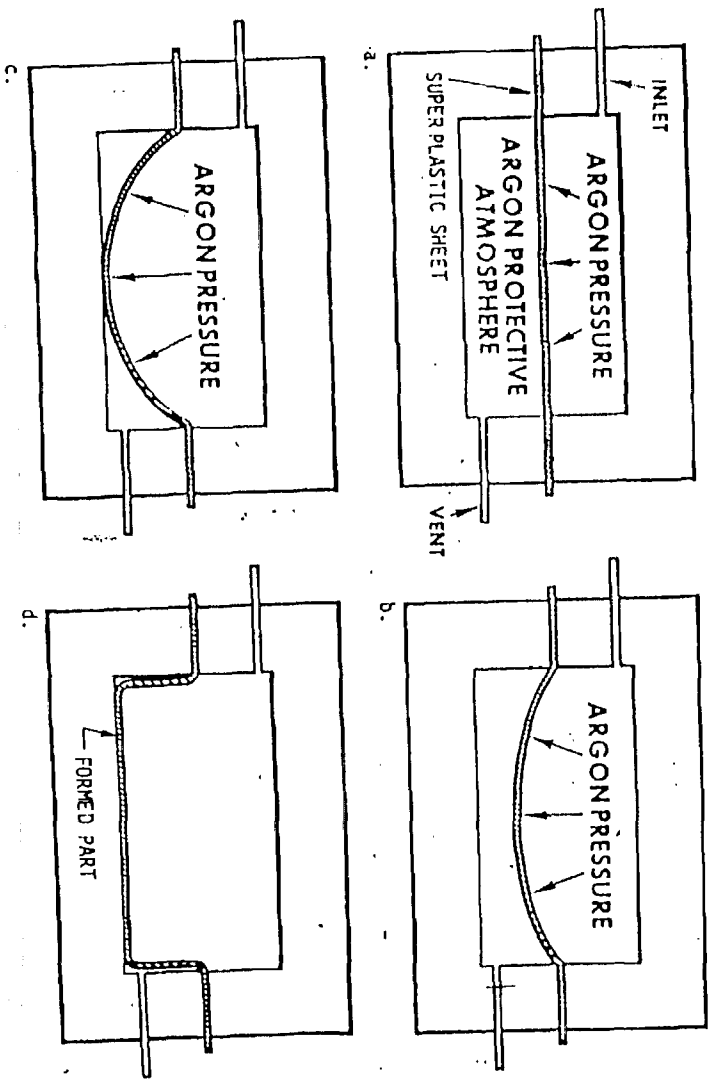
Figure 2 - Superform Male Forming Equipment

Detailed discussions of its operation has been covered elsewhere (Reference 2). The complete male forming process is illustrated by the progressive series of formings shown in Figure 3.

- complicated Shape
- One piece
- No string back
- low rate

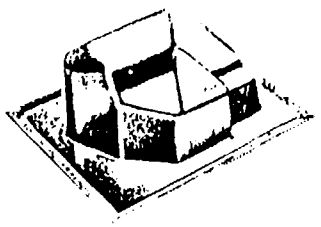
Figure 3 - The Complete Male Forming Cycle

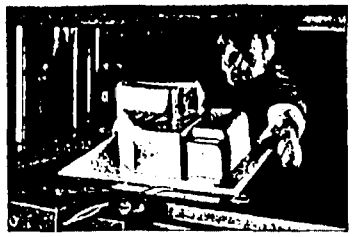
Fig 7-1



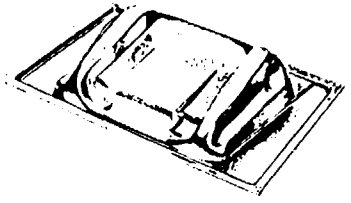
SC78-83

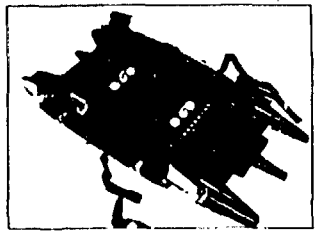
40% cost savings and improved dimensional accuracy compared with welded assemblies were achieved by the French Company, MESSIER HISPANO BUGATTI, by changing to SUPRAL. Four painted SUPRAL covers protect hydraulic pipework on the A300 Airbus undercarriage.



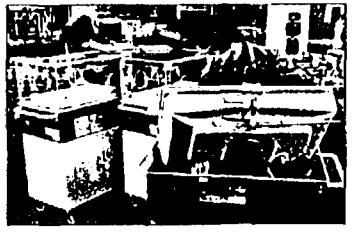


By selecting SUPRAL, M L. AVIATION achieved greater consistency and interchangeability on the fairings for the Alpha Jet Store Carrier. More complex one piece shapes reduced hand finishing and avoided the distortion which could occur when welding drop-hammered or rubber pressed parts.

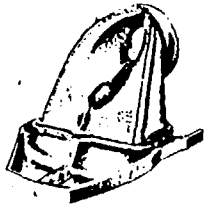




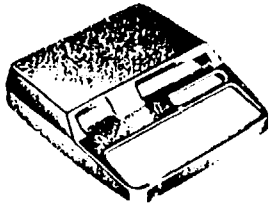
Increased life, simpler assembly and reduced costs were benefits gained by VICKERS MEDICAL when switching from glass fibre to SUPRAL inner bodies on infant incubator units.

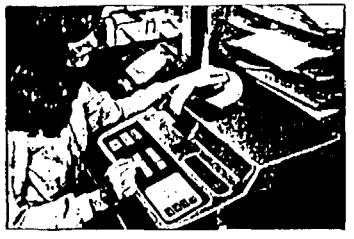


Short lead times and ease of assembly were benefits gained when WESTLAND HELICOPTERS designed a new configuration engine intake for an experimental programme. The 35 piece riveted and welded construction was formed from just 11 pressings which faithfully reproduced the master pattern.

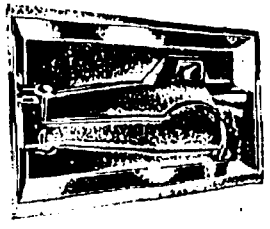


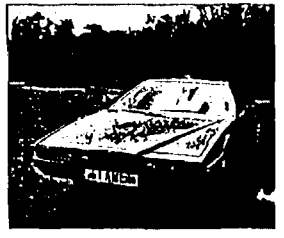
An attractive design strong enough to withstand harsh industrial environments. Screening against electrical noise and valuable heat sink properties were important benefits gained by SALTER INDUSTRIAL MEASUREMENTS on a weigh display unit.





SUPRAL panels form most of the external body work of the ASTON MARTIN LAGONDA giving distinctive lines and a reduction in hand finishing compared with rubber die pressed panels.





