

G. Avramović-Cingara*, H. J. McQueen**, G. L. 'Esperance***

**SUBSTRUCTURE DEVELOPED IN THE HOT WORKING OF
ALUMINIUM-LITHIUM ALLOYS**

Summary

Aluminium-lithium alloys are currently attracting great attention for application in the aircraft and aerospace industries. The interest in Al-Li alloys centers around the fact that Li is element that significantly decreases the density and simultaneously increases the elastic modulus. For aerospace and aircraft applications, decreases in density and increases in elastic modulus, strength and allowable stress for fracture, fatigue and stress corrosion can lead directly to weight savings.

However, relatively poor qualities in ductility, toughness and hot-workability seem to decelerate the progress of commercialization in these alloys (4,5). Microstructural control through thermomechanical processing is also expected as a measure improving those qualities as well as acquiring superplasticity. Several types of intermediate thermo-mechanical treatment were successfully developed in 2000 and 7000 series alloys, but only a few works seem to have been done on Al-Li alloys (1,6,9). In this view, as the fundamentals for thermo-mechanical processing, hot deformation characteristics and microstructural changes specific in Al-Li alloys should be made clear. In this study, hot deformation behaviour and microstructural changes accompanied by dynamic processes were systematically investigated in Al-Li-Cu-Mg-Zr alloy focusing on the effect of interaction dislocations and dispersoids.

*Dr Gordana Avramović-Cingara, docent, Fakultet tehničkih nauka, 21000 Novi Sad, V. Vlahovića 3.

**H. J. McQueen, Concordia University, Montreal, Canada H3G 1M8

***G. L. 'Esperance, Ecole Polytechnique, Montreal University, Montreal, Canada H3C 3A7

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SUBSTRUKTURA TOPLO DEFORMISANIH LEGURA Al-Li

Istraživane su dve legure Al-Li-Cu-Mg-Zr, koje su deformisane na toplo sa ciljem da se definišu mikroskopski mehanizmi koji kontrolišu SUPERPLASTIČNOST. Legure su deformisane torzijom na 300-500°C, pri brzinama deformacije 0,1-4 s⁻¹.

Legure su deformisane u dva stanja:

1. Rastvarajuće žarenje na 546°C i kaljenje do temperatura deformacije;

2. Taloženje 7,5^h na 400°C (prestarelo stanje).

Nadjeno je da napon zavisi od brzine deformacije preko sinh funkcije sa eksponentima 3,1 i 3,5 i od temperature preko Arrhenius-ove jednačine sa aktivacionim energijama 209 i 185 kJ/mol.

Ispitivanje mikrostrukture transmisijom elektronskom mikroskopijom potvrđuje ono što je indicirano iz oblika krivih napona - deformacija, tj. da se u toku procesa odigrava dinamičko oporavljanje. Nadjeno je da su subzrna legure prethodno žarene (1) veće od onih kod prestarele (2), koja poseduje disperzoidne čestice. Veličina subzrna raste sa porastom temperature deformacije od 300 do 500°C i sa smanjenjem brzine deformacije od 1 do 0,1 s⁻¹.

Dodatnim testom ispitivana je superplastičnost prestarelih legura. Uzorci su pri brzini deformacije 1,0 s⁻¹ do ekvivalentne deformacije 2, zatim su naglo promenjeni uslovi; primenjene su brzine deformacije 10⁻⁵ do 10⁻³ s⁻¹ i temperature oko 500°C. Merjenja koeficijenta osetljivosti brzine deformacije ($m \approx 0,37-0,5$) i substrukturne karakteristike potvrđuju da je postignuta superplastičnost legura Al-Li.

EXPERIMENTAL PROCEDURE

The alloys of composition shown in Table 1 were supplied by Alcan Aluminium Laboratories, Kingston.

Table 1. Composition

Alloy	Li	Cu	Zr	Mg	wt%
8090	2.52	1.44	0,10	0,60	
8091	2.76	1.86	0,12	0.80	

Metallography tests were performed on this material to determine axis and direction of rolling of the origin structure. After that material was cut in rods. The rods were solution-treated at 550°C in argon for 1 hr as a standard condition. The rods were machined to torsion specimens with gauge length of 25,4 mm and diameter of 6,35 mm (7).

The alloys were deformed in two conditions:

1. Solution treated at 546°C for 30 min, then cooled to test temperature;
2. Overaged 7,5 hrs at 400°C. The processing steps of overaging were: solution heat treatment in argon at 546°C for 1 hr, followed by cold water quenching and aging for 7,5 hrs. For torsion testing they were reheated to test temperature and held for 5 min to equilibrate.

