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The Role of Molybdenum in the Formation of the Microstructure and Properties of Al-Cu Alloys

S. Stąpór **, M. Górny * 💩, Ł. Gondek * 💩, B. Gracz * 💩, M. Kawalec * 💩

 ^a Faculty of Foundry Engineering, AGH University of Krakow, Reymonta St. 23, 30-059 Krakow, Poland
^b Faculty of Physics and Applied Computer Science, Department of Solid State Physics, AGH University of Krakow, Mickiewicza Av. 30, 30-059 Krakow, Poland
* Corresponding author: E-mail address: sylwiapaz@agh.edu.pl

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Abstract

The subject of the presented research is the influence of molybdenum on selected properties of alloys that are based on the Al-Cu system. It could be observed that the introduction of molybdenum to the multi-component alloy at levels of up to 0.5 wt.% showed increases in the degrees of undercooling ΔT along with the increasing contents of the introduced element. The addition of molybdenum contributed to the reduction of the size of the primary grains of the $\alpha(Al)$ phase. Molybdenum improved the strength of the alloy while achieving elongation at a significant level. This is an element that occurs in the alloy – both in the iron-manganese phases and in the segregates inside the grains. Most of the iron-manganese phases occurred in a more spheroidal form. Additionally, tests were carried out on higher molybdenum content in the alloy. The addition of the tested chemical element at a level of 1 wt.% caused the precipitation of the phases that contained molybdenum, which did not dissolve after heat treatment.

Keywords: Metallography, Al-Cu alloys, Tensile properties, Thermal analysis

1. Introduction

Alloys based on Al-Cu with the addition of molybdenum are materials that are worthy of our attention. There is not much information in the literature on the effect of this element on Al-Cu alloys – especially when in combination with other alloying additives. The introductions of Mo, Zr, and Y have a positive effect on the high-temperature properties of the Al-5.8Cu-0.3Mn-0.2Mg alloy [1]. Due to the scarcity of information on the influence of molybdenum on Al-Cu alloys, its influence on the Al-Si system was considered by analogy. The available information describes the effect of molybdenum on the Al-Si-Cu-Mg (A354) alloy at both

ambient and elevated temperatures [2] as well as on the AlSi10Mg(Cu) alloy [3]. As a result of testing the A354 alloy, the formation of block-shaped particles could be observed, which contained Al, Si, Fe, and Mo phases as well as Al, Mo, and Si in the form of a star with the addition of 0.3 wt.% of Mo in the alloy. As a result of the formation of these phases and in combination with Fe, molybdenum prevented the formation of iron phases in the form of β -Fe needles. In an alloy that contains 0.3 wt.% Mo, there are phases that contain Mo (at the nanoscale level). Its addition also translated into a 10% increase in the yield strength as compared to the base alloy at an ambient temperature and after heat treatment (T6) [2].



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The influence of molybdenum on the high-temperature properties of alloys is increasingly being studied and described in Al-Si alloys. Automotive parts must withstand high loads despite having thin-walled structures. The material should perform its function at high temperatures. The combined action of Mo and Zr in the modified Al-Si-Cu-Mg alloy produces intermetallic phases that are characterised by good thermal stability [3]. The Al₃Zr phase contributes to the grain refinement playing a nucleating role. The phases that are formed as a result of introducing Mo and Zr into the alloy hinder dislocation movement and prevent crack propagation. The result is an improvement in the strength properties of the alloy. Molybdenum in the Al-Si-Cu-Mg alloy improves its thermal stability by inhibiting the precipitation of intermetallic phases during the ageing process [3]. The introduction 0.2 wt.% Mo to the alloy increases the liquidus temperature by almost 2°C and lowers the temperature at which molybdenum-rich intermetallic compounds precipitate by about 3°C. However, the microhardness is slightly reduced as a result of introducing up to 0.2 wt.% Mo to the alloy [4].

