

# HEAT-RESISTING ALLOYS

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## INVESTIGATION OF THE MICROSTRUCTURE AND PROPERTIES OF CASTABLE NEODYMIUM- AND YTTRIUM-BEARING MAGNESIUM ALLOYS AT ELEVATED TEMPERATURES

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The properties, structure, and special features of the technological process of native (ML10, ML19) and imported (*WE43*, *WE54*) castable high-temperature magnesium alloys containing neodymium and yttrium are investigated. Recommendations are given for the use of these alloys in the aircraft industry and some other fields of basic engineering (automobile industry).

Studies of castable magnesium alloys possessing a quite high level of mechanical properties at elevated temperatures are conducted in many countries (see, for example, [1], [2]). The use of these alloys makes it possible to decrease considerably the mass of parts, increase the reliability of units and structures including cast preforms, and decrease the laboriousness of their production. The use of such materials is especially effective in parts located in aircraft zones (mostly close to operating engines) heated to a high (as applied to magnesium alloys) temperature. The alloying of magnesium by rare-earth elements (neodymium and yttrium) combined with zirconium increases the creep resistance of these alloys at a temperature of 250°C and higher [3, 4].

Cast preforms of parts (castings) operating in aircraft at a temperature of at most 250°C are commonly made of high-strength magnesium alloy ML10 developed on the basis of a Mg–Nd–Zn–Zr system. In addition to the high level of high-temperature strength, alloy ML10 has quite high mechanical properties at the operating temperature of aircraft ( $\pm 60^\circ\text{C}$ ). For this reason it is used for the production of cast preforms of critical fuselage and wing parts, control and pressure-regulating parts of helicopters, base parts of fuel-supply and hydraulic systems of planes and helicopters.

Castings from alloy ML10 are characterized by a high impermeability, the possibility of operation at a high temperature, and stability of the sizes of parts in combination with a high corrosion resistance.

In foreign countries the counterparts of alloys of the ML10 type are *QE21*, *QE22* (the Mg–Nd–Zr–Ag system) and *QH21* (the Mg–Nd–Zr–Th system). They pos-

sess a more complex chemical composition than ML10 and contain expensive elements (thorium and silver).

In Russia parts of aircraft engines subjected to a long-term temperature effect (up to 350°C) are made of alloy ML19 of the Mg–Nd–Y–Zr system, in foreign countries the counterparts are *WE43* and *WE54* alloyed by yttrium, neodymium, and other rare-earth elements (gadolinium, erbium, dysprosium) [2].

We studied the microstructure and properties of alloys ML10 and ML19 at room temperature and at elevated temperatures for individually cast specimens 10 and 12 mm in diameter and for specimens cut from castings. The mass of the laboratory heats was 10–20 kg, and that of the industrial heats was 200–300 kg. The alloys were melted by two tech-

TABLE 1

Heat	Content of elements,* %			
	Nd	Y	Zr	Zn
Alloy ML10				
1	2.60	—	0.52	0.30
2	2.76	—	0.48	0.40
3	2.80	—	0.49	0.40
4	2.55	—	0.50	0.30
As specified	2.2–2.8	—	0.4–0.1	0.1–0.7
Alloy ML19				
5	2.10	2.1	0.52	0.40
6	2.30	2.0	0.50	0.41
7	2.00	1.7	0.40	0.45
As specified	1.6–2.3	1.4–2.2	0.4–1.0	0.1–0.6

\* The remainder is Mg.

Note. In addition to the listed elements the alloys contained the following impurities (at most): 0.01% Fe, 0.03% Si, 0.03% Cu, 0.005% Ni, 0.02% Al, and 0.001% Be.

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nologies with the use of a flux and without it in a protective medium containing up to 1% SF<sub>6</sub>.

Zirconium was introduced into alloys ML10 and ML19 from a Mg–Zr alloying composition with 15–20% zirconium. Neodymium was introduced from a Mg–Nd alloying composition, and yttrium was introduced from a Mg–Y one.

We determined the chemical composition of the alloys, the structure, the mechanical properties at room and elevated temperatures and studied macrofractures of technological specimens. The specimens were tested in a cast state and after heat treatment by regime T6 (quenching + aging). The structure and the phase composition of the alloys were studied using a light microscope and a UKhA-840 electron microanalyzer.

For comparison, we studied the microstructure and the phase composition of imported alloys WE43 and WE54.

The corrosion properties of the alloys were tested after holding the specimens in a 3% solution of sodium chloride. The tests for flux corrosion were conducted in a chamber with an elevated moisture content (95–100%) at 20°C for 48 h. The density of the alloys (the microporosity) and the presence of internal defects in the specimens (slags, oxide spots, etc.) were determined in fractures by the method of x-ray control and using a special Dobatkin specimen.

