

## Method for Obtaining Ingots of the A34 Solder Based on an Investigation into the Relation between the Structure and Properties of Liquid and Solid Metals

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**Abstract**—A comparative analysis of the influence of the modifying method on the structure and phase composition of the ingots of the A34 solder is performed. Modification is performed by force of heating the melt to 1000°C and introducing the fine-crystalline remelt in an amount of 5% of the charge amount into the melt. The investigation is performed using DTA and metallographic analysis. The structure of the ingot of the A34 solder is additionally investigated at elevated temperatures (150 and 300°C) by the sc-AFM method using the NTEGRA Thermo probe nanolaboratory, and recommendations are given to optimize the modes of casting and thermal treatment.

**Keywords:** modification, microstructure, phase composition, scanning probe microscopy, differential thermal analysis

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The A34 solder is widely used to solder aluminum alloys [1] and is a refractory solder. Joints made of them are characterized by increased mechanical strength and resistance to atmospheric corrosion. According to *TU* (Technical Specifications) *I-92-46-76*, the A34 solder has the following composition: Al–(27–29)Cu–(5.5–6.5)Si, wt %;  $t_m = 525^\circ\text{C}$ . Its elemental composition is close to the eutectic composition for the Al–Cu–Si system [2]. The eutectic transformation  $L \rightarrow (\text{Al}) + \text{CuAl}_2 + (\text{Si})$ , according to various data, occurs at  $t = 520\text{--}525^\circ\text{C}$ , and the eutectic contains 26–31 Cu and 5.0–6.5 Si, wt %. Ternary compounds are not formed in the Al–Cu–Si system, and phases  $\text{CuAl}_2$  and Si are in equilibrium with the aluminum solid solution.

The main problem in producing the A34 ingots is the formation of a crude ternary eutectic (Al +  $\text{CuAl}_2$  + Si) and primary silicon crystals in their structure in the unmodified state [3]. These structural features substantially lower the manufacturing and operational properties of this solder. In order to optimize its production technology, we performed a comparative analysis of the influence of the modifying method on the structure and phase composition of the A34 ingots using the DTA and metallographic analysis. The modification was performed by two methods, namely, by force of overheating the melt to 1000°C and by the

introduction of charge materials with a fine-crystalline structure in an amount of 5% into the melt.

It has been known for a long time in the practice of foundry [4–10] that the overheating of the Al–Si melts to temperatures definite for each composition irreversibly transforms the melt into the state uniform at the atomic level (homogenizes), which leads to a substantial variation in the structure and the phase composition of the solid metal even with the subsequent cooling and crystallization even with moderate rates ( $\sim 1^\circ\text{C/s}$ ). According to [4, 10], the homogenization temperature of liquid metal ( $t^*$ ) is determined as the one corresponding to the beginning of the high-temperature coinciding segment of polytherms of heating and cooling of any structurally sensitive property of the melt, for example, viscosity.

It is known that, when heating silumin above 900°C and rapidly cooling it, it has a structure similar to the modified one [5]. The authors of [5, 6] explained this fact as follows. Solid particles of undissolved silicon or any other impurities are always present in the liquid melt. They always serve as the ready crystallization centers of silicon, and, in the course of this process, large needles of plates are formed from them. On the contrary, overheating to high temperatures leads to the destruction of these centers and, finally, a structure resembling the structure of the modified silumin is

formed. The fact that the degree of necessary overheating of the alloy to obtain the corresponding diminishment of silicon particles depends on the purity of metals used confirms the hypothesis [6] that the metal liquid has a microinhomogeneous structure.

Examples presented by the authors of [6] show that the temperature treatment of the melt is a simple method which does not require any additional facilities; it is available to improve the mechanical properties of the material in any conditions. A topical production problem is to determine the optimal conditions of the thermal treatment in the liquid state for each alloy. The authors of [4] described the scientifically substantiated method for determining the optimal conditions of the thermal treatment of metal in the liquid state. Its efficiency as applied to industrial silumins AL4 and AL9 in the liquid state to increase the level of mechanical properties using this method is described in [11].

In this study, using method [4] and based on an analysis of the temperature dependences of the kinematic viscosity of the A34 melt, which were found in the mode of heating and the subsequent cooling the sample, we determined its homogenization temperature of 950°C. Heating the A34 melt to  $t^* > 950^\circ\text{C}$  is considered a new independent method for improving the structure of cast metal. Since such overheating is directed to obtaining an ingot structure similar to the modified one, it should be expected that the additional modification of liquid metal will enhance this effect. With this purpose, we selected the known method, namely, the introduction of the fine-crystalline remelt (FCR) [12].