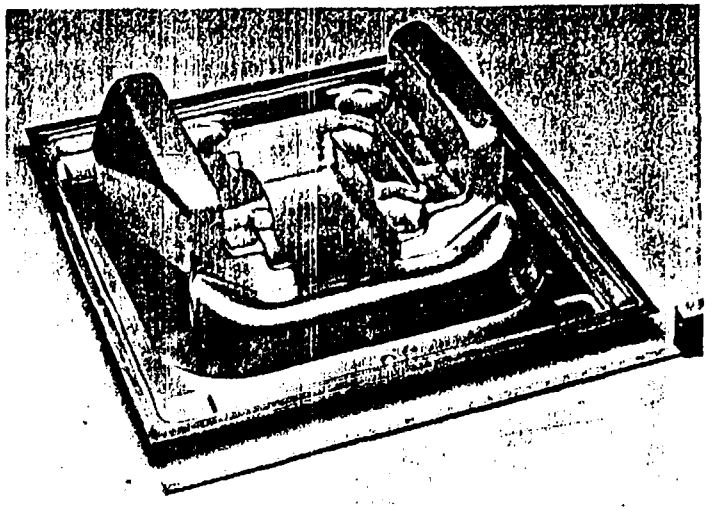


Figure 6 - Male Formed Ejector Seat Component Using Supral 100

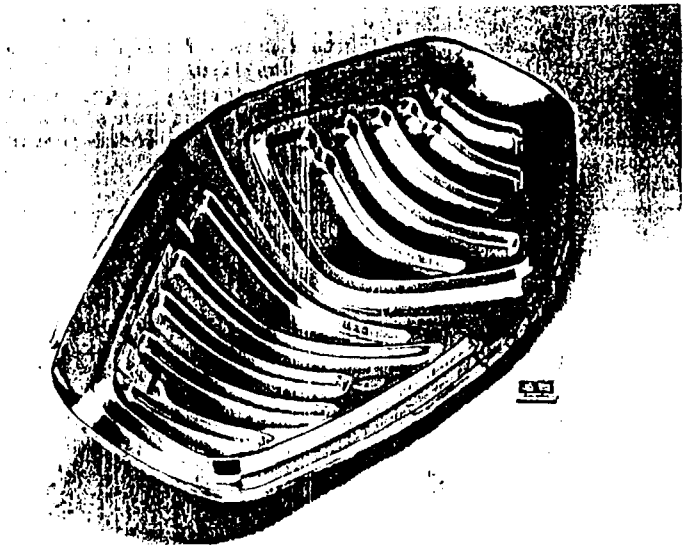
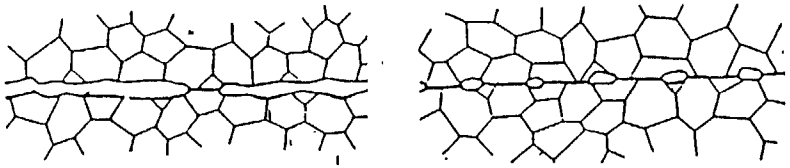


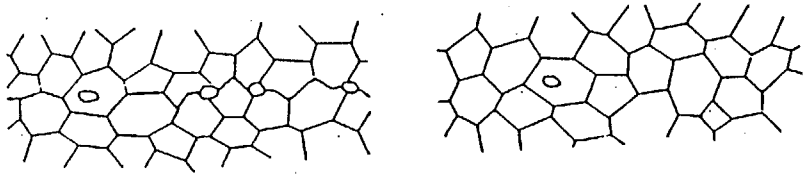
Figure 7 - Female Formed Pair of Helicopter Door Panels Using Supral 100

Diffusion Bonding



INITIAL:
ASPERITY CONTACT

FIRST STAGE:
DEFORMATION AND INTERFACIAL BOUNDARY FORMATION



SECOND STAGE:
GRAIN BOUNDARY MIGRATION AND PORE ELIMINATION

THIRD STAGE:
VOLUME DIFFUSION AND PORE ELIMINATION

INTERMETALLIC BLADE
(titanium-aluminum)

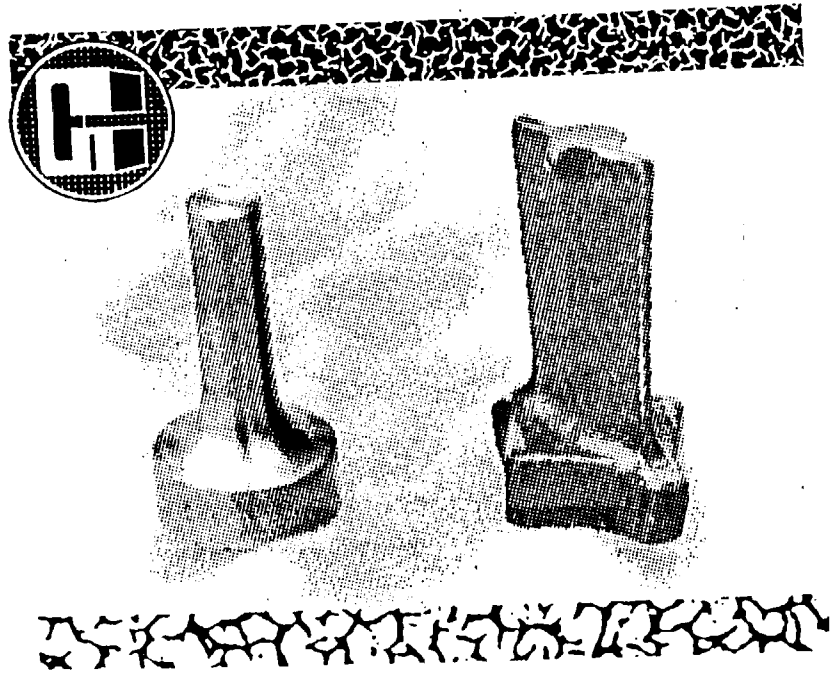
Blades are produced by superplastic forging out of the TiAl intermetallic alloy.

Mechanical properties:

yield point, MPa	600
relative elongation	2.5%
fatigue limit	320

Application:

Blades are used in aircraft engines. The wight of gas-turbine engines with TiAl blades used in compressors decreases by 10-15%.



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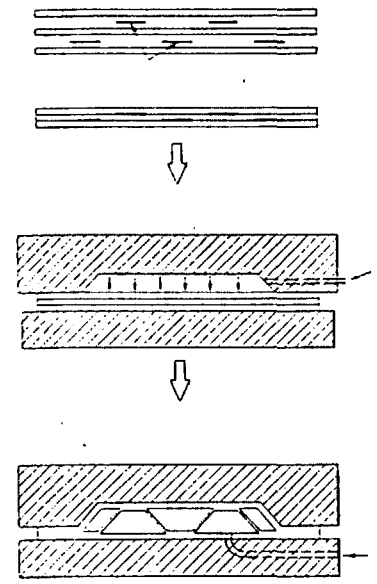