In recent years, interest in molybdenum and its influence on the properties of alloys has increased. In the Al-4%Cu alloy [5], the influence of molybdenum and nickel on the mechanical properties was examined. An increase in the hardness and plasticity of the alloy was achieved, but the alloy's plasticity was lost. To avoid brittleness in the alloy, molybdenum should be used in amounts of less than 1 wt.%. Molybdenum has a positive effect on the mechanical properties of the Al-4%Cu alloy, making it a material for use in the aviation and automotive industries. Among others, one important issue is the influence of molybdenum on the hightemperature properties of the Al-5.8Cu-0.3Mn-0.2Mg alloy [6]. In the alloy that contains the additions of Mo, Zr, and Y, precipitates that contain elements that are stable at high temperatures appear. The growth of the θ' phase is also slowed down at high temperatures. The introduction of the appropriate additives to the tested alloy (including Ag [6]) can improve high-temperature properties by creating stable high-temperature components. In this way, however, this mainly affects the grain refinement and, consequently, the θ' phase also precipitates [6]. The phases in the alloy that contain Mo, Zr, and Y (i.e. Al₃(Zr,Y), Al₃Zr, Al₃Y, and Al12Mo) are stable and may contribute to the slowing down of the growth of the θ' precipitates at high temperatures. An increase in the tensile strength of this alloy after introducing Mo, Zr, and Y at ambient and elevated temperatures was demonstrated [4]. The basis for interest in the influence of molybdenum is information on the improvement of the properties of the Al-Mn [7] and Al-Si [8] [9] alloys at high temperatures. This issue is explained by the low diffusivity of molybdenum in aluminium, which hinders the fragmentation of precipitates that contain molybdenum at high temperatures.

The addition of small amounts of transition elements (which also include molybdenum) that are characterized by low diffusivity and limited solubility in the solid state cause an increase in the strength in aluminium alloys and lead to a thermally stable material [10]. The alloy addition and the appropriate temperature of the solid solution influence the mechanical properties of the alloys. The tests on the Al-Cu alloy with the addition of 0.2 wt.% Cd showed that cadmium-rich nanoparticles were formed at a temperature of 530°C. Hybrid $\theta''+\theta'$ precipitates were also formed, which made the microstructure heterogeneous. Thanks to this, there was a quick

precipitation-hardening reaction and greater synergy of the strength and ductility in the discussed Al-Cu-Cd alloy [11]. A similar phenomenon may have applied to the Mo interaction in the Al-Cu alloy.

Molybdenum does not significantly affect the sizes of the primary grains in aluminium alloys [4] [7]. On the other hand, the addition of the combined elements Mo, Zr, and Y comminutes the primary grains in the Al-Cu-Mn alloy. However, the effect of the grain refinement alone does not determine the strengthening of the alloy at high temperatures. Refined grains strengthen the alloy due to smaller grains, and larger grain boundaries can serve as obstacles to dislocation motion. At high temperatures, the strengthening effect is not so significant due to the weakening of the role of the grain boundaries as obstacles in the dislocation motion [4].

An important parameter for each material is its thermal stability, which is reflected in its application. For aluminium alloys, this is a particularly important issue because, according to the literature, they are thermally unstable. The temperature and the time of use of the material are important. Attention should be paid to parts that are used in aircraft that may become hot due to the heat of the engines or exposure to the Sun [12] [13]. The subject of increasing the applicability of high-temperature materials is a current topic that is being undertaken by researchers [14]. For this purpose, research is being carried out to develop an aluminium alloy with various elements (mainly transition metals and rare earths), which will be characterised by a thermally stable microstructure and meet modern requirements. Using the great advantage of aluminium alloys (i.e. their lightness), it may turn out that, after their improvement, they can work at high temperatures and will become competitive with more-expensive alloys that are based on titanium and nickel [12].

The main topic of the research included determining the influence of molybdenum on the microstructure and properties of an alloy with a complex chemical composition that was based on the Al-Cu alloy. The molybdenum that was selected for the testing was selected due to the attempts that were mentioned in the literature to link it with improving the thermal stability of the alloy. Additionally, molybdenum is an element that is seldomly known in Al-Cu alloys. Due to the premise of proving the possibility of improving the properties of the alloy by introducing molybdenum, the issue of its impact on alloys from the Al-Cu system was verified in this work.

