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Structural Parameters of Partially Homogenized Ingots of High-Strength Al-Zn-Mg-Cu System Alloys

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Abstract

The microstructure of wrought products and, consequently its properties, depend on as-cast microstructure of ingots and homogenization practice. It has been investigated phase composition, volume percent of excessive soluble hardening phases and insoluble impurity-containing phases and possible local eutectic melting in the experimental and commercial ingots of high-alloying V96Zr-3pch (7449-type) and V95pch (7175-type) alloy with optical microscopy, X-ray diffraction (XRD), electron probe microanalysis and differential scanning calorimetry (DSC). Hot workability was observed by hot compession tests and by hot tensile tests. It has been examined cast structure after preliminary homogenization annealing at 350-460 °C. It has been shown that the particles of S(Al_2CuMg) phase and phases, containing Fe and Si – natural impurities ($Al_{23}CuFe_4$ a.o.) may be in the ingots at certain conditions.

1. Introduction

High-strength and superhigh-strength aluminium alloys of Al-Zn-Mg-Cu system are effective aerospace materials to save weight of airframes due to their high specific strength. They are mainly designated for structures compressively loaded in flight – upper wing in form of rolled and extruded products. As it is known material for upper wing panels is selected in terms of possessing high critical stresses at compression. Superhigh – strength aluminium alloys with Zr addition, having the highest yield strength (typical values of 580-630 MPa), especially correspond to this term.

For the first time in the world minor Zr addition was used in the most strength aluminium Russian V96Zr alloy (VIAM development in 1950s) [1-3]. Compared to manganese and chromium, Zr has ensured retaining more unrecrystallized structure, better hardenability and higher ductility in hot worked semiproducts. At first, the effective Zr amount (0,1-0,14 %) without formation of primary detrimental intermetallics $ZrAl_3$ compounds in ingots was established [4].

The superhigh-strength V96Zr-3pch is designated for advanced aviation and rocket technique to replace high-strength V95och (7475-type) alloys. It has been developed by

VIAM in 1970. Analogous superhigh-strength alloys 7055 (Alcoa), 7449 (Pechiney) appeared considerably later (the 1990s) [5].

One of the important technical problems associated with these complex highly alloyed materials is regulation of ingot microstructure to create desired level of service characteristics and their stability and also to provide good hot workability of semiproducts. In the present article composition of excessive coarse intermetallics based on the available phase diagrams, metastable local eutectic melting and also hot workability are considered.

2. Experimental Procedure

Samples were cut out the different zones of the large-scale DS ingots (up to 680 mm in diameter and 400 mm in thickness) of V96Zr-3pch (7449-type) and V95pch (7175-type alloys) and investigated.

The ingots were partially homogenized (pre-heated) followed by cooling in a furnace to ~300 ºC.

The phase compositions of ingot samples were observed by optical light microscopy, X-ray diffraction on DRON-3M (in CuK_{α} - radiation), scanning electron microscopy on LEO-430i equipped with system of local analysis LINK-ISIS-300. Differencial scanning calorimetry (DSC) using calorimeter DSC111 was performed to determine temperature range of melting. The specimens with a diameter of 5 mm were heated at rate of 5 ºC/min.

Hot tests to study technological ductility were done, using cylindrical specimens with diameter of 15 mm and height of 20 mm for compression and specimens with gauge diameter of 5 mm for tension.

3. Results and Discussion

Homogenization practice of ingots have some principal tasks: (i) maximum dissolution of soluble excessive coarse intermetallic particles, that distribute along dendritic boundaries; (ii) removal of dendritic microsegregation; (iii) improvement in ductility during plastic deformation and (iv) formation of dispersoids containing transition elements with specific morphology to control processes of product recrystallization.

High-strength aluminium alloys, having complex chemical composition, are multiphase alloys. According to investigations of quaternary Al-Zn-Mg-Cu phase diagram, including [6], the alloys of practical compositions may be near α_{Al} + S + M [$Mg(ZnCuAl)_2$], α_{Al} + M, α_{Al} + S(Al_2CuMg) phase regions in equilibrium conditions (Figure 1).

