TEM STUDY OF PRECIPITATION IN HIGH-STRENGTH 1987 ALLOY SHEETS AFTER SUPERPLASTIC FORMING.

O.G.Senatorova*, A.N.Uksusnikov**, J.N.Fridlyander*, J.Koshorst***, R.R.Romanova**, N.A.Ryazanova*, A.Galliot****, V.V.Cherkassov*

- * All-Russian Institute of Aviation Materials, 17 Radio Street, 107005 Moscow
- **Institute of Physics of Metals, Rus. Acad. of Science, 18 Kovalevskaya Street, 620219. Ekaterinburg
- *** Airbus Industrie, 31707, Blagnac Cedex, France
- **** Aerospatiale, 31060, Toulouse Cedex 03, France

ABSTRACT Superplastic high-strength Al-Zn-Mg-Cu alloys (1987 a.o.) have promising potential for application in aerospace industry. However the development of the manufacturing process for superplastic forming (SPF) of the alloys requires investigation concerning the influence of SPF on microstructure, which determines in its turn the properties.

Using high resolution transmission and scanning electron microscopy (TEM, SEM) and optical microscopy, the microstructural features such as precipitates, grain morphology, cavity were investigated for the 1987 experimental components deformed to $e_t \sim 0.8$ (equivalent elongation $\sim 125\%$) at T = 475°C, $\dot{\epsilon} = 10^{-3}$ s⁻¹ and then heat treated to T2 (T76) temper. The components were made of 2 mm sheet with initial unrecrystallized structure. It was found that the matrix and the grain boundary precipitation of the metastable and stable η (MgZn₂Cu) and η -phases was essentially identical in the initial sheet and SP formed component. SPF had not influenced on the precipitation kinetics during two-step T2 (T76) ageing. It was observed the uniform distribution of fine-dispersed precipitates in the grain body. Starting from sheet strain of $e_t \sim 0.25$ (elongation ~ 30 %) the grain structure is mainly recrystallized, fine (2-7 µm in diameter) due to dynamic recrystallization.

Keywords: subgrain/grain structure, precipitates, superplastic deformation/forming/alloy.

INTRODUCTION

High-strength aluminium alloys (7XXX series) are attractive for advanced superplastic forming process of manufacturing complex sheet structural aerospace components [1]. However determination of deformation parameters (including tolerant limits of strain values) requires to carry out investigations concerning the influence of SPF on evolution of microstructural features which determine in its turn post-formed properties and service properties, respectively.

The most widely studied alloy is high-purity 7475 (B9504 alloy – in Russia) with statically recrystallized fine-grained structure which is produced by special complex thermomechanical metallurgical processing [2-4]. And there is no sufficient data related to superplastic variants of high-strength Al-Zn-Mg-Cu alloys having initial unrecrystallized structure which is ensured generally by alloying minor additions. During early stages of superplastic deformation (SPD) the sheets from such alloys are dynamically recrystallized to a fine grain (subgrain) structure.

In the current study the precipitation process in the matrix and along grain (subgrain) boundaries during two-step ageing and also grain (subgrain) structure in different zones of experimental sheet part from superplastic 1987 alloy are considered. The alloy contains effective minor additions of transition elements which form very fine, coherent dispersoids to stabilize nonrecrystallized structure and to ensure additional hardening effect.

EXPERIMENTAL PROCEDURE

The material tested was a 2,15 mm thick 1987 alloy SP sheet, produced by traditional metallurgical technology. The sheet in T2 (T76) temper had mechanical properties as follows: UTS 520MPa; YS 440MPa; El 12,5 %.

According to hot uniaxial tensile tests (Fig. 1), elongation to failure was 500 - 700 % in the range of 450 - 475°C and at a constant strain rate of 10^{-3} s⁻¹, under atmospheric pressure. The gauge length and width of the specimen were 20 mm and 6 mm, respectively.

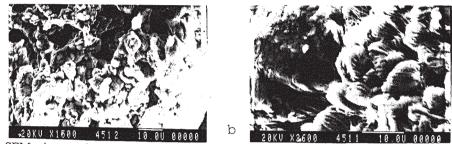
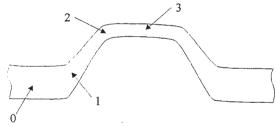


Fig. 1. SEM micrographs showing the structure of tensile tested specimens: a – the fracture surface; b – the longitudinal surface near the fracture location; $T = 450^{\circ}C$, $\dot{\epsilon} = 10^{-3}s^{-1}$, EI = 535%.

The sheet were superplastic deformed biaxially, that is, experimental parts with rigity ribs (Fig. 2) of 220×220×30 mm dimensions were manufactured at 470°C, without back pressure.

The parts were solution treated at 470°C followed by a water quench and a two-step ageing T2 (115°C, 5h + 165°C, 8h). The specimens for microstructural analysis with optical, transmission and scanning electron microscopy (TEM, SEM) were cut out from zones, noted with figures on Fig. 2. Precipitate structure was observed using a high resolution JEM 200CX TEM and JSM-840 SEM. Thin foils (after mechanical and electrolithic polishing) conformed approximately to mid-thickness of the part.



