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MICROSTRUCTURE AND MICROHARDNESS OF Al-7 ALLOY WT% BI

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Abstract

The microstructure and microhardness of the Al–7 wt % Bi alloy crystallized at an average melt cooling rate of ~10 K/s has been studied. Bismuth and iron precipitates are mainly localized at the boundaries of aluminum grains, the average size of which is 40 μm. Bismuth particles have a spherical shape. The average value of the diameters of their cross sections is 1.5 μm. The specific surface of grain boundaries is 0.12 μm^{-1}. The specific surface area of the aluminum-bismuth interface is 0.062 μm^{-1}. The microhardness of the alloy is (323 ± 13) MPa and decreases monotonically during isothermal annealing at 150 °C.

Keywords: aluminum, bismuth, iron, silicon, grain, microstructure**,** microhardness.

МИКРОСТРУКТУРА И МИКРОТВЕРДОСТЬ СПЛАВА Al-7 МАСС.% BI

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Реферат

Исследована микроструктура и микротвердость сплава Al–7 масс.% Bi, закристаллизовавшегося при средней скорости охлаждения расплава ~10 К/с. Для изготовления сплава использовался алюминиевый лом, содержащий до 0,3 масс.% кремния и железа. Выделения висмута и железа преимущественно локализованы на границах зерен алюминия, средний размер которых равен 40 мкм. Частицы висмута имеют шарообразную форму. Среднее значение диаметров их сечений равно 1,5 мкм. Удельная поверхность границ зерен составляет 0,12 $мкм^{-1}$. Удельная поверхность межфазной границы алюминий-висмут равна 0,062 $мкм^{-1}$. Микротвердость сплава равняется (323±13) МПа и монотонно уменьшается в процессе изотермического отжига при 150 °С.

Ключевые слова: алюминий, висмут, железо, кремний, зерно, микроструктура**,** микротвердость.

Introduction

As known, the rapid melt-quenching method is to cool a jet of liquid metal on the outer (disc quenching) or inner (centrifugal quenching) surfaces of rotating drums, or rolling the melt between cold rolls made of materials with high thermal conductivity. In this method, a nanocrystalline structure is created in an amorphous alloy by crystallizing it. Spinning, i.e. obtaining thin foils of amorphous metal alloys by means of rapid (at a speed of at least 10^6 K/s) cooling of the melt on the surface of a rotating disk or drum is well developed. The most typical methods of obtaining amorphous tapes and wires by rapid cooling are: a) quenching on a rotating drum, b) extraction of the melt by a rotating drum, c) cooling of a thin jet of melt with liquid. The amorphous tapes and wires are then annealed at a controlled temperature to crystallize. To create a nanocrystalline structure, annealing is carried out in such a way that a large number of crystallization centers arise, and the crystal growth rate is low.

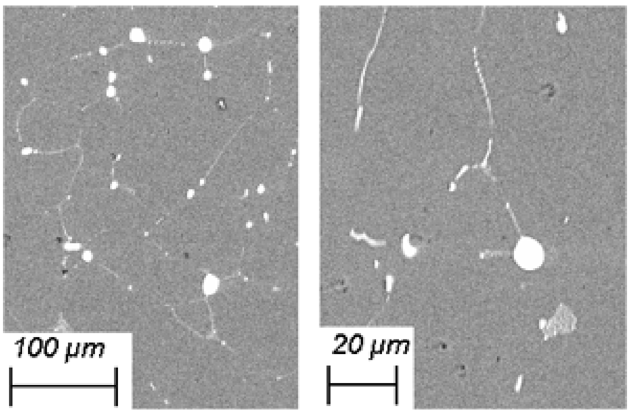
Aluminum alloys containing indium, lead, and bismuth (Al–In, Al–Pb, and Al–Bi) have not been sufficiently studied, due to their limited use in industry. But in the last two decades, scientific and practical interest in them has manifested itself [1-4]. For example, alloys of the Al–Pb system are used as antifriction and damping materials. At the same time, their mechanical and operational characteristics are determined by both the chemical composition and size, morphology and phase distribution, as well as operating conditions. Structural defects (dislocations, grain boundaries, stacking faults, etc.) also affect their mechanical and electrochemical properties. It has been established that aluminum and bismuth alloys can be used as anode materials in the protection of metals, such as iron and steel, from corrosion, which is of great practical importance in various industries, construction and transport. It has been experimentally revealed that alloys of the aluminum-bismuth system interact with water under certain conditions, causing the release of heat and hydrogen, which is of great practical importance for the development of hydrogen energy. At the same time, the possibilities of using hydrogen in various fields of human activity, for example, in medicine, motor transport, etc., are expanding. It has also been established that such an interaction depends on temperature and pressure, as well as on the chemical composition of the alloy and its microstructure formed during crystallization and heat treatment [5–9]. When water and aluminum interact, various compounds are formed, including aluminum oxides, which can also be used in industry. To reduce the cost of hydrogen production, it is planned to use aluminum scrap (for example, aluminum tubes from end-of-life refrigeration units, aluminum wire from exhausted power lines, disused aluminum cookware, end-of-life structural aluminum products, etc.) instead of aluminum obtained using expensive electrolysis. The concentration of silicon and iron in the aluminum tube is ≈ 0.3 wt.%. In this regard, a study of the microstructure and microhardness of the Al–7 wt.% Bi alloy, made on the basis of aluminum tubes containing ≈ 0.3 wt.% of silicon and iron, and bismuth with a purity of 99.999%, was carried out, and its thermal stability was investigated by measuring the microhardness of the alloy during isochronous and isothermal annealing.