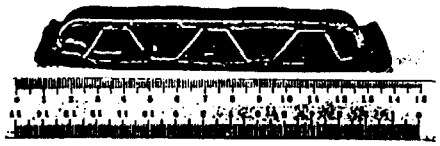

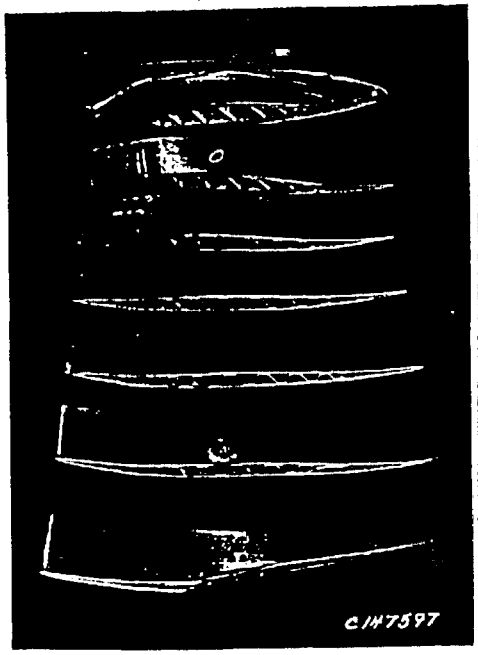

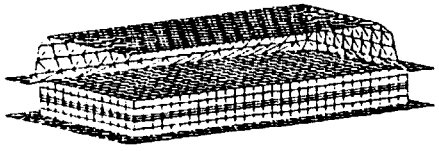

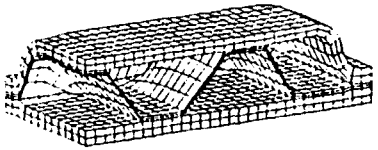

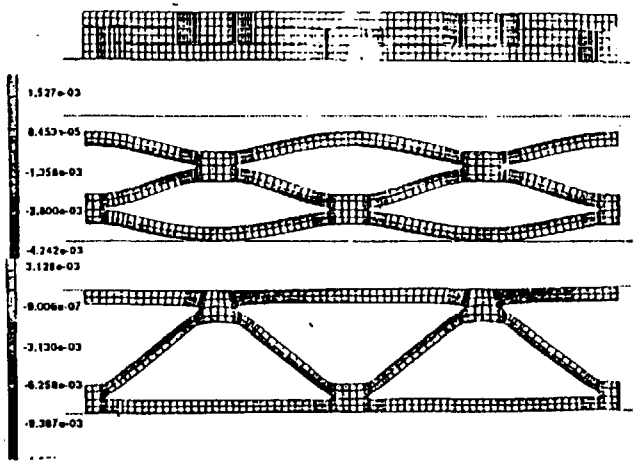

Three pieces

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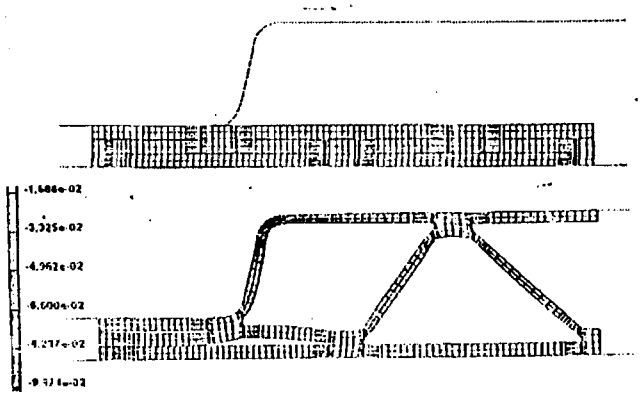

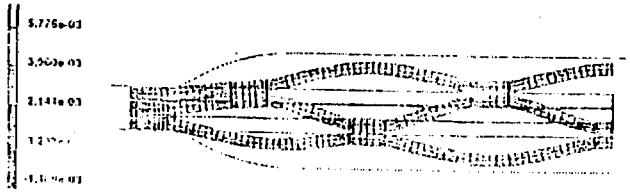

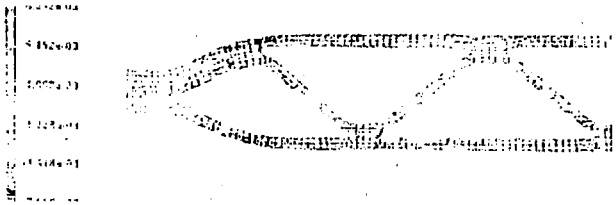

Subject Covered

* Superplastic (SP) Materials Development:
  (A) Aircraft-used and commercial aluminum alloys:
  (B) Advanced materials, including aluminum matrix composites, intermetallic compounds:
  Thermomechanical treatments (TMTs)
  Modified powder metallurgy
  Recrystallization routes
  High-T and low-T; low-rate and high-rate SP

* SP Tensile Tests:
  Constant crosshead speed and constant strain rate
  With or without back pressure
  Strain rate change

* Superplasticity Mechanism Analyses:

* Post-SP Characterization:
  Microstructure characterization
  Grain boundary sliding contribution
  Mechanical property evaluation

* Superplastic Forming (SPF):
  Forming system setup
  SPF practices
  Comparison between theories and experiments
  Post-SPF property evaluation

* Computer Simulation:

* Joining for SP Thin Sheets:
  Diffusion bonding
  Electron-beam and laser-beam welding

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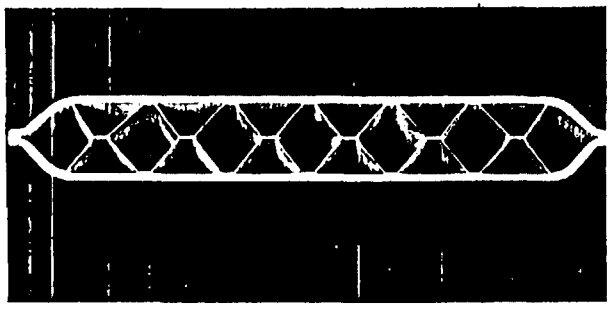
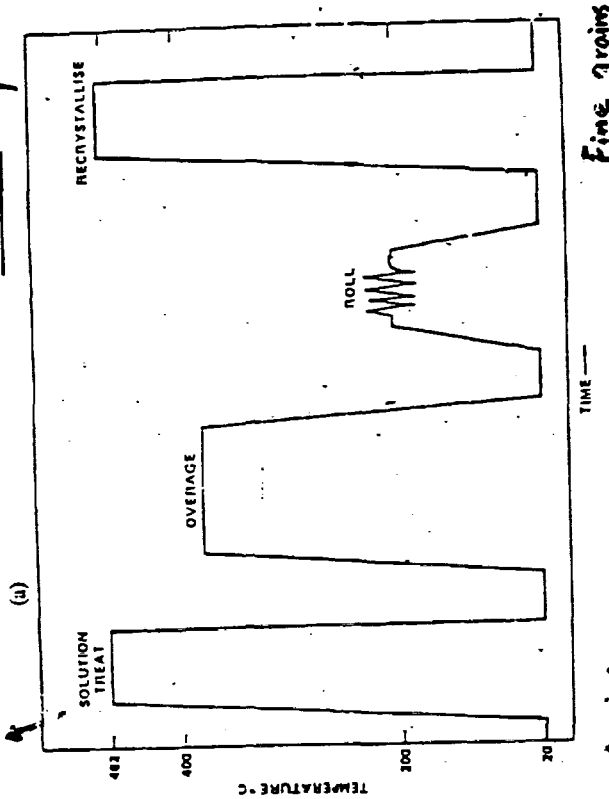


Figure 2 SPF/DB of titanium heat exchanger duct (Ref. 2).



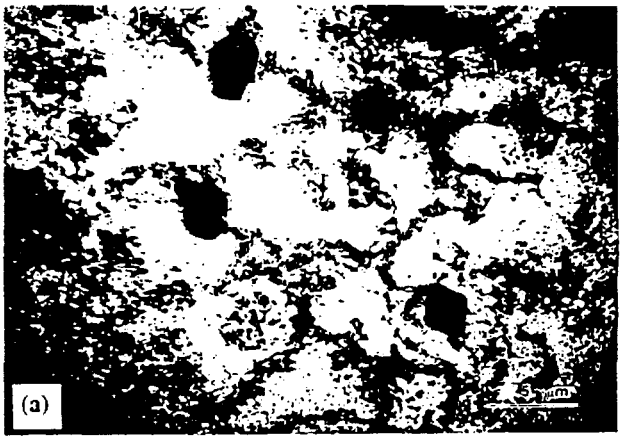
Fine grains

Coarse grains

Need:
① Large particles ~ 1-2 µm
to act as grain nucleation sites
② Small particles ~ 0.1 µm
to pin the grain boundaries