2. Materials and Methods

The presented research is based on a series of melts of an Al-Cu alloy with a variable molybdenum content of 0.0–0.5 wt.%. The first melting was carried out in an electrical induction furnace. The furnace charge consisted of an alloy that was characterised by a the chemical composition this is listed in Table 1 as well as an AlMo10 master. The weighed material was placed in a crucible, and the melting process was started. After the metal melted and reached a temperature of 750°C, it was held for 10 minutes; then, the thermal analysis cup and green sand mould for casting with a 15 mm wall thickness were casted. The crucible was then placed back into the furnace, and the AlMo10 master alloy was added to it. This was again held for about 10 minutes. The procedure was repeated for each level of molybdenum content in the alloy. The AlMo10 master alloy was added to the base alloy in order to obtain molybdenum at a levels of about 0.3 and 0.5 wt.%. During the melting, the crystallisation and cooling curves were recorded using thermal analysis cups with a K-type thermocouple and an Agilent 34970A digital recorder. Then, the samples were prepared for micro- and macrostructure analyses. The preparation of the samples for the macrostructure testing consisted of the electrolytic etching of the prepared metallographic specimens. The electrolytic etching was performed based on the ASTM E407-07 standard. The macrostructure was studied using a Leica MZ6 stereomicroscope and Leica OWin Standard software, which was used to determine the average grain diameter on a section of the specimen.

Table 1.

Chemical composition of base alloy

Chemical composition, wt.%								
Cu	Mn	Mg	Ti	Fe	Si	Zn		
4.45	0.46	0.39	0.19	0.05	0.04	0.04		

In order to test the tensile properties, the melts were repeated in the vacuum furnace; the samples for the strength tests were then cut from the obtained casting according to Fig. 1.

Table 2.

List of conducted research steps



Fig. 1. Dimensions of samples for tensile tests

During the melting of the non-grain-refined samples, the charge was melted to a temperature of 750°C, held for 10 minutes at this temperature, and followed by the mould pouring. The subsequent grain-refined samples differed only in their additions of the AlTi5B1 grain refiner after melting the charge. The melt was then held for 10 minutes at 750°C, and the mould was poured. The purpose of the melts in the vacuum furnace was to avoid casting defects (e.g. porosity). The samples for the tensile tests were prepared in a grain-refined version with the AlTi5B1 master alloy (0.2% as related to the weight of the charge) and in a non-grainrefined version. The heat treatment was carried out according to the following scheme: 525°C for 12 hours, quenching, initial ageing at an ambient temperature for 8 hours, and ageing at 200°C for 3 hours. Table 2 presents a list of the conducted research steps.

	non-grain-refined							
		Mo content, wt.%						
	0	0	.3	0.5				
thermal analysis	+		+	+				
macrostructure, average diameter of grains	+		+	+				
$R_m R_{p_{0,2}}$ and A after heat treatment	+		+	+				
thermal stability, after heat treatment	+		+					
	grain-refined							
	Mo content, wt.%							
	0	0.3	0.5	1.0				
$R_m R_{p_{0,2}}$ and A, after heat treatment	+	+	+					
chemical element distribution map, without heat			1					
treatment			Т					
analysis of chemical composition with scanning				+				
electron microscope, without heat treatment				I				

In addition, the melting was carried out in a vacuum furnace and grain-refined with the AlTi5B1 master alloy in which the molybdenum content was at a level of 1 wt.%. The purpose of using an excessive amount of molybdenum (1 wt.%) was to reveal the distribution of the molybdenum in the test material in order to identify it more easily in the samples with lower contents. For this reason, only a microstructural analysis was performed without considering the other tests for the sample with the content of 1 wt.% Mo. The occurrence of phases that contained molybdenum with its characteristic shape was observed. These phases did not occur in the samples with lower molybdenum contents.