The tests were conducted in a radiant furnace controlled by thermocouple pressed against the gauge section and in argon atmosphere (7,1). The computer-directed, servo-controlled hydraulic torsion machine measured the torque generated during tests at strain rates, $\dot{\epsilon}$, of 0.1, 1,0 and 4.0 s⁻¹ to fracture or a fixed strain ($\epsilon=4$). The specimens were deformed at 300-500°C (1). At fracture or arrest, the specimens were quenched with a wear spray in 3 sec to preserve the hot-worked structure.

Sets of multistage tests were conducted on overaged specimens to check for superplastic behaviour (1,2,4,10,11,12). The specimens were deformed at 250 or 300°C and $\dot{\epsilon} = 1,0 \text{ s}^{-1}$ (or $0,1 \text{ s}^{-1}$) to $\epsilon = 2.0$, in order to develop a fine dense substructure in the outer annulus. During an arrest of 300 sec, the temperature was raised to 450 or 500°C. A series of

strains of $\epsilon = 0,3$ with 5 sec intervals were performed as $\dot{\epsilon}$ was progressively changed from 8×10^{-2} to $5 \times 10^{-3} \text{ s}^{-1}$ in 8 steps. The superplastic behaviour was concluded from the values of strain rate sensitivity (m) determined. Such a technique depends on the outer annulus (about 1 mm thick) having the most important influence on torsional stress. High superplastic strains are not directly observable since the normal torsion strains are quite high. Tension tests are not feasible since core, which does not contain the fine structure, becomes more important (1,2).

For metallography the gauge section was polished to provide flat section normal to the radius about 0,5 mm below the surface. The samples were anodized using a 3% solution of a 50% HBF_4 at 30 V for 4 mins and were examined in polarized light.

Transmission electron microscopy was performed on JEOL FX at 200 kV. The samples for TEM were cut normal to the radius, just below the surface of the torsion specimens. Electropolishing was performed at 20 V and -40°C . Subgrain size was determined using a planimetric method and were based on at least 10 micrographs. The orientation relationship of grains and subgrains were determined by recording appropriate diffraction patterns. The subgrain misorientation angle was measured on low angle boundaries. The tilt component of the misorientation across low-angle boundaries was determined by measuring the shift of Kikuchi line intersections. The rotation component was measured by observing the angle of rotation of SAD pattern on crossing the boundary.

RESULTS AND DISCUSSION

Representative flow curves (Fig. 1) for both alloys exhibit a rapid hardening to a peak (σ_p, ϵ_p), followed by work softening toward a steady state regime. The curves indicate lower strain hardening as T rises and $\dot{\epsilon}$ declines.

The steady state values of flow stress were plotted as functions of T and ϵ (1) to determine the applicability of the constitutive equation

$$\dot{\epsilon} = A(\sinh \alpha \sigma_p)^n \exp(-Q_{HW}/RT), \quad \text{Equ. 1}$$

where A, α, n, Q_{HW} and R are constants. Similar behaviour was observed in both alloys (1,6,9). For 8090 alloy, $Q_{HW} = 185$ kJ/mol and $n=3,1$, whereas for 8091 are 209 kJ/mol and 3,5. There is considerable scatter in the data points, which is result of modifications from common structure as a result of small variations in torsion test heating procedure.