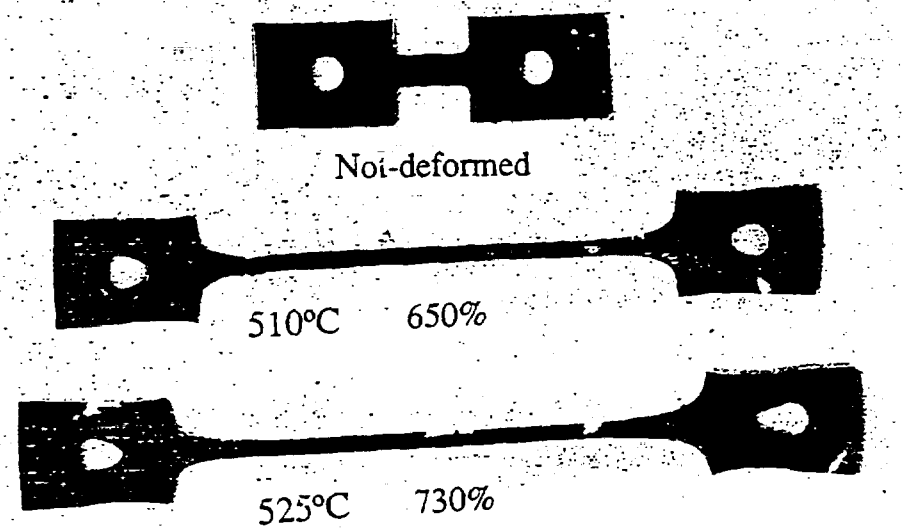
η ~ 2 µm

Cr-rich particles
~ 0.1 µm



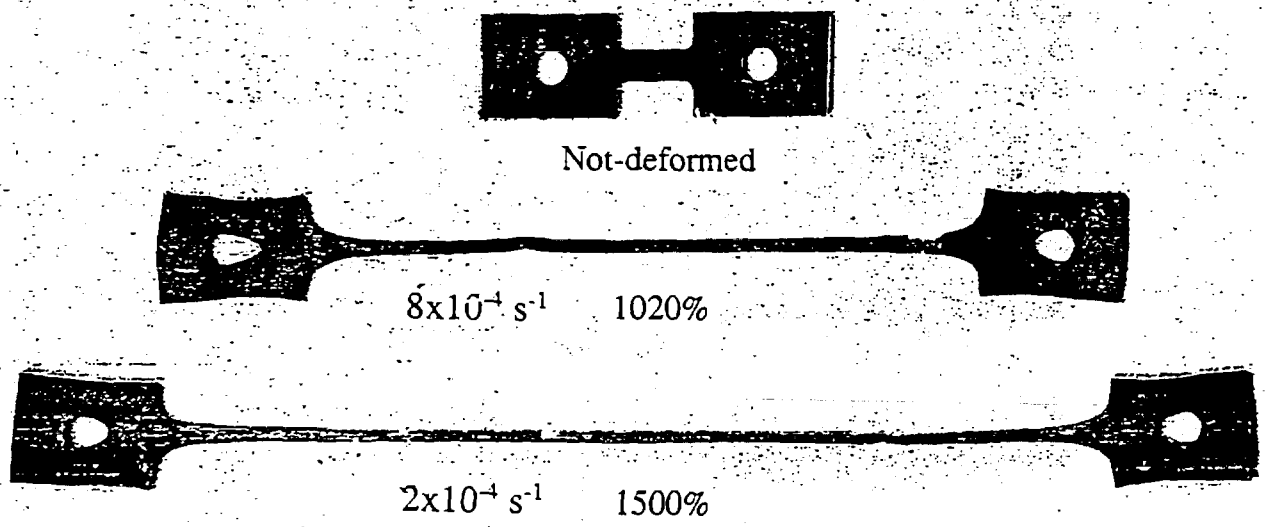
TEM micrographs of the precipitate and grain structure seen in the 7475 alloys: (a) large η precipates in the as-rolled samples (b)-(d) small and stable grains and the Cr-rich particles. The pinning effect can be clearly seen in (c) and (d).

7475 Route 1 (Reheat time~ 15 min)



Tensile elongations of 7475 route 1 for initial strain rate $2x10^{-4}$ s^{-1} at different temperatures

7475 Route 3 (Reheat time ~5 min)



Tensile elongations of 7475 route 3 at different initial strain rates 510°C

Fig. 1 Processing routes for producing the LTSP and HTSP thin sheets from a starting T3 thick plate.

The common working temperature for superplastic Al-Li alloys is

510-530 °C

(the high-temperature superplasticity, HTSP)

* **Surface Li- and/or Mg-depletion zone to 100-200 μm**
* **Severe grain growth from the initial 5 μm to final 20 μm**
* **Appreciable cavitation associated with concurrent grain growth**
* **Degraded post-form RT mechanical properties**

→ To develop

a superplastic Al-Li sheet with optimum working temperature of

350-450 °C

(the low-temperature superplasticity, LTSP)

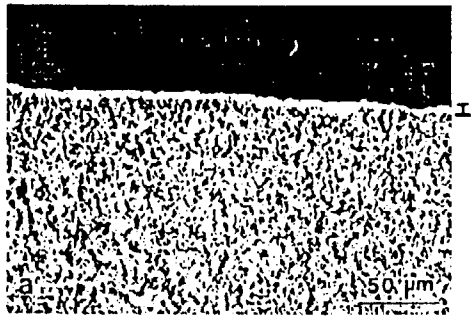
★ 5083 Al-Mg 6061 Al-Mg-Si

OM (in air)

~150%

SP elongation

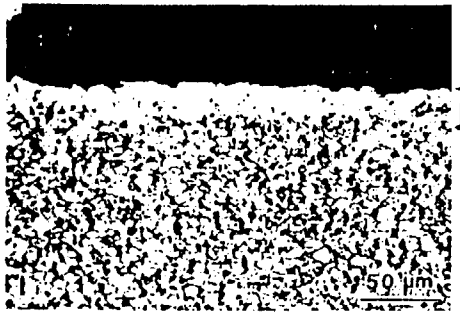
LTSP



53 μm

350°C

LTSP



43.9 μm

450°C

HTSP

90.4 μm

525°C

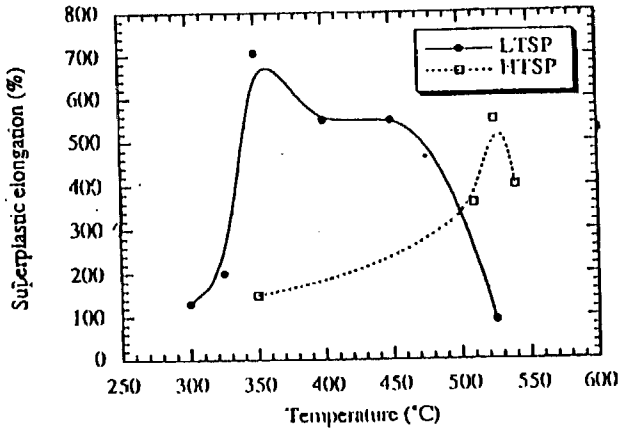
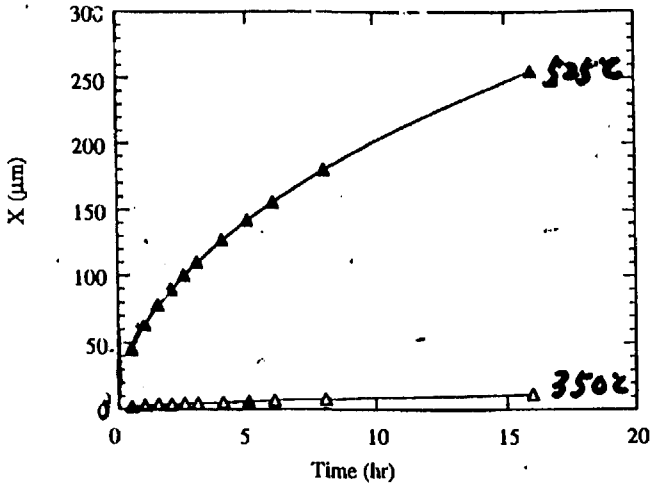


Fig. 1 The variation of superplastic elongations of the LTSP and HTSP sheets as a function of test temperature at an initial strain rate of $8 \times 10^{-4}\ s^{-1}$.