The strength samples were broken on the AGX-V 100kN machine with an extensometer. The maps of the element distribution and chemical composition were made on a Tescan MIRA4 GMU

scanning electron microscope. The thermal stability tests were carried out in an argon-protective atmosphere. A measurement step of 5 degrees was used in the X-ray studies. The measurement was carried out from an ambient temperature to 350°C. DSC thermal analysis measurements were performed in the DSC Q20 device in copper crucibles in a protective atmosphere of argon (40 ml/min). The heating and cooling were carried out at 10°C/min within a range of 400°-700°C.

X-ray diffraction studies were made using a Malvern Panalytical Empyrean powder diffractometer that is operated with Bragg-Brentano geometry and equipped with a Cu Ka radiation source. The refinements were made via the Rietveld method, with the profile parameters being derived from the measurement of the NIST660 (LaB₆) standard sample. For the high-temperature

studies, an AntonPaar HTK 1200N furnace was used. During the measurements, the sample position was corrected thanks to the motorised sample stage.

3. Results and Discussion

3.1. Thermal analysis

Based on the cooling curves, the maximum degree of undercooling ΔT was determined from Equation (1). The formula captured the influence of the alloy additions on the equilibrium temperature of the crystallisation of the dendrites of the α (Al) phase [15]:

(1) $\Delta T = T_L - T_{min}$,

Table 3.

Calculation results with exact chemical compositions of tested allows

where:

$$T_L = T_F + \overline{m}_L \ \bar{C}_L \tag{2}$$

$$\bar{m}_L = \sum_{i=1}^n (m_L^i C_L^i) / \bar{C}_L \tag{3}$$

 \overline{m}_L – average slope factor of liquidus line;

- \bar{C}_L sum of concentrations of all chemical elements in liquid metal:
- T_F crystallisation temperature of pure aluminium $(T_F = 660.45^{\circ}C);$
- T_L equilibrium crystallisation temperature for α (Al) dendrites; T_{min} – minimum temperature at beginning of alloy crystallisation;

 m_L^i – slope factor of liquidus line of i-th component;

 C_{I}^{i} – concentration of i-th component.

Table 3 presents the results of the calculations that were used to determine the maximum degree of undercooling ΔT .

calculation results with exact chemical compositions of tested anoys										
wt.% Mn	wt.% Cu	wt.% Ti	wt.%Mg	wt.% Fe	wt.% Mo	m_L	C_L	T _{min} , °C	T∟, °C	ΔT, °C
0.44	4.39	0.16	0.57	0.079	0.00	-0.71	5.56	649.90	656.51	6.61
0.43	4.42	0.15	0.57	0.079	0.28	0.34	5.85	650.52	662.46	11.94
0.42	4.32	0.13	0.61	0.085	0.50	1.14	5.98	649.50	667.30	17.79

Fig. 2 shows the cooling curves for the alloys with different molybdenum contents in the tested alloys. In the case of the minimum temperature at the beginning of alloy crystallisation Tmin, an increase was observed for the content of 0.3 wt.% Mo, followed by a decrease after the next dose of the AlMo10 master. In this case, the influence of the different molybdenum contents on the crystallisation and cooling curves was analysed.



Fig. 2. Cooling curves of tested alloys with variable molybdenum content

Fig. 3 shows that the maximum degrees of the undercooling ΔT increases with the molybdenum contents.



Fig. 3. Undercooling degree depending on molybdenum content

3.2. Macrostructure

The macrostructures for the samples before heat treatment from the thermal analysis cups are shown in Fig. 4.



Fig. 4. Macrostructures of investigated alloys in non-grain-refined state

Based on the obtained macrostructures (Fig. 4), the average diameter of the primary grains \overline{d} was determined using the Leica QWin Standard software and compared with the various molybdenum contents. It could be observed that the primary grains of the α (Al) phase were fragmented to a slight extent (Fig. 5).