Experimental methods

Al–7 wt.% Bi alloy is obtained by fusion of aluminum tubes and bismuth with a purity of 99.999 % at a temperature of 750 °C. Then the melt was poured at room temperature into a graphite mold, where it crystallized in the form of an ingot with a cross section of 6×6 mm^2 and a length of 7 cm. The average melt cooling rate was ~10 K/s. Samples were cut from the middle part of the ingots to study the microstructure. The microstructure of the alloy was studied using a scanning electron microscope LEO-1455 VP. The microscope has an attachment for X-ray spectral microanalysis. The operating voltage of the electron microscope is 20 kV. The surface of the section was polished with a special paste containing dispersed solid particles of the abrasive substance. The microstructure parameters (mean chord of aluminum grains and bismuth precipitates, specific surface area of aluminum grain boundaries and aluminum–bismuth interphase boundary) were determined using a stereometric analysis of measurements obtained by the method of random secants [9]. The relative error in measuring the parameters of the microstructure was 8–15 %. Microhardness measurements were performed on a microhardness tester (marking in Russian language ПМТ-3) using a load of 20 g. The load time for measuring microhardness is 80 s. The microhardness value was calculated by measuring the diagonals of ten indenter imprints on the polished surface of the alloy under study. The relative error of its determination was 4 %. The thermal stability of the resulting alloy was investigated by microhardness using isochronous annealing carried out from 20 to 160 °C through 20 °C and holding at each temperature for 30 minutes, and isothermal annealing carried out at a temperature of 150 °C for 16 hours.

Results and discussion

Images of the section of the studied alloy Al–7 wt.% Bi at various magnifications are shown in Fig. 1 (a, b). At high magnifications (Fig. 1 (b)) there is a dark background, white and gray precipitates. The distribution of components along the electron beam scanning line over the surface of the alloy under study is shown in Fig. 2. The dark background matches the aluminum. White precipitates, as shown by X-ray spectral microanalysis, correspond to bismuth. The boundaries of aluminum grains are decorated with bright precipitates of other phases. The average chord of the sections of aluminum grains is d_A = 25 µm, the calculated average size of aluminum grains is D = 40 µm. The specific surface area of aluminum grain boundaries is S_A = 0.12 µm^{-1}. Most bismuth particles are spherical in shape. The average value of the diameters of their cross sections is d_B = 1.5 µm. The specific surface area of the aluminum-bismuth interface is $S_{A\text{-}B}$ = 0.062 µm^{-1}, which is two times less than S_A.



(a) (b)

Figure 1 – Precipitates of bismuth and iron phases at the grain boundaries of aluminum (a) and (b) alloy Al–7 wt.% Bi at different magnifications

Precipitates with a gray tint and a striped structure in the area of their accumulation contain iron. In these areas, as follows from the distribution (Fig. 2), the average concentration of iron reaches 10 wt.%. The silicon distribution is characterized by insignificant peaks (up to 2 wt.%) located at a distance of ~1 µm from each other, which indicates a more uniform distribution of silicon precipitates than the distributions of bismuth and iron precipitates in the Al–7 wt.% Bi alloy under study after crystallization at a cooling rate of 10 K/s.