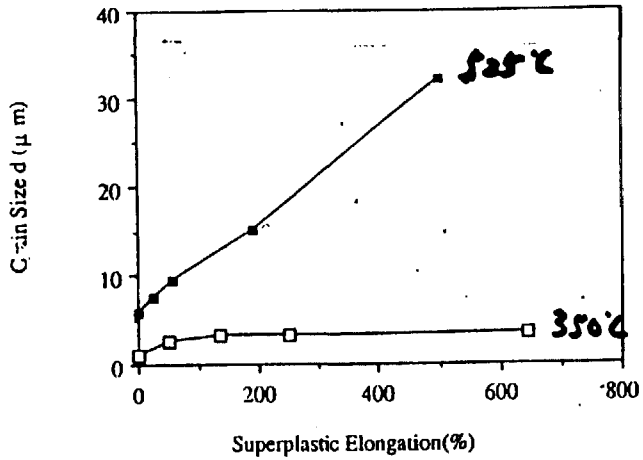
SEM 450°C 185%

$R_{GBS} = 44 \pm 5\%$

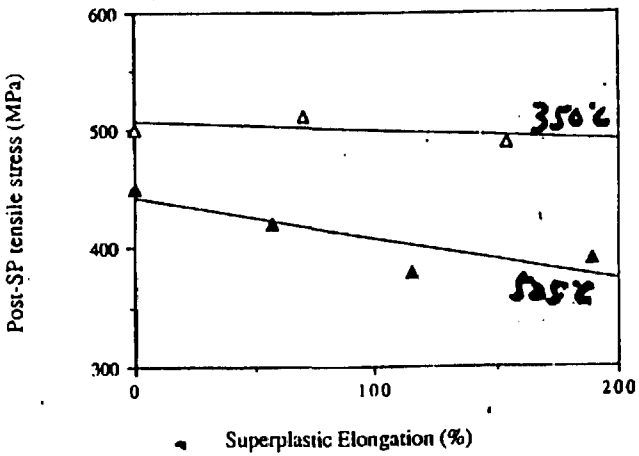
$\longleftrightarrow \sigma$



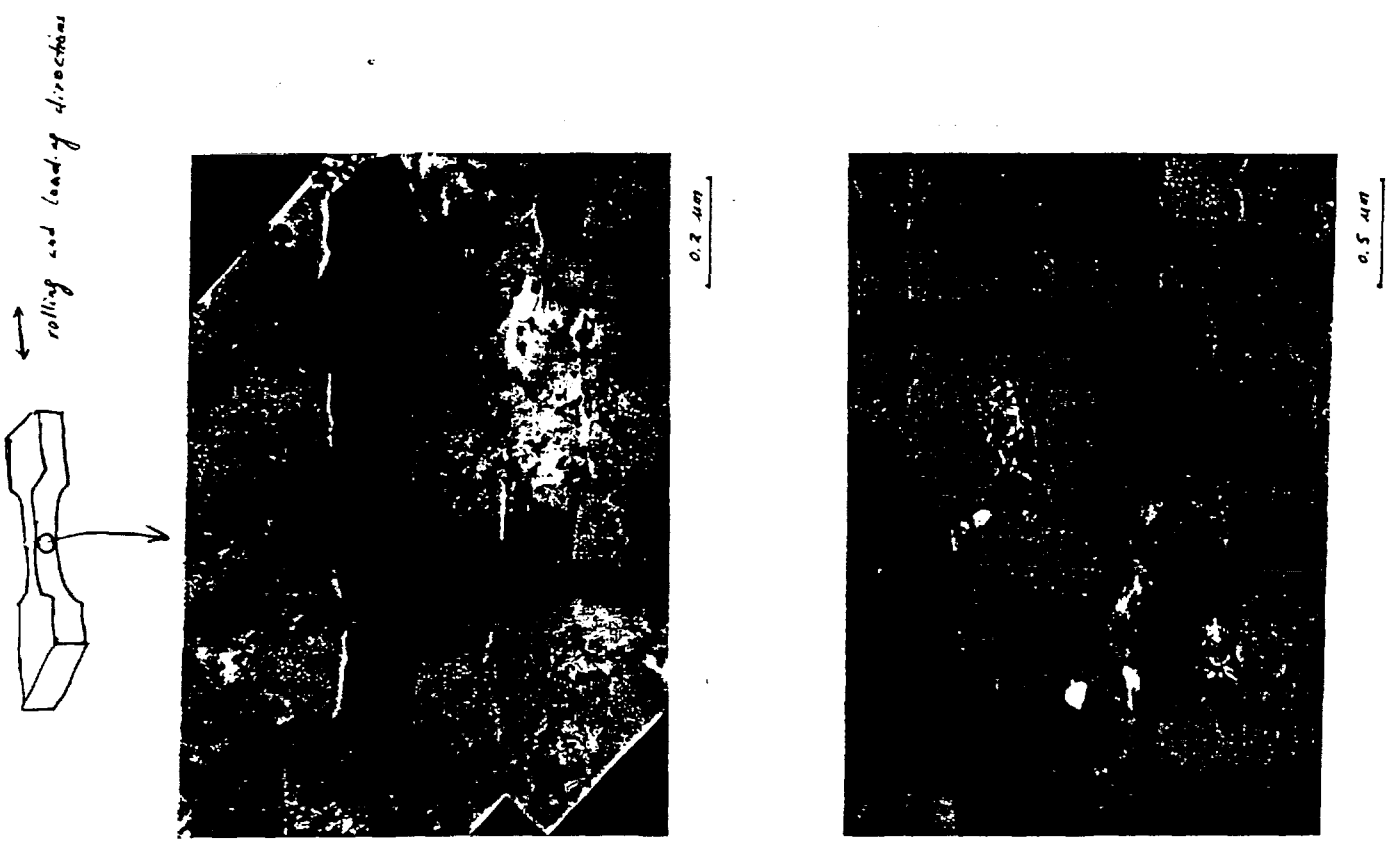
Li-depletion



grain size



post-SP σ_{UTS}



Superplasticity in Metal Matrix Composites

For conventional superplastic alloys $\dot{\varepsilon}=10^{-4} \sim 10^{-3}\ s^{-1}$

For MMCs $\dot{\varepsilon}=10^{-1} \sim 10^{1}\ s^{-1}$

Nieh et al. (1984)[1] first demonstrated

Nieh et al. (1991)[21] termed HSRS

High Strain Rate Superplasticity

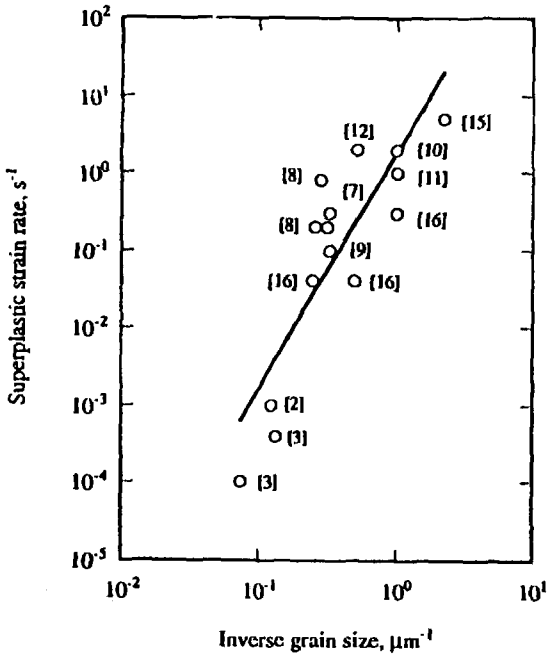


Figure 1 Grain size dependence of superplastic strain rate in alumunum matrix composites.

$$\dot{\epsilon} = A \frac{G b D}{kT} \left(\frac{b}{d}\right)^{2} \left(\frac{\sigma - \sigma_0}{G}\right)^{2}$$