The chemical composition of alloys ML10 and ML19 is presented in Table 1, and the physical and technological properties are given in Table 2.

Since the production of alloys ML10 and ML19 is not too complicated, the chemical composition of industrial heats virtually always corresponds to the composition of the charge. Table 1 presents typical compositions of industrial heats of alloys ML10 and ML19.

Table 3 presents the mechanical properties of separately cast specimens of alloy ML10 T6 produced with the use of different refinement methods.

Table 4 presents mechanical and corrosion properties of specimens of alloy ML10T6 cut from parts cast with the use of flux-free melting under industrial conditions.

It follows from Tables 3 and 4 that specimens of alloy ML10 obtained in melting without a flux and with the use of protective and refining fluxes possess close mechanical properties that meet the specifications. The melting technology without a flux is preferable, because it is safer ecologically and eliminates flux corrosion while increasing the total corrosion resistance of alloy ML10. This is connected with the absence of chlorine compounds, which are contained in protective and refining fluxes.

Table 5 presents the mean values of mechanical properties of separately cast specimens of alloy ML19 T6.

Table 6 presents the results of corrosion tests of specimens of alloy ML19 in different states in a 3% solution of NaCl.

TABLE 2

Alloy	$\gamma$ , g/cm <sup>3</sup>	$\alpha \times 10^{-6}$		$\lambda$ , J/(cm · sec · K)			Linear shrinka- ge, %	Hot- shortness, mm	Flow- ability, mm
		20–100°C	100–200°C	100°C	200°C	300°C			
ML10	1.78	27.7	28.7	1.13	–	–	1.2–1.5	15–20	250
ML19	1.79	26.9	28.6	0.89	0.92	0.96	1.2–1.5	20	250–300

Notation:  $\gamma$ ) specific mass of the alloy,  $\alpha$ ) coefficient of linear expansion,  $\lambda$ ) thermal conductivity.

Note. The hot-shortness is represented by the width of rings, and the flowability is represented by the length of rods.

TABLE 3

Method of refining in melting	$\sigma_r$ , N/mm <sup>2</sup>	$\sigma_{0.2}$ , N/mm <sup>2</sup>	$\delta$ , %
Without a flux	240–270	140–160	3.5–5.0
With a flux	235–260	140–160	3.5–5.0
As specified	230	140	3.0

Note. The mean properties after testing specimens from 10 heats of alloy ML10 T6 are presented.

TABLE 4

Part	$\sigma_r$ , N/mm <sup>2</sup>	$\delta$ , %	$\Delta m$ , g/(m <sup>2</sup> · day)	$H$ , cm <sup>3</sup> /(cm <sup>2</sup> · day)
Support	265	5.2	7.5	0.59
Plate	275	7.8	6.0	0.48
Casing	275	5.6	4.8	0.41

Notation.  $\Delta m$  and  $H$  are the decreases in the mass and in the amount of the emitted hydrogen in corrosion tests of alloy ML10T6.

TABLE 5

Specimen of alloy ML19T6	$\sigma_r$ , N/mm <sup>2</sup>	$\sigma_{0.2}$ , N/mm <sup>2</sup>	$\delta$ , %
1	275	195	3.0
2	255	175	3.0
3	265	175	3.3
As specified	220	120	3.0

TABLE 6

Specimen	$\Delta m$ , g/(m <sup>2</sup> · day)	$H_2$ , cm <sup>3</sup> /(cm <sup>2</sup> · day)
1	5.0	0.50
	4.6	0.40
2	5.3	0.50
	4.8	0.40
3	9.6	0.80
	9.0	0.75
4	9.5	0.81
	9.0	0.67
5	8.0	0.75
	7.0	0.65

Note. The numerators present the properties of alloy ML19 in a cast state, the denominators present the values in state T6.

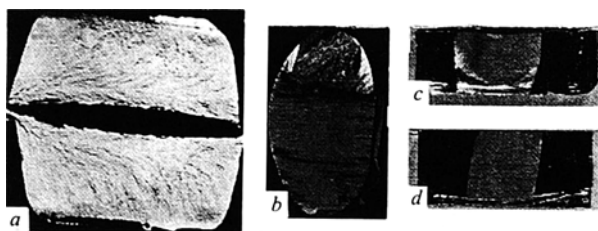


Fig. 1. Macrofractures in alloys ML10 (a), ML19 (b), WE43 (c) and WE54 (d).