## EXPERIMENTAL

The specimens for investigations were sampled from the ingots of the A34 solder obtained at the Center of Modern Foundry Technologies, Samara State Technical University. We investigated four parties of the samples fabricated by various methods:

- (1) the standard method: overheating temperatures of the melt ( $t_o$ ) and casting ( $t_c$ ) are 600°C each;
- (2) overheating of the melt above homogenization temperature  $t^* = 950^\circ\text{C}$  at  $t_o = 1000^\circ\text{C}$  and  $t_c = 600^\circ\text{C}$ ;
- (3) the same + modifying with FCR additives in an amount of 5%;
- (4) the introduction of 5% FCR into the melt at  $t_h = t_c = 600^\circ\text{C}$ .

As the FCR, we used a ribbon made of the A34 alloy up to 2 mm thick, which was obtained in a roll crystallizer with a cooling rate of  $\sim 10^3$  K/s. In smeltings of series 2–4, the melt was held at  $t_o$  for 10 min and then heated in a crucible in air to  $t_c = 600^\circ\text{C}$  with a rate of  $\sim 10$  K/s. The modified additive (the ribbon made of the A34 alloy) was introduced into the melt at the casting temperature for 2 min before pouring. Then ingots of the A34 solder 4 mm in diameter and

150 mm in length were obtained by casting into a chill mold.

To investigate the structure and the phase composition of the samples, we used the following microscopes: a Neophot-32 optical microscope, an MBS-9 binocular microscope, an NTEGRA Thermo scanning probe microscope equipped with a thermostable, and a Zeiss AURIGA CrossBeam scanning electron microscope with the function of electron probe microanalysis (EPMA).

Microhardness was measured using a PMT-3 device at loads of 2N and 5N. Metallographic characteristics, namely, the volume fraction of the phases and average sizes of the grains and primary crystals, were determined using the standard procedures.

Thermograms were recorded using a VDTA-8M3 high-temperature differential thermal analyzer [13]. The experiments were performed for samples of about 1 g in weight shaped like cylinders  $\sim 7$  mm in diameter and  $\sim 10$  mm in height. We used crucibles made of beryllium oxide with an inner diameter of 7 mm and 13 mm in height. The working chamber of the thermal analyzer was preliminarily evacuated before each measurement to 0.001 Pa, and then helium was puffed to a pressure of  $\sim 10^5$  Pa. Thermograms were recorded in modes of heating and subsequent cooling the samples with the rates of 10, 20, and 40 K/min. We used the zone-refined tungsten single crystal (470–1100°C) and aluminum of the A999 grade (470–610°C) as the reference samples. Phase-transition temperatures were determined as the intersection points of the zero line of the thermogram ( $\Delta t = 0$ ) with the continuation of straight-line segments of endotherms and exotherms. Critical points were evaluated accurate to  $\pm 3^\circ\text{C}$ .

## RESULTS AND DISCUSSION

The DTA results for the samples of the A34 solder of series 1 and 2 are presented in Table 1, while the typical thermogram of the alloy is presented in Fig. 1.

The satellite peaks observed in Fig. 1 are possibly associated with various destruction stages of a microinhomogeneous structure of metal liquid [4]. Their area (Table 1) slightly varies for the samples of all four parties, which indicates the incompleteness of homogenization upon heating the melt above  $t^* = 950^\circ\text{C}$ . According to [14–16], the area of the thermogram peak determines the phase transition heat during sample melting and crystallization. According to the Gibbs–Folmer homogeneous nucleation theory, a decrease in the thermal effect during sample crystallization means an increase in the potential barrier height and the critical radius of the solid phase nucleus [17], which manifests itself in an increase in the supercooling of liquid metal on the crystallization front and leads to modifying the structure of cast metal even after slow cooling [8–10].

**Table 1.** DTA results

Parameters	Batch 1			Batch 2		
Reference substance—aluminum						
Heating rate, K/min	10	20	40	10	20	40
$t_s$ , °C	499	501	504	504	504	507
$t_L$ , °C	516	521	533	521	527	539
Peak area, mm <sup>2</sup>	57	126	216	51	106	233
Peak width, mm	6	8.5	17	6	8	12
Peak height, mm	26	34	51	26	33.5	47
Cooling rate, K/min	10	20	40	10	20	40
$t_s$ , °C	484	479	470	487	482	470
$t_L$ , °C	504	501	499	510	507	504
Peak area $S$ , mm <sup>2</sup>	53	106	201	40	90	214
Peak width, mm	7	9	13	7	8	12
Peak height, mm	26	35	47	27	36	47
Reference substance—tungsten						
Heating rate, K/min	40	80	40	80		
$t_s$ , °C	504	518	510	—		
$t_L$ , °C	541	562	544	—		
Peak area, mm <sup>2</sup>	227	561	248	—		
Peak width, mm	13	22	13	—		
Peak height, mm	43	65.5	50	—		
Cooling rate, K/min	40	80	40	80		
$t_s$ , °C	468	462	470	—		
$t_L$ , °C	499	496	504	—		
Peak area, mm <sup>2</sup>	160	338	221	—		
Peak width, mm	14	16	17	—		
Peak height, mm	50.5	69	52	—		