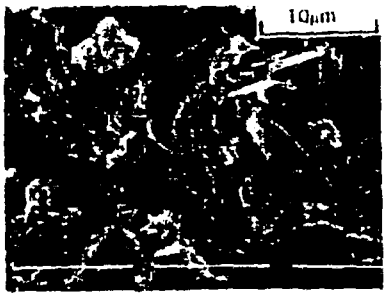
Table 1 Superplastic behavior in aluminum matrix composites

Composites	ε, %	T_{sp}, °C	$\dot{\epsilon}$, s^{-1}	m	d, μm
2124/SiC/20_w [1]	300	525	0.33	0.33	---
PM-64/SiC/10_p [2]	450	500	10^{-3}	0.5	8
2014/SiC/15p [3]	160	480	10^{-4}	0.4	13.5
7475/SiC/15p [3]	97	515	$4x10^{-4}$	0.43	7.3
2124/β-Si_3N_{4w} [4]	250	500	0.17	0.5	---
6061/β-Si_3N_{4w} [5]	200	525	0.16	0.5	-
6061/SiC/20_w [6]	300	550	0.17	0.32	---
6061/α-$Si_3N_4/20_w$ [7]	150	545	0.3	0.4	3
6061/β-$Si_3N_4/20_w$ [7]	260	545	0.2	0.4	3.1
Al-Zn-Mg/α-$Si_3N_4/20_w$ [8]	160	525	0.8	0.4	3.5
Al-Zn-Mg/β-$Si_3N_4/20_w$ [8]	230	545	0.2	0.4	3.8
6061/$Si_3N_4/20_p$ [9]	450	545	0.1	0.3	3
6061/$Si_3N_4/20_p$ [10]	620	560	2	0.3	1
5052/$Si_3N_4/20_p$ [11]	700	545	1	0.3	1
6061/$Si_3N_4/20_p$ [12]	500	560	2	0.5	1.9
6061/SiC/20_p [13]	207	580	0.27	0.33	---
6061/SiC/17.5_p [14]	350	580	0.1	0.5	---
IN9021/SiC/15_p [15]	610	550	5	0.5	0.45
2124/β-$Si_3N_4/20_w$ [16]	280	545	0.04	0.3	4
2124/β-$Si_3N_4/20_p$ [16]	280	500	0.3	0.3	1
2124/β-$Si_3N_4/20_p$[16]	840	515	0.04	0.3	2
6061/β-$Si_3N_4/25_w$ [17]	173	545	0.02	0.3	---
7075/α-$Si_3N_4/27_w$ [18]	260	500	0.18	0.35	---
2024/α-$Si_3N_4/27_w$ [19]	175	500	0.17	0.3	---
2024/SiC/21_w [19]	140	510	0.09	0.38	---
8090/SiCp [20]	300	575	0.18	0.53	---

Filament appearance on fractured surfaces

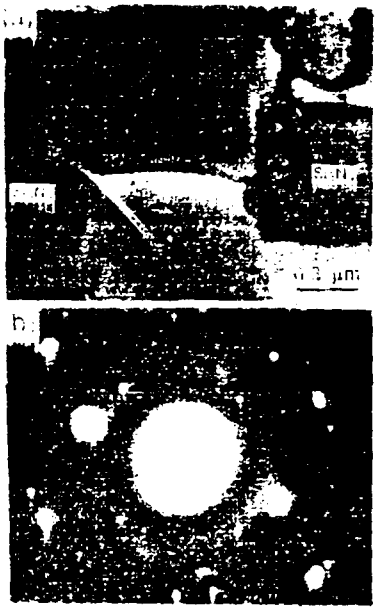
Hikosaka et al. [13]

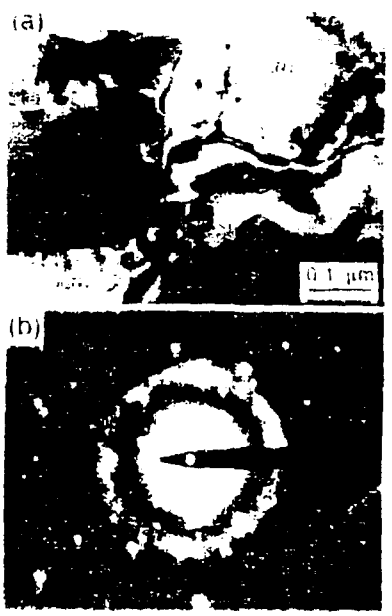
Nieh et al. [14]

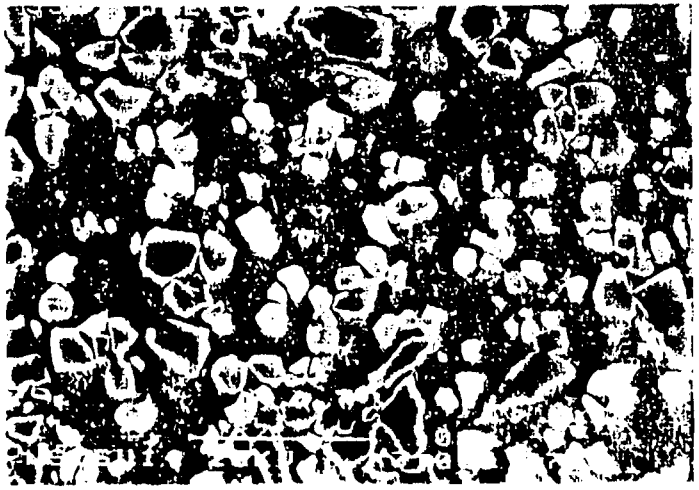


Koike et al. [41] In situ observations in partial melting

Composites	T_{sp}	T_i	T_s
2124/Si_3N_{4p}	515	511	502
5052/Si_3N_{4p}	545	548	607
6061/Si_3N_{4p}	545	549	582

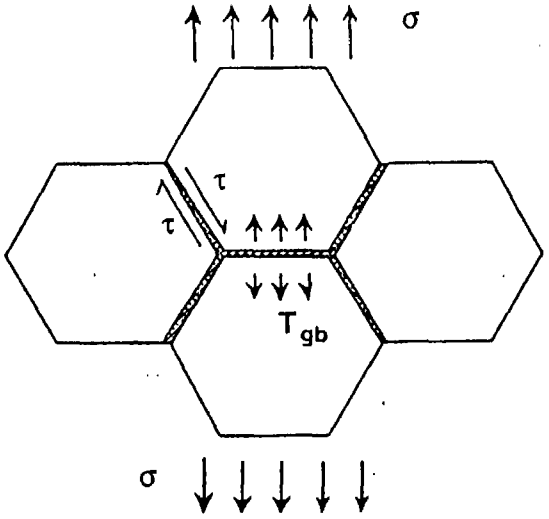






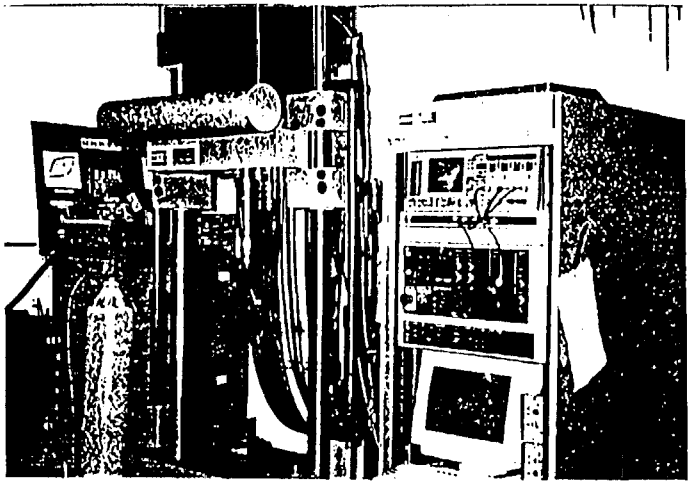
2024Al/15% SiC(p)