M [$Mg(ZnCuAl)_2$] phase is quaternary solid solution, which forms by isomorphic η($MgZn_2$) phase of Mg-Zn system and U($(Cu_xAl_{1-x})_2Mg$) phase of Al-Cu-Mg system. The ternary S(Al_2CuMg) compound is detected in wide range of concentrations; Zn is not practically soluble in the S-phase.

The real boundaries of phase regions in alloys are displaced as a consequence of practical commercial conditions of ingot solidification and also due to additional alloying by transition elements (Zr, Mn, Cr, Ti) and occurance of permanent natural impurities (Fe, Si).

After partial homogenization at 400 °C, 6 h to relieve residual cast stresses, the excessive soluble M and S constituents which lie along dendritic arm boundaries were found in the microstructure of V96Zr-3pch alloy ingot (at 7,9 % Zn; 1,8 % Cu; 2,0 % Mg) (Figure 2). These phase have dark color in optical microscope (Figure 3 a & b). S-phase particles dissolve after long soak at 460 °C. In the alloys with higher Cu content (more 2 %) excessive coarse constituents of S-phase do not completely dissolve during homogenization heating and solid solution heating and is retained.

Interdendritic coarse particles of M-phase dissolve faster than S-phase. However fine M-precipitates re-appear on dendrites and along boundaries as a result of precipitation from solid solution during ingot cooling.

In the cast microstructure of V96Zr-3pch (with 0,13 % Fe; 0,04 % Si) insoluble coarse impurity – containing constituent particles along dendritic boundaries have also been revealed. They were identificated as $Al_{23}CuFe_4$ (see Figure 2) using electron probe microanalysis. The compound is in equilibration with aluminium solid solution in Al-Cu-Fe system (Figure 4); it is revealed as grey phase in optical microscope (Figure 3 a, b).

In the cast microstructure of high – strength V95pch alloy (with 0,3 % Mn; 0,18 % Fe) the particles of insoluble $Al_6(FeMn)$ phase were also detected.

As a consequence of local non-equilibrium eutectics, localized melting might have occurred during fast heating of ingots from superhigh – strength V96Zr-3pch alloy to temperature of 470 °C (Figure 3c). As shown the endothermic peaks in DSC thermograms (Figure 6), melting reactions are detected in 471 – 490 °C temperature interval (maximum heat flow at 480 – 482 °C) depending on dimensions and casting parameters of ingots. The homogenizing heating at 460 °C and long soak (>24 h) leads to complete remove of lightmelting eutectic.

In accordance with electron – diffraction analysis Al_3Zr (β') dispersoids (10-35 ηm) are coherent with matrix in V96Zr-3pch [2,8]. It was shown the Zr content should be limited to 0.13-0.14 % to prevent formation of primary $ZrAl_3$ intermetallics [4].

As shown by the investigations (Figure 6) and practical experience, V96Zr-3pch is characterized by good hot workability during extruding, rolling, forging. Proper parameters of homogenization insure high technological ductility of the alloy as a consequence of maximum dissolution and spheroidization of brittle coarse excessive constituent particles and due to optimum degree of solid solution precipitation.