**Table 2.** Metallographic characteristics of the samples of the A34 solder

Batch no.	Preparation technology of the sample	Size of $\alpha$ -Al dendrites, $\mu\text{m}$	Eutectic microhardness, MPa	Size of the Si crystals, $\mu\text{m}$	Volume fraction of Si, %
1	$t_o = t_c = 600^\circ\text{C}$	327	2158	20	5.5
2	$t_o = 1000^\circ\text{C}$ , $t_c = 600^\circ\text{C}$	200	2020	17	4.3
3	$t_o = 1000^\circ\text{C}$ , $t_c = 600^\circ\text{C}$ , 5 wt % FCR	300	2530	14	6.9
4	$t_o = t_c = 600^\circ\text{C}$ , 5 wt % FCR	310	2059	17	5.8

The results of the metallographic investigation into the microstructure of the samples of the A34 solder, which were prepared by various methods, are presented in Table 2 and in Fig. 2.

The sample of batch 1 possesses a structure corresponding to the hypereutectic composition and includes the Si primary crystals, the  $\text{CuAl}_2$  primary dendrites, and a multiphase eutectic (which is based on the  $\alpha_{s,s} + \text{Si} + \text{CuAl}_2$ ). The Si primary crystals have

a faced morphology and complex growth forms are met, for example, a five-pointed star. Their distribution over the section is uniform, the volume fraction is 5.5%, and the average size is  $\sim 20 \mu\text{m}$ . Crystals of the  $\text{CuAl}_2$  intermetallic compound grow preferentially in a form of dendrites with a rounded section of branches. Their arrangement over the section is uniform. This phase is characterized by constancy of the angles between the branches of adjoining orders and

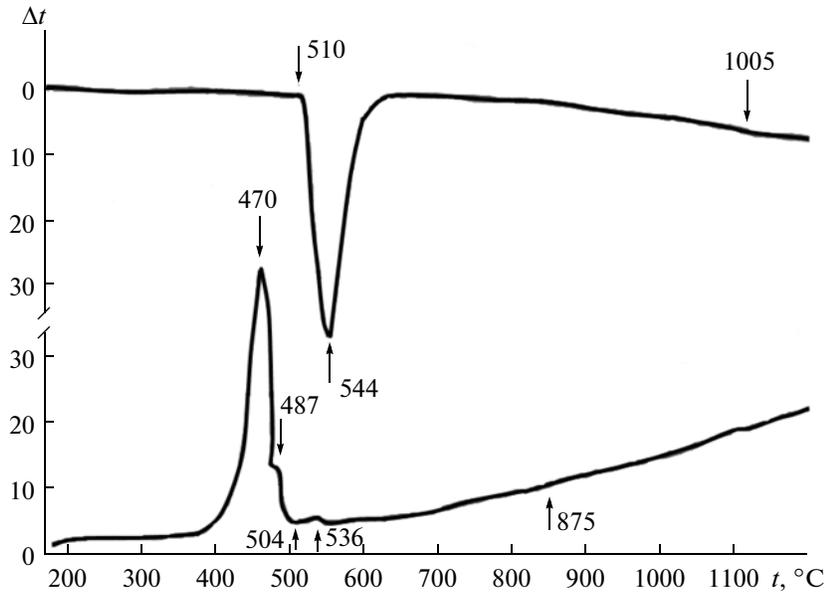


Fig. 1. Thermogram of the A34 alloy. Batch 2, reference substance is tungsten, and the heating rate is 20 K/min.