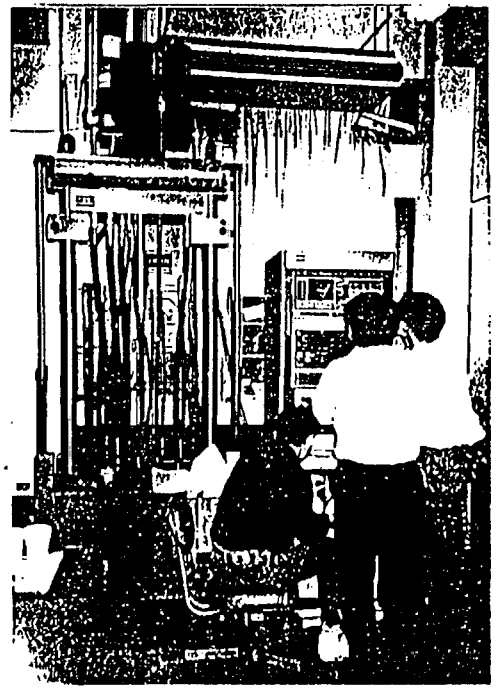
$d_p \sim 2.5\ \mu m$



relieves stress concentration

acts as lubricants





MTS
819
5-ton
servohydraulic
dynamic
high-rate
system

* Accumulator
* Kistler 9361A force link
* Kistler 5004 Charge amp
* LVDT
* Nicolet 420 oscilloscope
* Anti-rotation
* Tension, fati Compression Punching Charpy, Bendin
* up to 16 m/s (E 1-5 x 10
* HP-VEE-Test Soft...

6061 Al / 15% SiC(p)

d_p - 0.5 ± 0.4 μm

6061 Al / SiC (d ~ 0.03 μm)

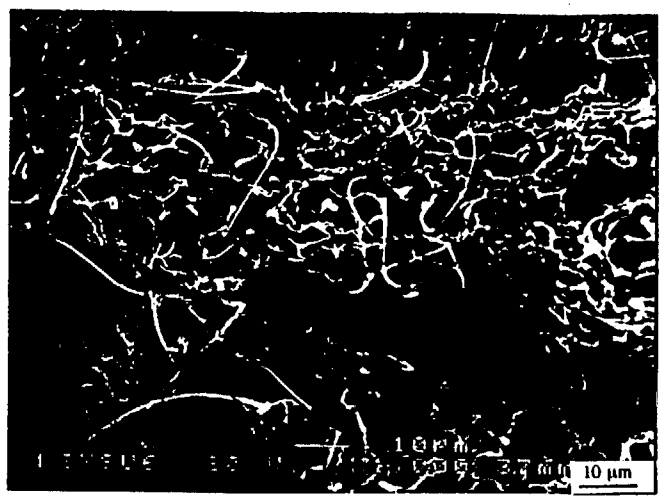
6061 Al / Si (d ~ 0.03 μm)

Comparison of the test conditions and the optimum results obtained

	Particle size d_p (μm)	Grain size d (μm)	T (°C)	$\dot{\varepsilon}$ (s^{-1})	ε
7091/30%B_4C	5.0	5.0	545	$2x10^{-2}$	110%
6061/15%SiC	2.5	3.5	580	$1x10^{-2}$	150%
2024/15%SiC	2.5	3.5	525	$1x10^{-1}$	167%
6061/15%SiC	0.5	0.8	525	$1x10^{-1}$	286%
2024/15%SiC	0.5	0.8	580	$1x10^{-1}$	230%
Pure-Al/ 15%SiC	0.5	0.8	640	$1x10^{0}$	280%

6061/SiC 0.03 0.1 ?

(a)



(b)

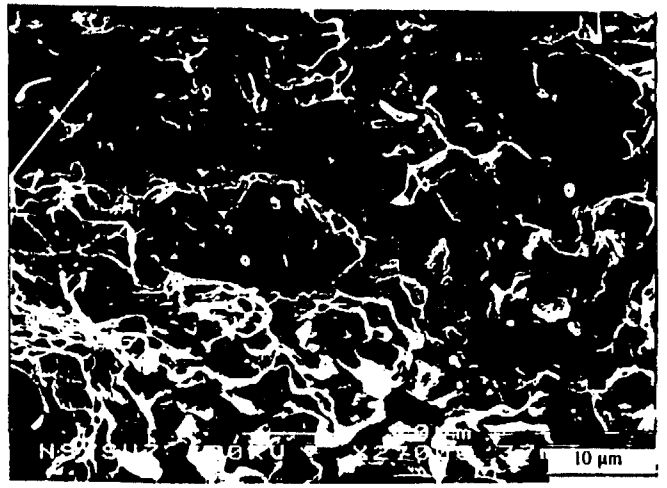


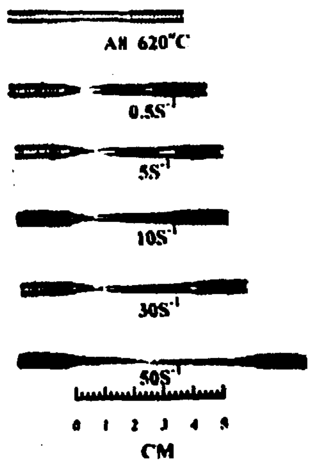
Fig. 43 SEM factographs showing the (a) more extensive hair-like morphology, and (b) partial melting seen in the 6061 composite tested at 580 °C at $5x10^{-4}$ s^{-1}.

MA Al/Al_3Ti

Prof. P. W. Kao

National Sun Yat-Sen University

	T (°C)	$\dot{\epsilon}$ (S^{-1})	ϵ (%)	ΔA (%)	U.T.S (MPa)	$\sigma_{0.1}$ (MPa)
A8	550	0.5	17	81	90	51
		5	38	89	80	59
	575	0.5	20	85	73	42
		5	55	92	78	63
		50	37	78	105	99
	600	0.5	29	93	50	32
		5	50	92	70	51
		50	97	85	94	76
	620	0.5	34	94	44	25
		5	65	94	45	29
		10	61	86	58	44
		30	94	86	63	57
		50	194	90	68	63



Al/Al_3Ni

Wan, Chen, Lui (1997)

National Cheng-Kung University

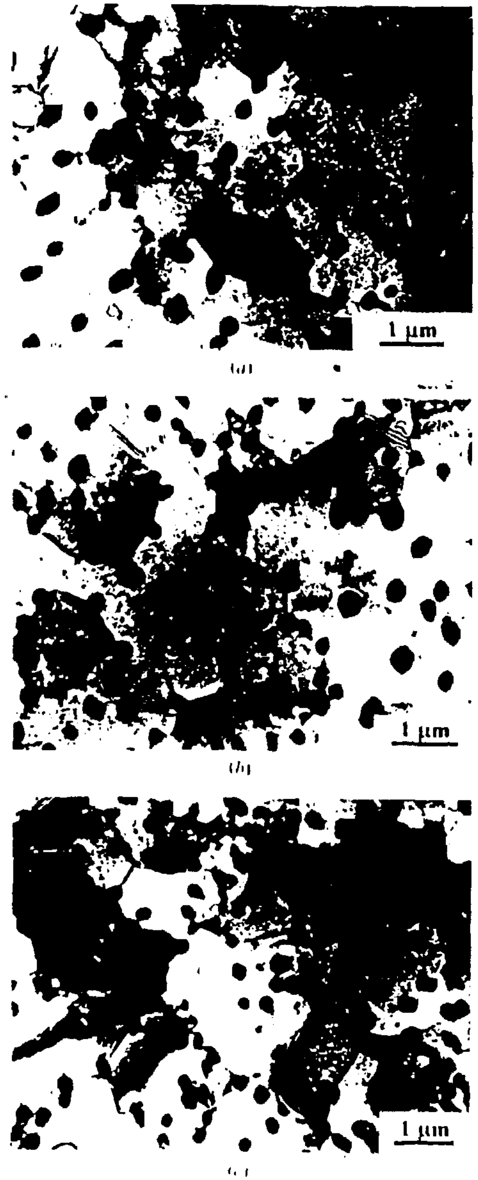


Fig. 2—[illegible]