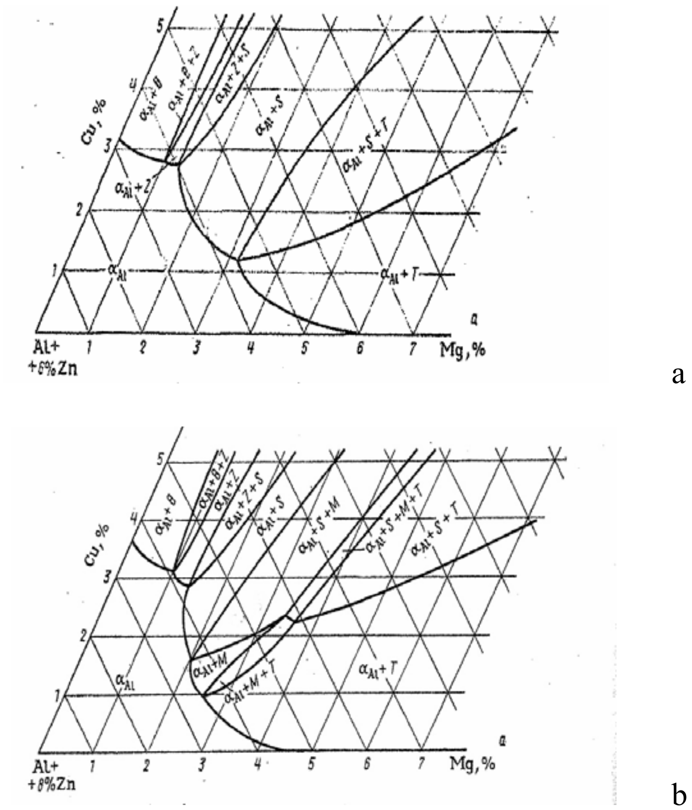


Figure 1: Isotherm section of Al-Zn-Mg-Cu system at 460 °C with 6 % Zn (a) and 8 % Zn (b) [6].

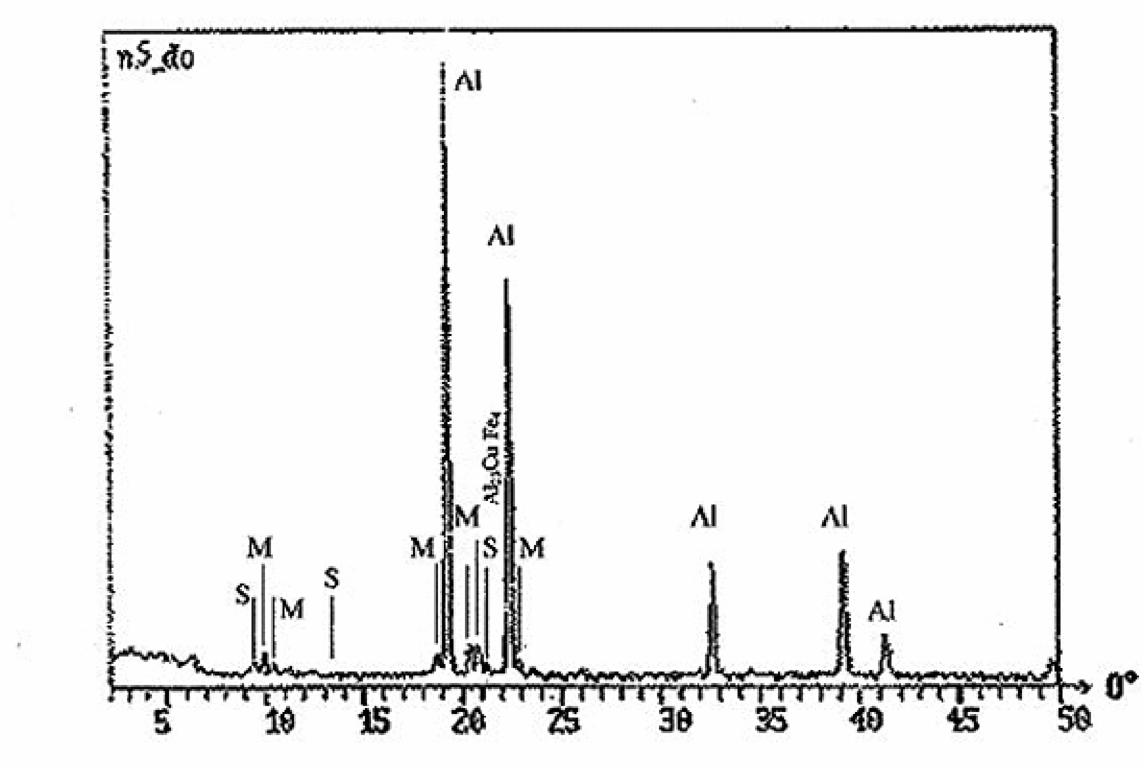
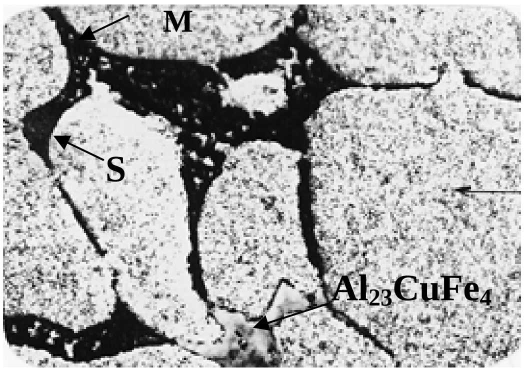
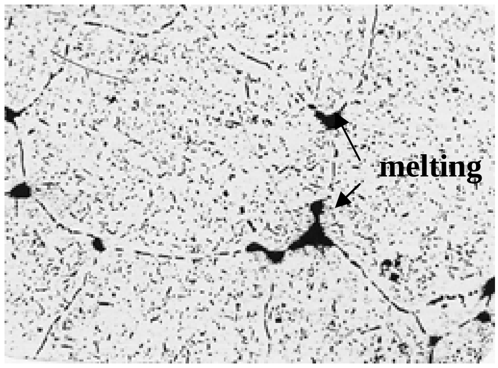