Fig. 5. Average grain diameter of $\alpha(Al)$ primary grains as function of molybdenum content

3.3. SEM analysis

In order to try to determine the influence of the molybdenum on the iron-manganese phases, an analysis of the distribution of the elements in a sample that contained 0.5 wt.% Mo (without heat treatment, grain-refined alloy) was carried out. Based on a map of the elements, it could be observed that the molybdenum occurred in the iron-manganese phases and segregated inside the grains. Fig. 6 shows the distributions of the elements in the analysed area.



Fig. 6. Maps of element distribution for sample that contained 0.5 wt.% Mo without heat treatment, modified alloy

Due to the attempt to demonstrate the presence of molybdenum-containing phases, it was decided to carry out the melting process in a vacuum furnace based on the alloy with the chemical composition that is given in Table 1 and containing excess Mo (at a level of 1 wt.%). Using a scanning electron microscope, the presence of molybdenum-containing phases was demonstrated. These were characterised by shapes that resembled "butterflies". The obtained picture is shown in Fig. 7a, along with an analysis of the chemical composition at individual points and in the area that covered the matrix. Figure 7b shows the sample in the polished state after heat treatment with visible phases that have not dissolved.



Fig. 7. Scanning electron microscope image without heat treatment a) and polished surface after heat treatment b) of a sample that contained 1 wt.% Mo, modified alloy

Table 4.				
Results of chemical-com	position anal	ysis at Points	1–4 for Fig.	7a

Point 1			Point 2			Point 3			Area 4			
	Atomic,	Conc.,	Error,	Atomic,	Conc,	Error,	Atomic,	Conc,	Error,	Atomic,	Conc,	Error,
	%	wt.%	σ	%	wt.%	σ	%	wt.%	σ	%	wt.%	σ
Mg	1.74	1.38	0.11	0.57	0.37	0.08	0.34	0.24	0.04	0.50	0.48	0.05
Al	86.96	76.61	0.85	69.80	49.90	0.86	85.31	64.17	0.70	98.52	97.25	0.76
Ti	0.02	0.02	0.02	0.17	0.22	0.06	0.63	0.84	0.11	0.04	0.07	0.03
Mn	2.56	4.59	0.31	0.13	0.18	0.06	1.14	1.74	0.19	0.15	0.30	0.08
Fe	3.32	6.05	0.38	0.24	0.36	0.09	0.07	0.11	0.05	0.11	0.23	0.07
Cu	5.29	10.97	0.69	29.09	48.97	1.46	0.61	1.08	0.20	0.57	1.32	0.22
Mo	0.12	0.38	0.11				11.90	31.82	0.89	0.11	0.39	0.11
whe	where: conc. – concentration (error refers to wt.%)											

Table 4 presents the results of the analysis of the chemical composition of both phases that contained molybdenum as well as the matrix and eutectics.

At Point 1, there was an Al-Cu-Fe-Mn phase that also contained small amounts of magnesium. At Point 2, the Al₂Cu eutectic was identified. Based on an analysis of the chemical composition of Point 3, the presence of molybdenum in the AlMo phases that also contained small amounts of manganese, titanium, and copper was clearly demonstrated. The visible phase had a characteristic shape. It could also be observed that these phases did not dissolve after applying the heat treatment. Area 4 concerns the analysis of the chemical composition of the matrix of the tested alloy.

3.4. Tensile tests

The samples that were used for the tensile tests were prepared in non-grain-refined and grain-refined versions using the AlTi5B1 master alloy. These were also heat treated. Fig. 8 shows the graphs of R_m (the tensile strength), $R_{p0.2}$ (the yield strength), and A as a function of the molybdenum content; it could be observed that its addition improved the strength of the alloy. An elongation value was obtained that was within a range of approximately 12 to 14% (for those alloys with molybdenum).



Fig. 8. Diagram of a) R_m, b) R_{p0.2} and c) A depending on molybdenum content

3.5. Thermal stability

Thermal stability is an important issue that provides information about the conditions in which castings can be used. The changes in the parameters that were obtained on the bases of the XRD tests were analysed. Information was obtained on the phase share, lattice parameters, lattice deformation, and stress in the microstructure. Figure 9 shows the XRD patterns as a function of the temperature for two samples after heat treatment for different molybdenum contents (0.0 and 0.3 wt.%).