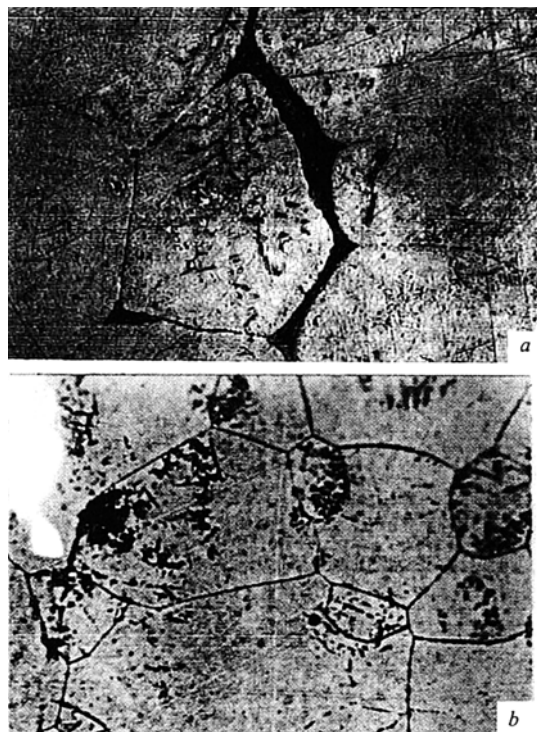


Fig. 2. Microstructure of alloy ML10 in cast (a) and heat-treated (T6) (b) state,  $\times 800$ .

A certain scattering of the parameters of corrosion resistance is explainable by the difference in the purities of the charged materials. Alloy ML19 has a higher corrosion resistance than alloy ML5 (AZ 91).

The quality of molten alloys was estimated by methods of x-ray control and visually by the structure of rupture surfaces. It was established (Fig. 1) that alloys in a cast state exhibit fine-grain pure ruptures without slag and nonmetallic inclusions.

The results of microstructural analysis of the alloys are presented in Figs. 2–4.

Alloys ML10 and ML19 are strengthened by heat treatment and used in states T6 and T61 (Table 7).

The phase composition of alloy ML10 is represented by a solid solution of neodymium, zinc, and zirconium in magnesium over the boundaries of which a stronger eutectic mixture

TABLE 7

Alloy	State	Heat treatment regime
ML10	T6	Quenching from $540 \pm 5^\circ\text{C}$ (8–12 h) in air, aging at $205 \pm 5^\circ\text{C}$ for 12–18 h
	T61	Quenching from $545 \pm 5^\circ\text{C}$ (4–8 h) in water with $t_w \geq 80^\circ\text{C}$ , aging at $205 \pm 5^\circ\text{C}$ for 8–12 h
ML19	T6	Quenching from $535 \pm 5^\circ\text{C}$ (4–8 h) in air, aging at $205 \pm 5^\circ\text{C}$ for 8–16 h

Note. After aging by all the regimes the cooling was conducted in air.

of the same solid solution and the  $\text{Mg}_{12}\text{Nd}$ ,  $(\text{MgZn})_{12}\text{Nd}$  phases is positioned (Fig. 2).

Alloy ML19 does not contain toxic and scarce additives and its high-temperature characteristics exceed those of alloys of the Mg–Al and Mg–Zn–Zr systems and of alloy ML10. The high-temperature strength of alloy ML19 is explained by the presence of Y, Nd, and Zr in its composition, which form a complexly alloyed thermally stable  $\alpha$ -solid solution in magnesium and  $\text{Mg}_{12}\text{Nd}$  and  $\text{Mg}_{24}\text{Y}_5$  strengthening phases contained in the composition of binary  $[\alpha + \text{Mg}_{12}\text{Nd}]$  and  $[\alpha + \text{Mg}_{24}\text{Y}_5]$  eutectics. At a certain proportion of components in the alloy a triple  $[\alpha + \text{Mg}_{12}\text{Nd} + \text{Mg}_{24}\text{Y}_5]$  eutectic can be formed.

The microstructure of high-temperature alloys ML10 and ML19 in the cast state consists of grains of an  $\alpha$ -solid solution of Zn, Nd, Y, and Zr in magnesium and nets of excess phases formed as a result of degeneration of the eutectics. The net forms a rigid skeleton of refractory compounds of magnesium with the alloying components, which hampers shear processes at elevated temperatures, providing a high high-temperature strength in the cast state.