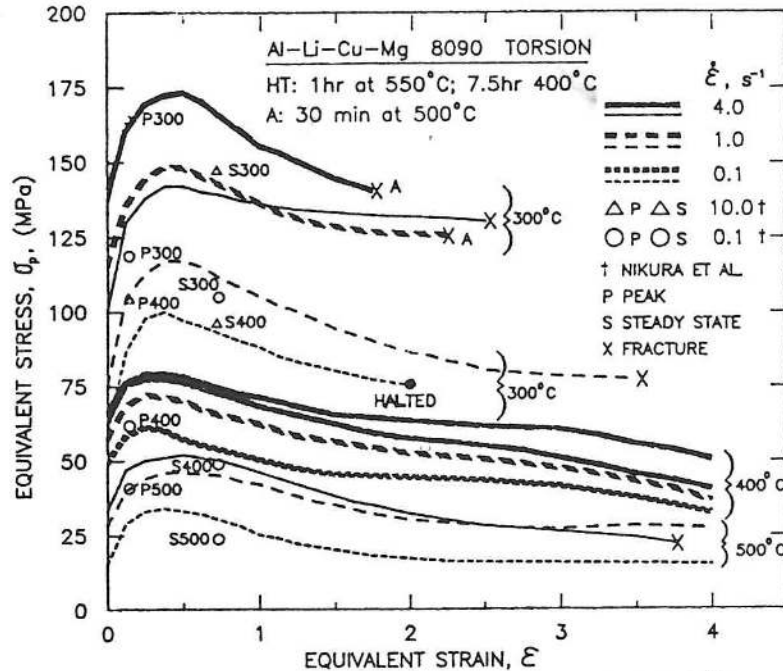


Fig.1: Stress-strain curves for 8090, T=300-500°C and $\dot{\epsilon} = 4,0-0,1$ s⁻¹. Specimens were overaged 7,5 hrs at 400°C.

The flow curves of the multistage tests are much lower for the high T, low $\dot{\epsilon}$ stages. In a plot of $\log \sigma$ vs $\log \dot{\epsilon}$ for stages 2 to 9 performed at 450 or 500°C and at a low $\dot{\epsilon}$ slopes lie between 0,37 and 0,5 which are adequate indication of superplastic behaviour (1).

The anodized specimens observed in polarized light consist of elongated grains which have normal to and thinned down in the axial direction as on Fig. 2a and 2b: The specimens of both alloys at the same temperature are very similar so they will be described together. At 300°C the structure was mainly irresolvable and with elongated grains. At 400°C, the subgrains are resolved as somewhat elongated subunits within the grains. With increasing the temperature, the subgrains develop more contrast and become larger, more polygonal and equiaxed. The subgrains in solution treated state are noticeably larger than those in overaged state.

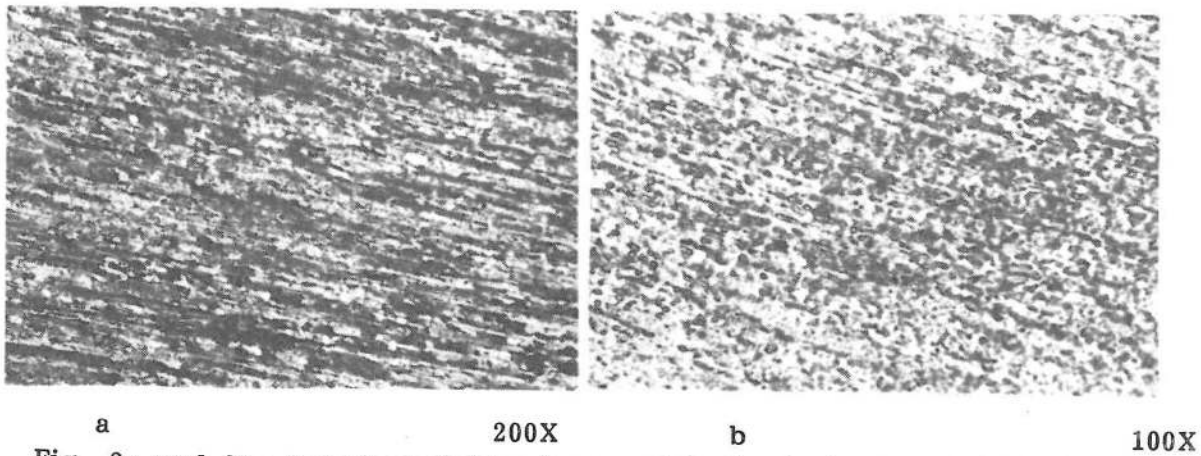


Fig. 2a and 2b: Polarized light micrographs of solution treated 8090 alloy; a - 500°C, 0,1 s⁻¹; b - 300°C, 1 s⁻¹