Zone	Thickness,	El.,%	Subgrain
	mm		/grain size,
			μm
0	2,15	0	0,5 - 4,5
1	1,65	30	2 - 5
2	0,95	125	/5-7
3	0,98	120	/5-7

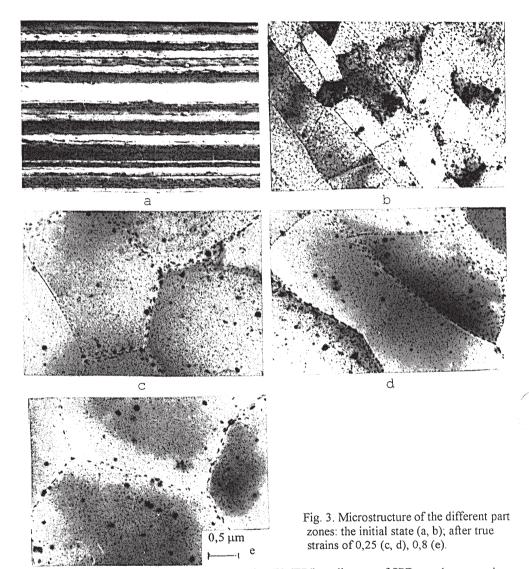
Fig. 2. Fragment of the part cross-section. Investigated zone is indicated by the figures.

RESULTS

The initial undeformed but only heated sheet (0 zone) is characterized by a very banded unrecrystallized structure with subgrain size of $0.5 - 4 \mu m$ (Fig. 3, a, b).

Analysis of electron micrographs great numbers, showed, that in the sheet SP deformed by 30% ($e_t \sim 0.25$), mainly fibrous structure with considerable fraction of recrystallized equiaxed grains (Fig. 3, c, d) is observed. After SPF up to ~ 125 % ($e_t \sim 0.8$) the structure is mainly fine grain, recrystallized (Fig. 3, e).

Volume fraction of cavity for cross-sections 2 and 3 determined by quantitative metallography of as-polished specimens is evaluated as ~2 % (without imposed back pressure); in section 1 cavity was not observed practically.



The matrix precipitation after two-step ageing T2 (T76) at all zones of SPD part (cross-section are 0-3) is characterized by high dispersion and homogeneous distribution of the main strengthening metastable η '-phase (MgZn₂Cu) (Fig. 4).

Histograms, obtained by the results of measuring not less than 500 particles by dark field images, showed that in all part cross-sections the size of dominating fraction (\sim 80 %) of η '-particle is within the ranges of 9 – 15 nm (Fig. 5).

Grain boundary precipitates consist, in general, of stable η - phase (particles size of 20-80nm) (fig. 6) and a certain fraction of η '-phase (particles size of 16-20 nm).

The width of precipitation free zones (PFZ) adjacent to the grain boundaries does not change practically; it is 20-28 nm. It should be noted, that in the article [3] a considerable expansion of PFZ under influence of SPF - process in the sheets from statically recrystallized 7475 alloy was noted.

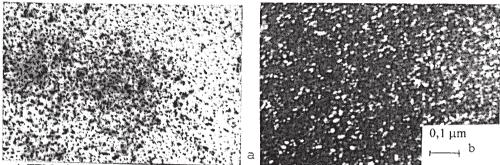


Fig. 4. TEM micrographs showing η '- precipitates: a-bright field image, b-dark field image in reflex $\bar{1}02\eta$ '.

In grains body and on their boundaries the dispersoids particles size of 10-35 nm are observed, which are coherent to the matrix, it is evident from a contrast on dark field images.

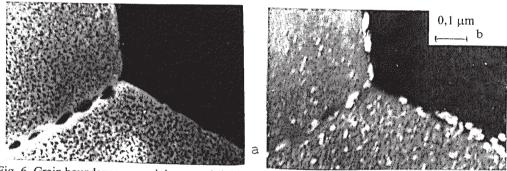


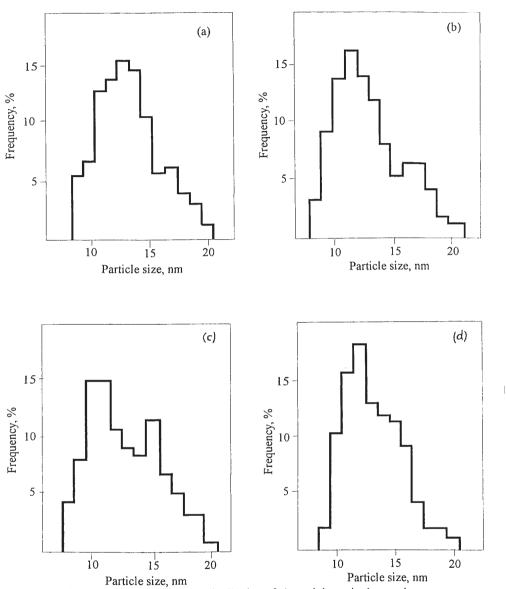
Fig. 6. Grain boundary η - precipitates: a- bright field; b- dark field.

CONCLUSION

- 1. Under SPF the part to true strain $e_t \sim 0.25$ (El 30 %) the structure of sheets is mainly unrecrystallized, subgrained. After strain $e_t \sim 0.8$ (125 %) a fine grain structure (5-7 μ m) is formed generally as a result of dynamic recrystallization.
- 2. No considerable difference was observed betweens the matrix and grain boundary precipitation of a solid solution during step ageing T2 (T76) in the initial sheet and after strain up to $e_t \sim 0.8$ (El 125%) SPF parts. It can be supposed that the post-formed properties which are depended on the ageing treatment would be not changed. No influence of coherent dispersoids upon kinetics of ageing was observed.

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Particle size, nm Particle size, n Fig. 5. Histograms showing the size distribution of η precipitates in the matrix. Sections: 0 (a), 1 (b), 2 (c), 3 (d).