Heat resistant magnesium alloys are strengthened by heat treatment that consists of quenching followed by an artificial aging. In heating for quenching the excess phases pass into the solid solution. In the aging process refractory phases in the form of finely dispersed submicroscopic particles are segregated and create microheterogeneities inside the grains of the solid solution, blocking diffusion and shear processes at elevated temperatures. This improves the mechanical properties of the alloys at a high temperature, i.e., the ultimate long-term strength and the creep resistance. According to the data of N. M. Tikhova, V. A. Blokhina, and A. P. Antipova (All-Russian Institute of Aircraft Materials) a 100-h hold at  $250^\circ\text{C}$  virtually does not change the high-temperature strength of alloy ML10. An increase in the heating temperature to  $300$ – $350^\circ\text{C}$  decreases this characteristic considerably. After a hold at  $250^\circ\text{C}$  disperse particles of the  $\text{Mg}_{12}\text{Nd}$  strengthening phase segregate inside grains of the solid solution. As the heating temperature is increased to  $300^\circ\text{C}$ , this phase begins to dissolve partially, and the remaining particles coagulate, making the structure coarser and thus decreasing the high-temperature strength of the alloy. With an increase in the test temperature to  $350^\circ\text{C}$  the disperse particles dissolve completely, and the retained ones become still coarser. The com-

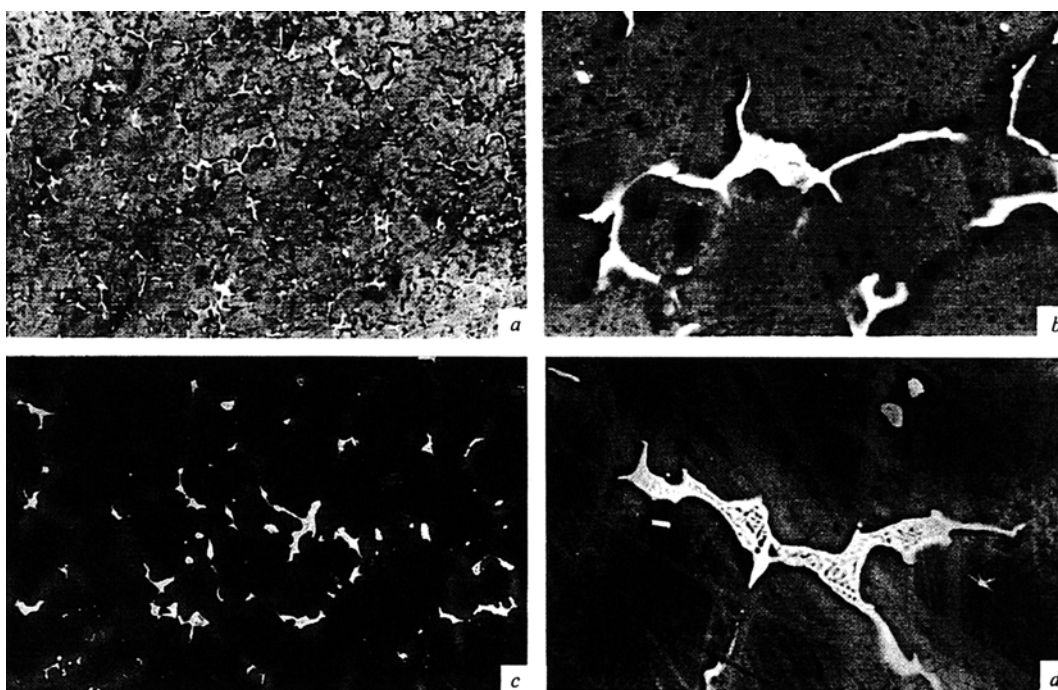


Fig. 3. Microstructure of alloys ML19 (*a, b*) and WE54 (*c, d*) in cast state: *a, c*)  $\times 200$ ; *b*)  $\times 1500$ ; *d*)  $\times 800$ .

position of the eutectic, segregated over grain boundaries, changes; it laminates and partially decomposes. The creep resistance of the alloy decreases.

An yttrium additive to a manganese alloy containing neodymium and Zr (ML19, WE43 and other alloys) promotes grain disintegration in the cast and heat treated states and stabilizes the strengthening phase at elevated temperatures. As a result, the strength and the ductility of the alloys at elevated temperatures increase.

The results of a local x-ray spectral analysis of alloys ML19, WE43, WE54 conducted using a UKhA-840 microanalyzer are presented in Table 8. The local spectral analysis was conducted over the surface of a microscopic specimen for a region of the solid solution and at a point.



Fig. 4. Microstructure of alloy WE43 in heat-treated state,  $\times 1500$ .