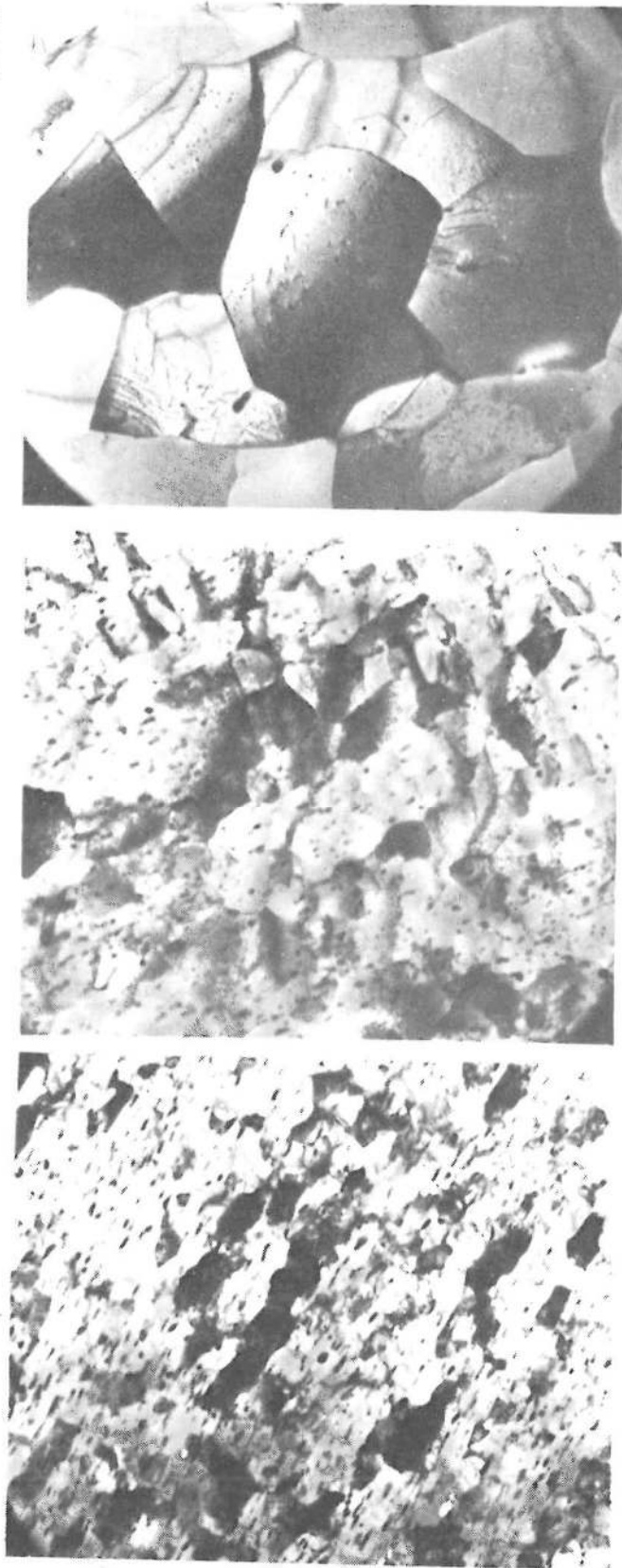
The microstructural evidence determined optically confirms the indications from flow curve shape that the mechanism is dynamic recovery.

The TEM microstructures of both alloys were investigated after aging and deformation processing step. Examples for the different temperatures and strain rates are shown in Fig. 3a,b and c, respectively. These low magnifications micrographs illustrate the presence and uniform distribution of coarse (<0,5 μm) second phase particles. The precise identifications of these precipitates is currently being investigated and will be reported at a later date. Subgrain sizes are changed from 0,85 μm at 300°C/4 s⁻¹ to 1,7 μm at 400°C/0,1 s⁻¹ and 5,5 μm at 500°C/0,1 s⁻¹. As the electron micrograph of Fig. 3 illustrate, the substructure become more fully polygonized as the temperature rose and as the strain rate decreased. The increased polygonization consisted of enlargement of the cells or subgrains and the arrangement of the subboundary dislocations into neater networks. For 500°C/0,1 s⁻¹ the particles are dissolved.

The TEM structures for material solution treated at 550°C are shown in Fig.3a, b and c, respectively. The subgrain size is generally larger as above and changed from 1,1 μm at 300°C/1,0 s⁻¹ to 2,8 μm at 400°C/0,1 s⁻¹ and 5,8 μm at 500°C/0,1 s⁻¹. The substructure observed from 300 to 500°C exhibit an increasing level of dynamic recovery with a decrease in interior dislocation density and with walls developing a longer link length, becoming more neatly arranged and appearing sharper and less ragged.

The subgrain boundaries have been examined on a random basis which showed that the majority of neighbors are tilted 2-5° about the foil normal. On occasion, boundaries of 20° are observed; however, they could be serrations of the original grain boundaries.