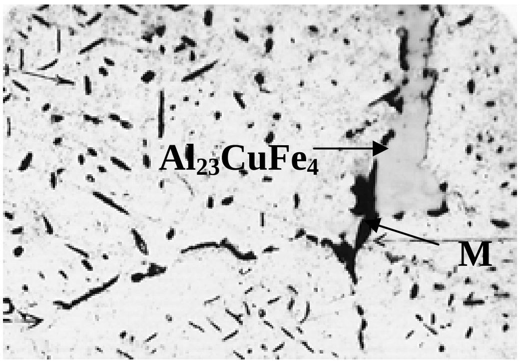
Figure 2: The XRD pattern of ingot sample from V96Zr-3pch – type alloy after preheating treatment (400 °C, 6 h).



a



c



b

Figure 3: Microstructure of samples after heating:
400 °C (a), ×800;
460 °C (b), ×500;
470 °C (c), ×200

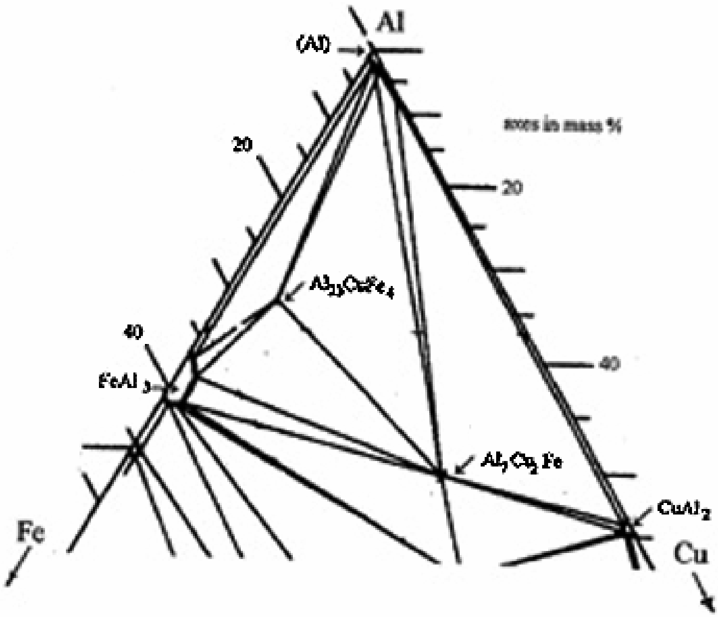


Figure 4: Isotherm section of Al-Cu-Fe system at 20 °C [7].