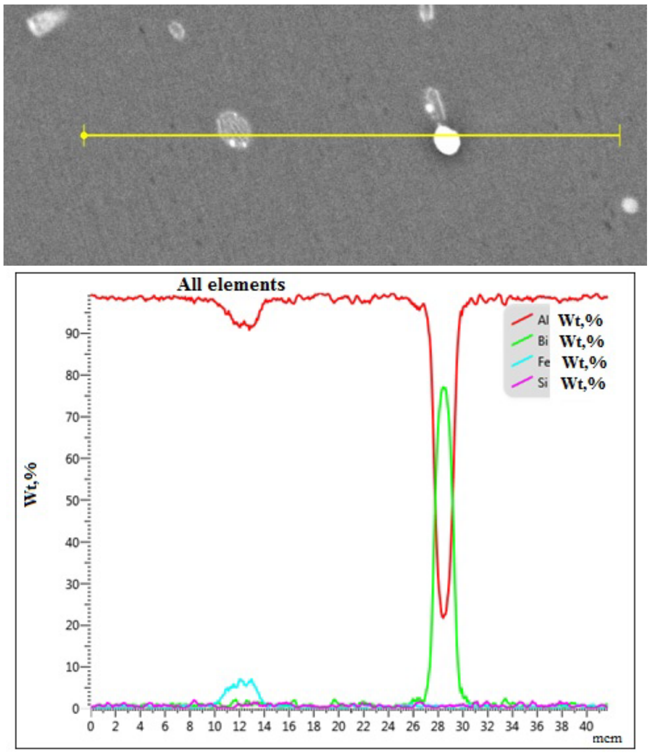


Figure 2 – Distribution of components along the electron beam scanning line on the surface of the Al–7 wt.% Bi alloy

The distribution of chord lengths of random secants on bismuth precipitates by size groups is shown in Fig. 3. 3. The total number of images of bismuth precipitates used in the construction of the histogram was at least 150. The largest proportion (0.27) of chords falls on the group with a minimum size of 0.5 µm. With an increase in the length of chords in bismuth sections, their fraction decreases. The proportion of group chords with their maximum length (4 µm) is 0.05. The distances between bismuth precipitates reach 2 µm or more.

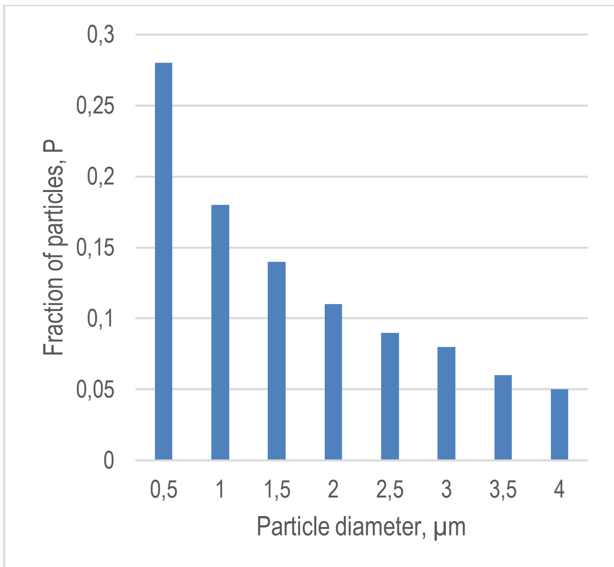


Figure 3 – Distribution of chord lengths of sections of bismuth particles of Al–7 wt.% Bi alloy by size groups

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The concentrations of the components in different parts of the section of the Al–7 wt.% Bi alloy under study were determined (Fig. 4). Their values are presented in Table 1. In dark areas (spectra 3 and 4), the concentration of aluminum reaches 99.5 wt.%, and the concentrations of silicon and iron are equal to 0.5 wt.% and less than 0.03 wt.%, respectively, which is associated with a slight equilibrium solubility of these elements in aluminum and cooling of the alloy at a rate of 10 K/s. In the white area (spectrum 5), the concentration of bismuth is 98.0 wt.%, the concentration of aluminum is 1.7 wt.%, the concentrations of silicon and iron reach 0.5 and 0.03 wt. %, respectively. The value of the aluminum concentration in bismuth is overestimated, which is due to the excitation of the aluminum surrounding this bismuth precipitate by an electron beam.

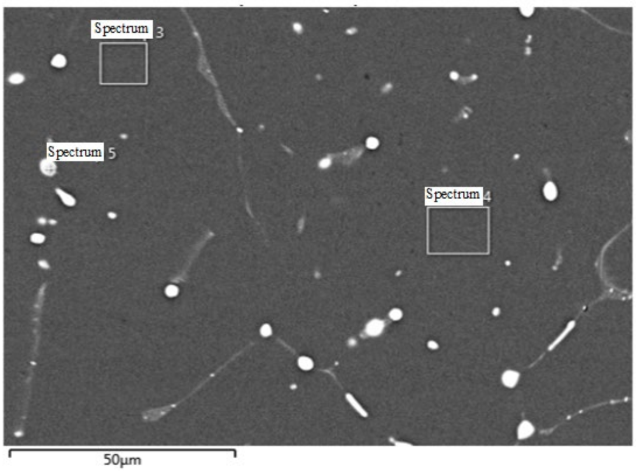

Figure 4 – Image of the surface of the section of the alloy Al–7 wt.% Bi

Table 1 – Concentration of components in various areas of the section of the Al–7 wt.% Bi alloy