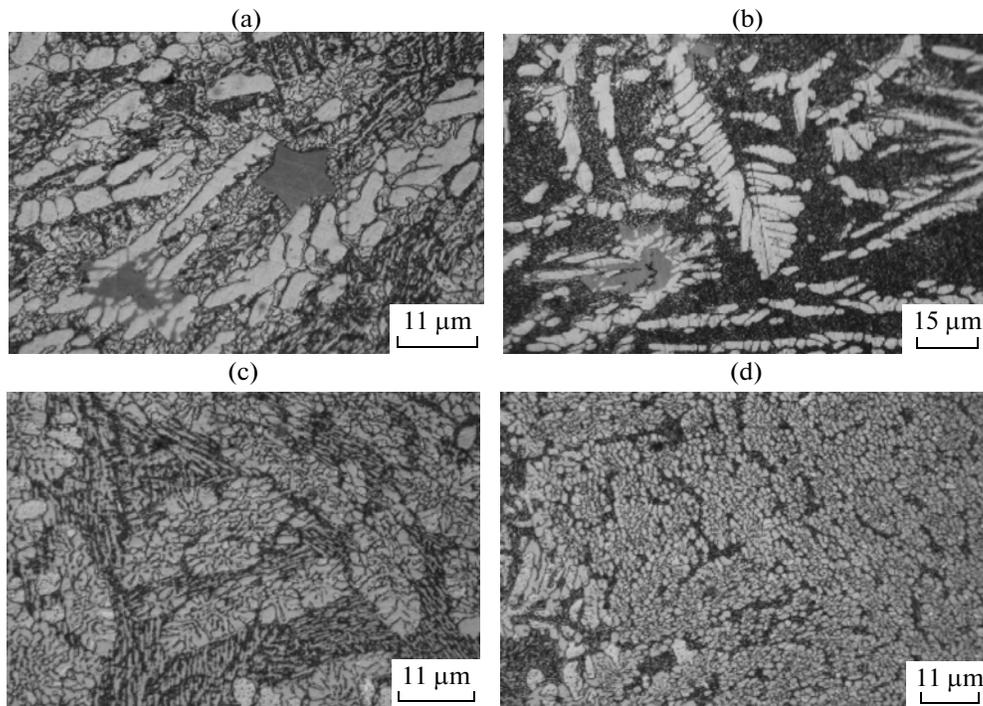


Fig. 2. Microstructure of the sample of the A34 alloy (optical microscopy). (a) Batch 1, (b) batch 2, (c) batch 3, and (d) batch 4.

the anisotropy of the growth rate, which manifests itself in the preferential development of the first-order trunks. The ternary eutectic has a cellular structure (Fig. 2a). Dark interlayers, which contain Si crystals, are arranged over the cell boundaries. It seems likely that the segregation in regards to silicon is observed in

the melt; it solidified in the last turn and the formed segregation zones emphasize the subgrain boundaries. Such eutectic can be referred to a “rosette” type with granular isolations along the cell boundaries.

The sample of phase 2 has a phase composition completely identical to the inhomogenized sample of

series 1. Its structure corresponds to the hypereutectic composition with the presence of Si crystals,  $\text{CuAl}_2$  dendrites, and a multiphase eutectic. A defect structure in the form of pores is seen for this sample. According to [5, 7], the method of diminishing the structure of silumins by overheating, as a rule, leads to the saturation of the solution with gases. The Si crystals are arranged nonuniformly over the sample section, and their amount is larger in the central part, where they are coarser. The volume fraction of these crystals is somewhat lower, i.e., the alloy has a composition closer to the eutectic one, which indicates a shift of the eutectic point into the region of lower values of Si and Cu. The  $\text{CuAlO}_2$  crystals retain their dendritic morphology, but the dendrites have thinner trunks and their number is smaller than for the sample of batch 1. Such structures are characteristic of large super cooling on the crystallization front.

The phase composition of all studied samples is similar. The values of microhardness of eutectics for the samples of series 1, 2, and 4 are close as the grain size ( $d = 300\text{--}200\ \mu\text{m}$ ). The sample of series 3 possesses the elevated microhardness when compared with other structures: 2530 against  $\sim 2000$  MPa (Table 2).

The primary silicon crystals are preferentially dendritic in the structure of the sample of batch 4 (homogenization + modification), which is characteristic of the high-temperature overheats of the melt. An insignificant diminishment in sizes of the primary Si phase and an increase in its volume fraction are fixed.

Thus, the comparative metallographic analysis of the samples of the A34 solder obtained by various methods showed that their phase composition is invariable. The homogenization of the melt affected the volume fraction, sizes, and morphology of isolations of the primary Si crystals. The passage from faced to dendritic forms of these crystals is observed.

The EPMA data for the samples of the A34 solder (batch 2), which were obtained from the overheated melt, indicate the anomalously high supersaturation of the  $\alpha$  solid solution with copper and silicon. The additional modification of the FCR insignificantly enhances the mentioned influence of homogenization of the metal liquid on the structure and the phase composition of crystallized samples of the A34 solder.

## CONCLUSIONS

(i) A new method of obtaining the A34 alloy, which is based on the overheating of liquid metal above the previously determined (from viscosity measurements) homogenization temperature  $t^* = 950^\circ\text{C}$ , is proposed.

(ii) It is shown that overheating of the A34 solder to  $t_0 = 1000^\circ\text{C} > t^*$ , even if cooling and crystallization are performed at slow rates, excludes the formation of the rough ternary eutectics ( $\text{Al} + \text{CuAl}_2 + \text{Si}$ ) and primary silicon crystals in the alloy structure. The addi-

tional modification of the A34 alloy by the force of introducing the fine-crystalline remelt into the melt somewhat enhances this effect, but the independent modification of the A34 alloy with the FCR additives is inefficient.

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