2 μm



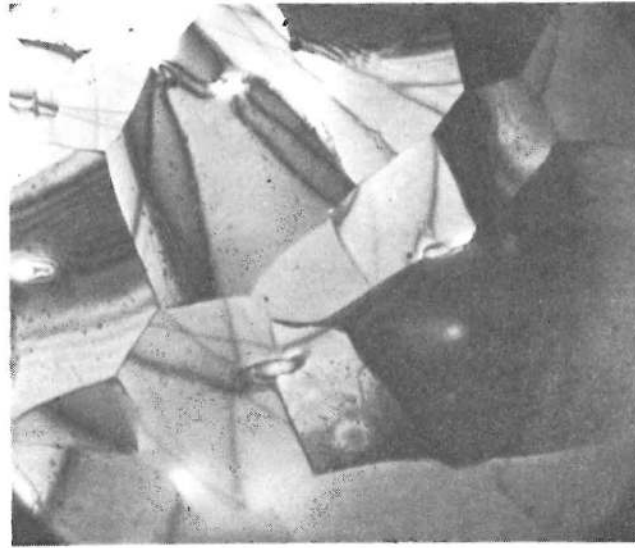
a. 300°C 1,0 s⁻¹
d = 1,4 μm

b. 400°C, 0,1 s⁻¹
d = 1,7 μm

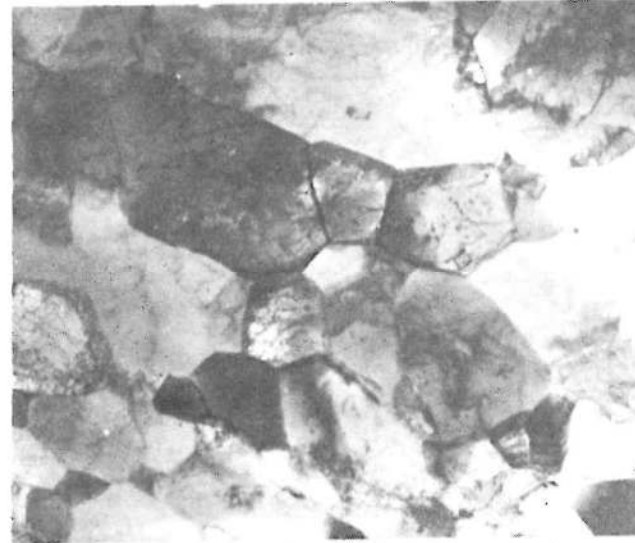
c. 500°C, 0,1 s⁻¹
d = 4,5 μm

Fig. 3. Subgrain sizes for overaged 8090 alloy.

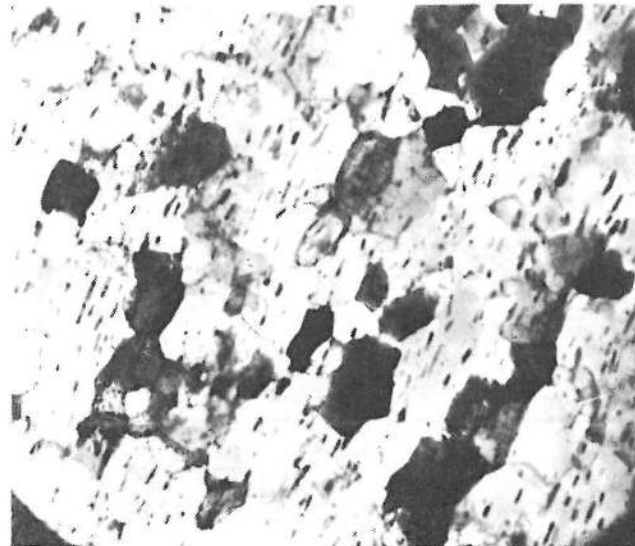
2 μ m



c. 500°C, 0,1 s⁻¹
d = 5,7 μ m



b. 400°C, 0,1 s⁻¹
d = 2,8 μ m



a. 300°C, 1,0 s⁻¹
d = 1,8 μ m

Fig. 4. Subgrain sizes for solution treated 8090 alloy.

Example of fine recrystallized microstructure of the overaged specimen after test of superplasticity is shown in Fig.5. It is important to recognize that these fine grains have high-angle boundaries and are not low or medium angle subgrains.

The precipitate particles are uniformly distributed. In many cases they lie in the sub-boundaries and interact with the arrays, being responsible for a considerably augmented local dislocation density. In situations where they are within the subgrain they have dislocation loops around them with links to the neighboring walls (Fig. 6a).

Fig. 6b shows dislocation substructure for alloy solution treated at 546°C. Electron microscopy reveals rows of primary and secondary prismatic loops and voids.