TABLE 8

Alloy	State	Content of elements, at.%									Note
		Mg	Nd	Y	Zr	Zn	La	Cd	Er	Dy	
Solid solution											
ML19	Cast	98.6	0.36	0.5	0.15	0.2	—	—	—	—	Analysis of the specimen surface
WE43	HT	97.3	0.50	1.6	0.18	—	—	—	—		
WE54	Cast	97.3	0.30	1.9	0.20	—	—	—	—		
Phase 1											
ML19	Cast	89.9	4.9	2.5	—	3.1	—	—	—	—	Analysis at a point
WE43	HT	91.3	6.0	1.0	0.08*	—	0.8	—	—	—	
WE54	Cast	86.6	5.0	7.4	0.20	—	0.4	0.1*	0.09*	0.07*	
Phase 2											
WE43	HT	54.4	1.9	42.0	0.5	—	—	0.3	0.4	0.7	
WE54	Cast	42.0	1.0	53.8	1.0	—	—	0.5	0.7	1.0	

\* Within the scale of the measuring instrument.

Notation: HT is used for heat treatment.

Note. The analysis of the solid solution on the surface of the specimens did not show the presence of La, Gd, Er, or Dy.

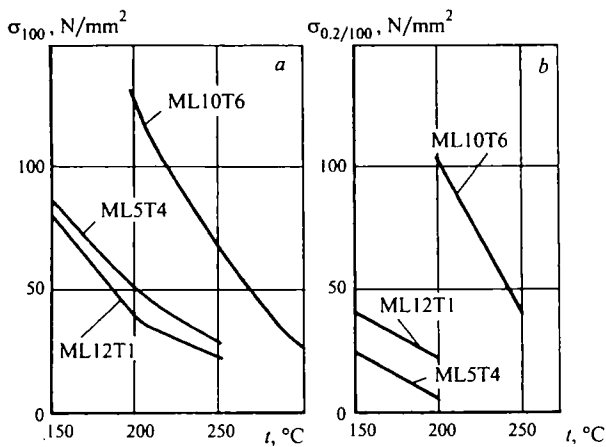


Fig. 5. Curves describing the long-term strength (a) and creep (b) in cast magnesium alloys ML5T4, ML10T6, and ML12T1 (100-h tests).

An analysis of the microstructure of alloys ML19, WE43, and WE54 has shown that the solid solution of the alloys in the cast state consists of magnesium (90.6–94.6%), yttrium, neodymium, and zirconium. The strengthening phase, based on magnesium (58–80%) and positioned over grain boundaries, contains yttrium, neodymium, and zirconium (14–18%). The WE54 alloy contains other rare-earth elements (Cd, Er, Dy). In addition, alloys WE43 and WE54 exhibit a rectangular phase containing 66.9–74.8% Y.

The high-temperature properties of alloys ML10, ML12, and ML5 are presented in Fig. 5 and those of alloys ML10, ML19, and WE54 are presented in Table 9.

A comparative analysis has shown that alloys of the Mg–REM–Zr system (ML10, ML19) possess a higher yield strength at room and elevated temperatures than alloys of the Mg–Al–Zn–Mn system (ML5), an elevated creep resistance in long-term tests, and a lower sensitivity of the mechanical properties to the variation of the cross-section and to the presence of microporosity.

## CONCLUSIONS

1. Heat-resistant castable magnesium alloys ML10 and ML19 containing rare-earth metals and zirconium have a

TABLE 9

Alloy	$t_{\text{test}}, ^\circ\text{C}$	$\sigma_r, \text{N/mm}^2$	$\sigma_{0.2}, \text{N/mm}^2$	$\delta, \%$	$\sigma_{100}, \text{N/mm}^2$	$t_{\text{oper}}, ^\circ\text{C}$
ML10	20	230–260	140–160	6–9	–	250
	250	150	–	–	70	
	300	125–145	–	–	25	
ML19	20	220–250	120–150	6–8	–	300
	250	200–220	–	–	115	
	300	150–160	–	–	60	
WE54	20	255	185	–	–	300
	250	225	–	–	–	
	300	160	–	–	–	

\* Maximum temperature of long-term operation.

high level of mechanical and technological properties and corrosion resistance, which makes it possible to recommend them for the production of castings with a complex configuration and large mass and size for aircraft structures.

2. Alloy ML10 possesses the higher technological properties.

3. The high-temperature alloy ML10 is suitable for long-term operation at 250°C and alloy ML19 can operate at 300°C.

4. In order to obtain high-quality castings from alloys ML10 and ML19 they should be melted without a flux in a protective gas medium containing up to 1% SF<sub>6</sub>, especially for alloys containing Nd and Y, because these elements prevent oxidation and ignition of the melt. This decreases the loss of expensive rare-earth elements, eliminates flux corrosion, and improves the purity and the density of the parts.

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