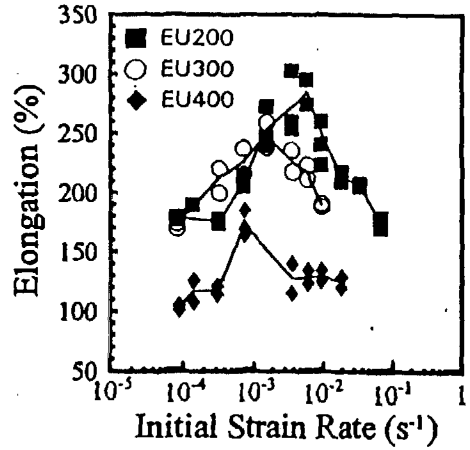
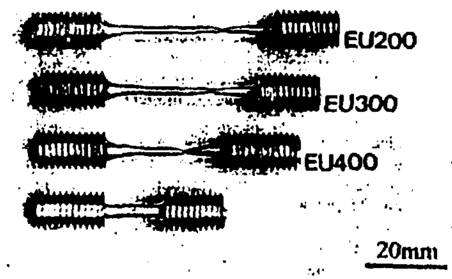


Fig. 3—Elongation as a function of initial strain rate for EU200, EU300, and EU400.



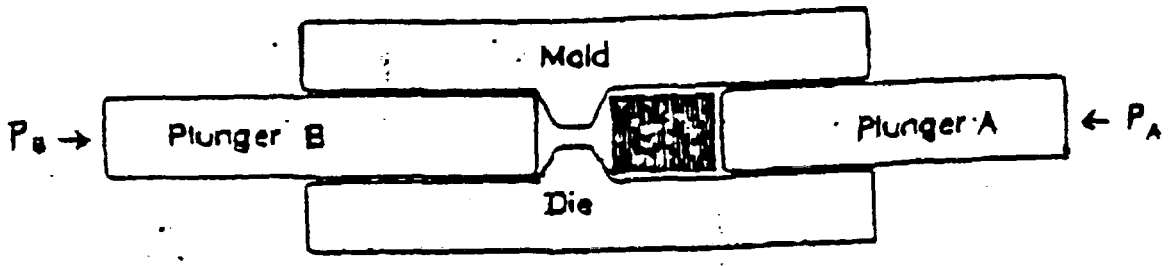
(b)

Fig. 4—(a) True tensile stress vs true tensile strain and elongation of the three test materials obtained at their initial strain rates of maximum elongation. (b) Undeformed specimen and those deformed to failure at their initial strain rates of maximum elongation.

Prof. J. W. Yei

National Tsinghua University

Reciprocal Extrusion



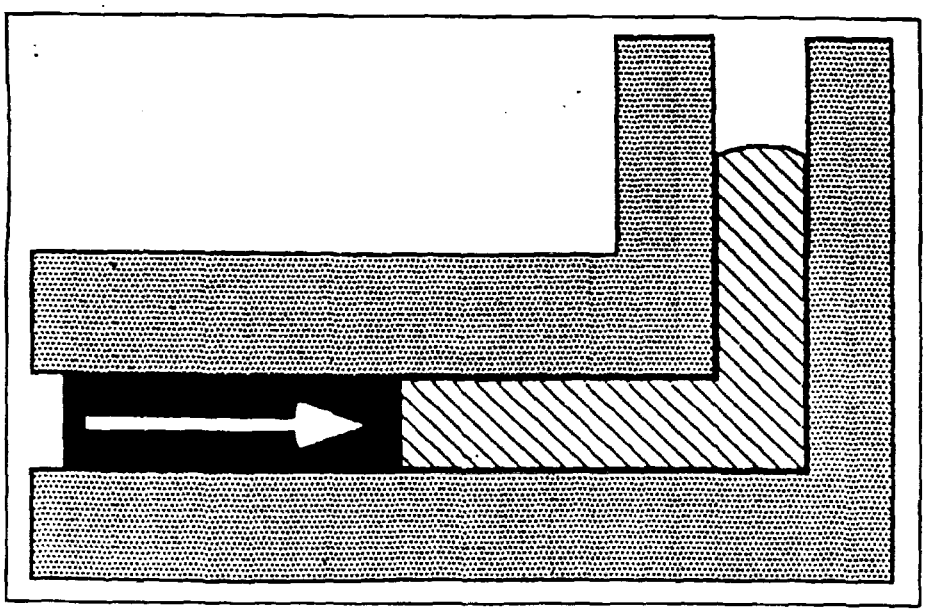


Figure 2. Equal channel area extrusion is a way to impart much working without causing a change in cross sectional area.[37]

Tsenev, Valiev, Kujeev (1997)

Parameters of superplastic deformation of alloys under study .

Alloy	d, μm	T, K	$\dot{\varepsilon}$, s^{-1}	m=dlgσ/dlg$\dot{\varepsilon}$	δ, %	σ_{20}, MPa
Al - Cu - Zr	8	773	$3 \cdot 10^{-3}$	0,50	800	6
	0,3 220°C,	493	$3 \cdot 10^{-4}$	0,48	320	23
	8	493	$3 \cdot 10^{-4}$	0,19	130	34
Al alloy 1420	20	793	$4 \cdot 10^{-4}$	0,50	560	8,5
	0,8 300°C,	573	$1 \cdot 10^{-4}$	0,41	330	15
Al alloy 1460	6	793	$3,5 \cdot 10^{-4}$	0,52	650	4
	0,9 330°C,	603	$1 \cdot 10^{-4}$	0,37	370	12

T. R. McNelley and H. E. McMahon
Naval Postgraduate School, USA
Al-10 Mg-0.12

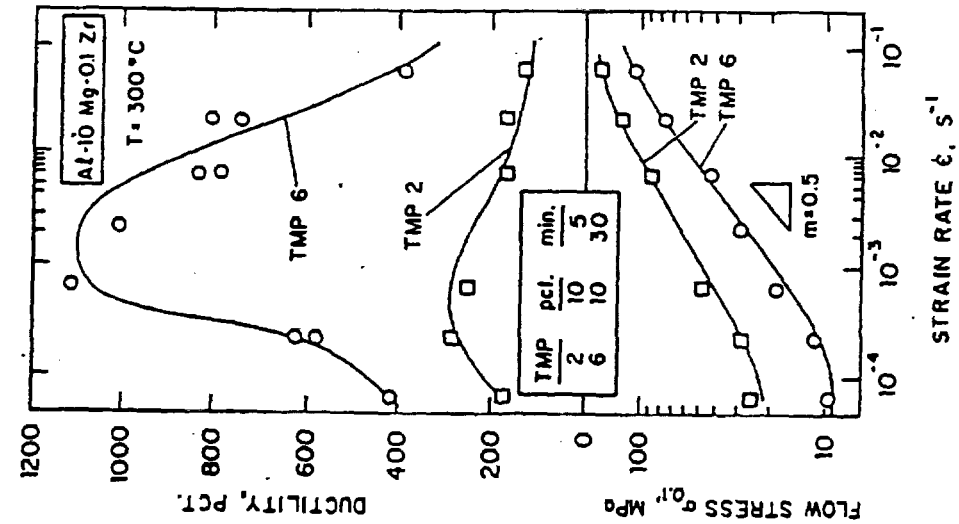


Fig. 1—Mechanical property data illustrating the variation in mechanical behavior at 300 °C for the two processing schedules.[5]

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