CONCLUSIONS

The 8090 and 8091 alloys were subjected to thermo-mechanical processing which produced a range substructures similar for both alloys. The alloys were deformed in two conditions: 1. Solution treated at 546°C, then cooled to test temperature; 2. Overaged 7,5 hrs at 400°C. The flow stresses were found to depend on the strain rate through a sinh function with exponent 3,1 and 3,5 and on temperature through Arrhenius term with activation energies 209 and 185 kJ/mol. The microstructural evidence confirms the indications from flow curve shape that the mechanism is dynamic recovery. The subgrains in solution treated state are larger than those in overaged state. TEM examination revealed a greater subgrain size as temperature rises from 300 to 500°C and strain rate declines from 1 to 0,1 s⁻¹. In additional tests, a hot working stage at high strain rates was followed by straining at 10⁻¹ to 10⁻¹ s⁻¹ and 450-500°C; measurement of strain rate sensitivity and substructure showed that superplastic behaviour was induced.

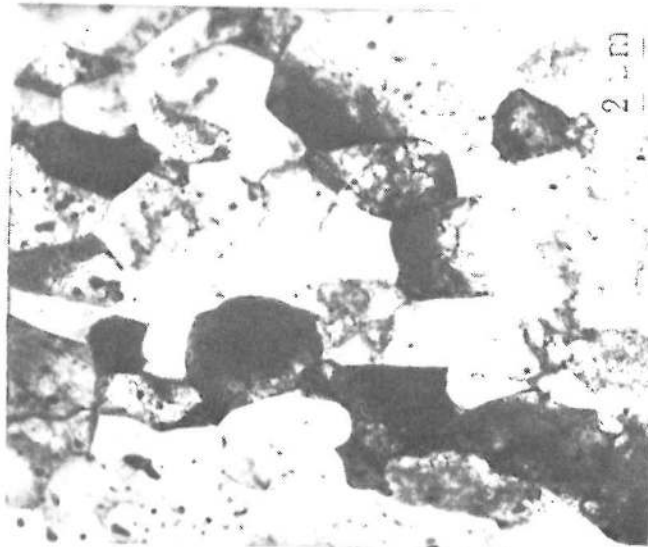
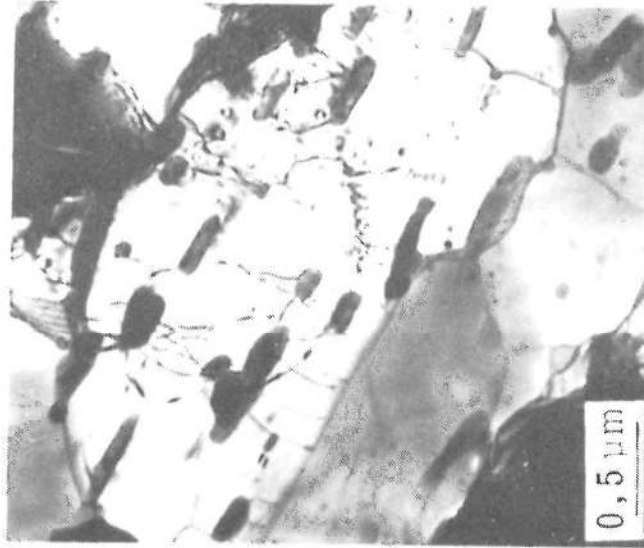
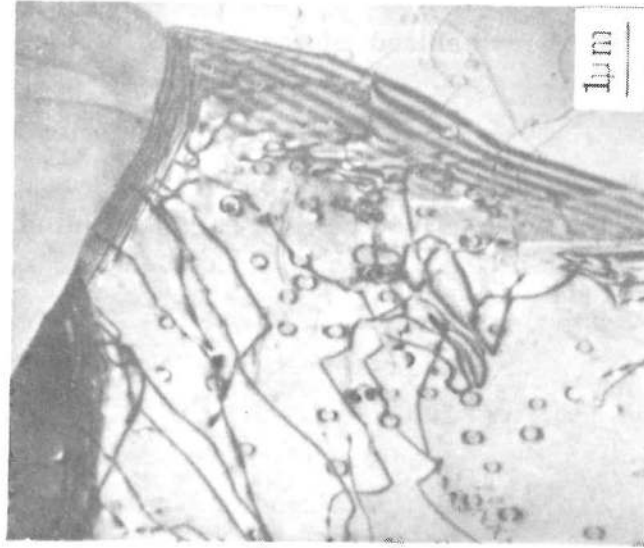


Fig. 5. Fine grains with high angle boundaries



a. Overaged 8090 alloy



b. Solution treated

Fig. 6. Dislocations substructure

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