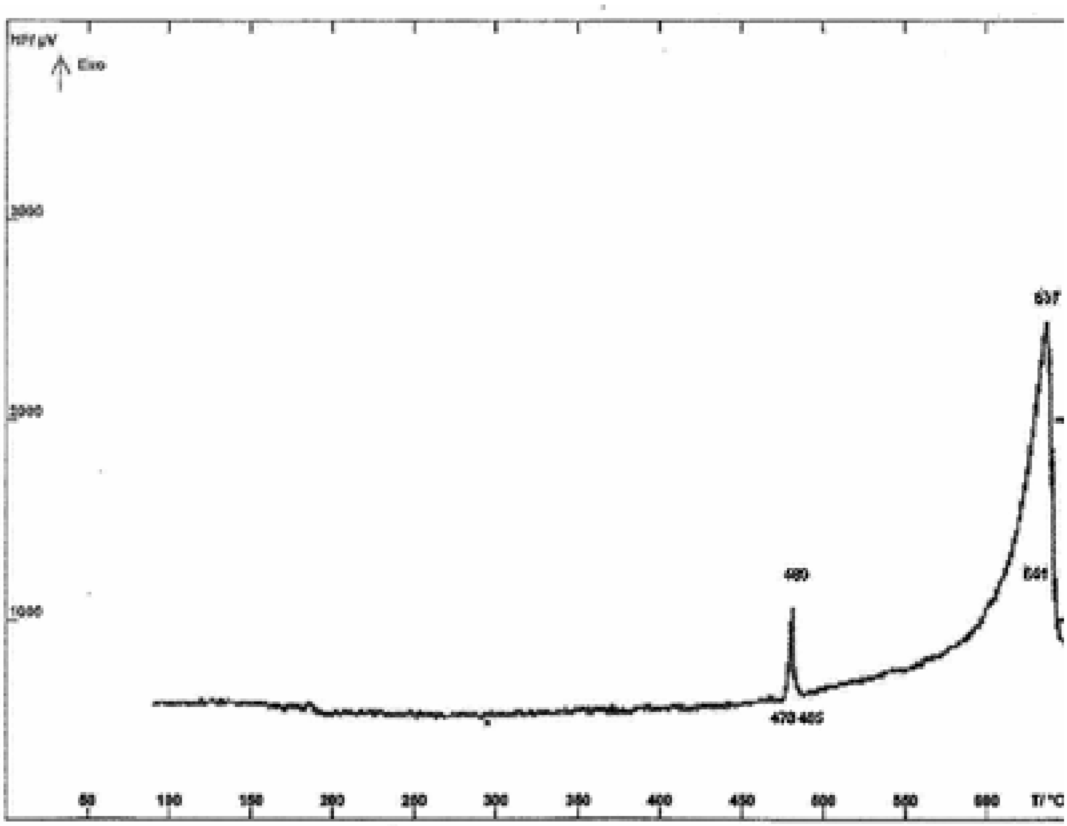


Figure 5: Example of a DSC trace recorded on ingot sample from superhigh-strength V96Zr-3pch – type alloy after preheating treatment (400 °C, 6 h).

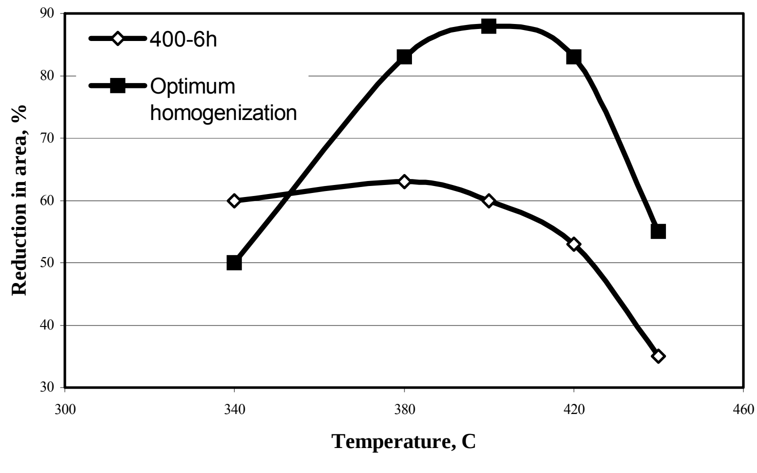


Figure 6: Hot ductility result under compession.

4. Conclusions

Important commercial alloys of 7xxx series belong to the Al-Zn-Mg-Cu-Zr system. Optimization of casting and homogenization practice is actual problem to insure good technological and final characteristics of these alloys.

After partial homogenization (400 ^{o}C, 6 h) of large-scale ingots of superhigh-strength V96Zr-3pch alloy (7449-type), excessive coarse soluble phase particles M [$Mg(ZnCuAl)_2$]

and S (Al_2CuMg) and nonsoluble phase particles Al_3CuFe_4 were detected along dendritic boundaries. They were correlated with the phase diagrams. Some amount of nonequilibrium local eutectic may be in the range of temperatures 471-490 ^oC; that's why ingot homogenizing must be kept low enough to avoid local melting.

Good hot workability of ingots was observed after optimum homogenization practice.

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