Area	Concentration of components, wt.%			
	Al	Bi	Si	Fe
Spectrum 3	99,40	0,09	0,51	0,00
Spectrum 4	99,46	0,02	0,49	0,03
Spectrum 5	1,69	97,96	0,15	0,19

The formation of an inhomogeneous structure in a bulk Al(Fe, Si)–7 wt % Bi alloy is due to the peculiarities of the phase diagram of the Al–Bi system [11]. The mutual solubility of the components in the solid state is less than 1 mass%. In the alloys of the system, there is a stratification of the liquid phase L into two liquids L_1 and L_2, differing in composition, when heated above 657°C. The volume fraction of liquid L_1 is much larger than the volume fraction of liquid L_2. When the melt is cooled below 657°C, the monotectic transformation of the L_1 liquid first occurs. In this case, aluminum is first released, and the bismuth and iron atoms are pushed to the boundaries of aluminum grains. The bismuth-rich L_2 liquid undergoes a eutectic transformation when further cooled below 270°C, in which bismuth and aluminum are released. Bismuth precipitates are larger, preferably located in areas located at the junctions of three grains. The released aluminum is attached to the grains of aluminum, which were formed earlier during monotectic transformation. Iron precipitates, as well as their accumulations, are predominantly located at the boundaries of aluminum grains. There are accumulations of dispersed precipitates of bismuth and iron located at the grain boundaries.

The microhardness of the Al–7 wt.% Bi alloy under study is equal to (323±13) MPa. When carrying out isochronous annealing in the temperature range of 100–160°C, a monotonic decrease in the microhardness of the alloy is observed.

Isothermal annealing of the alloy under study, carried out at a temperature of 150 °C, also causes a monotonous decrease in microhardness (Fig. 5). The change in microhardness at the initial stage of isothermal annealing is described by the ratio

$$(H_0 - H_t) = (H_0 - H_k)\ exp(-at),$$

where H_0, H_k и N_t are the microhardness values at initial, final and current time t of isothermal annealing. The calculation showed that the value of the coefficient a = 0.3.

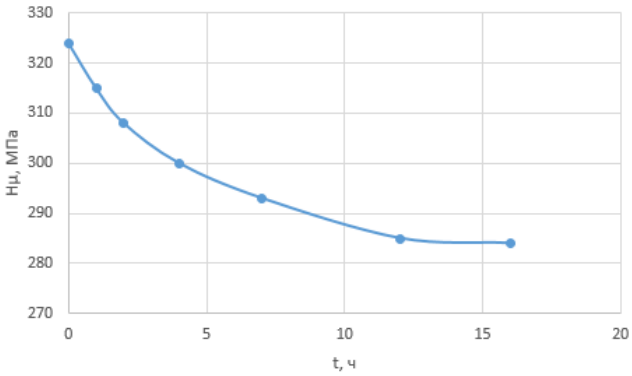

Figure 5 – Dependence of the microhardness of the Al–7 wt.% Bi alloy on the holding time during isothermal annealing

The ratio of the temperatures of isothermal annealing and the beginning of melting of the alloy under study on the Kelvin scale is ≈ 0.45, i.e. at a temperature of 150°C, diffusion processes actively occur in the volume of grains and their boundaries of the alloy, causing the dissolution of small particles of the second phase (bismuth) and the growth of its larger particles [12]. This reduces the total number of bismuth particles and increases their average size and distances between them. Therefore, this process is energetically advantageous and leads to a decrease in the contribution of the dispersion mechanism to the hardening of the alloy, thereby causing a decrease in microhardness during isochronous and isothermal annealing [12, 13].

Conclusions

Crystallization of Al–7 wt. % Bi, made on the basis of aluminum scrap, in which the concentration of iron and silicon is ≈ 0.3 wt. %, at an average cooling rate of ~10 K/s leads to the formation of a microcrystalline structure. The average chord of the sections of aluminum grains is d_{Al} = 25 μm, the average size of aluminum grains is 40 μm. The specific surface of aluminum grain boundaries is S_{Al} = 0.12 μm^{-1}. Predominantly dispersed precipitates of bismuth and iron are localized at the grain boundaries. The distribution of silicon in the alloy is more uniform compared to the distribution of iron and bismuth precipitates. The average value of bismuth particle diameters is d_{Bi} = 1.5 μm. The specific surface area of the aluminum-bismuth interface is S_{Al-Bi} = 0.062 μm^{-1}. During ichohronous annealing in the temperature range of 100–150°C and during isothermal annealing at a temperature of 150°C, a decrease in microhardness occurs due to the coarsening of bismuth particles and a decrease in their dispersion contribution to the hardening of the alloy.

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