Fig. 9. XRD patterns for two samples with different molybdenum contents: a) 0.0 wt.%; b) 0.3 wt.%

The Al-type FCC cubic $(Fm\overline{3}m)$ structure was the only phase identified at room temperature. When rising the temperature above roughly 225°C the additional reflections that originated from the AlFe₂Mn-type cubic ($Fm\overline{3}m$) structure could be observed. Above 325°C, additional small reflections could be noticed; these could have been attributed to the Al2Cu-type tetragonal I4/mcm structure. These additional phases (which suggested the disproportionation of the alloy) seemed to appear at higher temperatures for the Mo-enriched sample. Slight discrepancies in the intensity of the reflections for Al were observed at a value of about 45 degrees. Fig. 10 shows the XRD patterns for both samples in the enlargement of the area from Fig. 9 for the range of 42-45 degrees. In both cases, there was a shift of 44.25-45.00 degrees in the intensity of the reflections to lower values of 2θ at the higher temperatures. For Alloy B, the intensity of the reflections in the area of 42.0-44.5 degrees was higher than it was for Alloy A. Focusing more precisely on the range of 44.25-45.00 degrees, it

could be observed that, for Sample B (Fig. 10b), the highest intensity of the reflections occurred at temperatures of 210° -335° C. For Sample A (Fig. 10a), on the other hand, the area with the highest intensity was clearer and extended over almost the entire measuring range.



Fig. 10. XRD patterns for samples with different molybdenum contents, enlarged area: a) 0.0 wt.%; b) 0.3 wt.%

The Rietveld method was used to test the thermal stability of the alloys; this allowed for determining the changes in the parameters of the crystal lattice. Fig. 11a presents the data on the changes in the parameters of the crystal lattice depending on the temperature. An increase in the a-lattice parameter as a function of temperature could be observed. During the heating (at about 200°C), a transformation took place. After cooling the samples, the lattice parameter did not return to its original value. From this, it was concluded that the transformation at about 200°C was irreversible, thus leading to the disproportionation of the parent alloys. Once again, the aforementioned transition occurred at higher temperatures for the Mo-enriched alloy. In the Al-Cu alloys, the Guinier Preston (GP) zones were transformed into the θ " transition phase in the ageing process at above approximately 100°C (a tetragonal lattice that was coherent with the aluminium's crystal lattice). In the next stage, a transformation occurred at a temperature of 200°C in which a metastable transition phase θ' was formed with a regular structure that was partially coherent with the matrix. The θ' phase significantly strengthened the alloy structure. At higher temperatures, the transformation of θ' into θ (Al₂Cu) occurred at the interfaces. The θ' phase was stable and had a tetragonal structure that was incoherent with the matrix crystal lattice. As a result, local stress fields were created, which blocked the movement of the dislocations: as a result, the allov became stronger. The intermediate precipitates of the Al2Cu phase that were formed within a temperature range of 150°-200°C during the artificial ageing contributed to the high strength of the alloys from the Al-Cu system.



Fig. 11. Lattice parameter a) and lattice strain b) for samples with different molybdenum contents depending on temperature

The occurring transformation caused an increase in the strain of the crystal lattice (Fig. 11b). The step increase in the lattice strain for the alloy without the addition of molybdenum was greater (from 0.49% (135°C) to a max. of 0.54%). However, the addition of molybdenum reduced the strain; the increase in its value was from 0.45% (135°C) to a max. of 0.49%. After cooling the lattice, the strain were much lower than the initial values in both cases; however, the strain value after the cooling of the sample without the molybdenum was higher (0.28%) than that of the sample with 0.3 wt.% Mo (0.24%). From the presented results, it follows that the addition of molybdenum reduced the lattice strain; this was a beneficial phenomenon from the point of view of the thermal stability. Even at an ambient temperature, it could be seen that the alloy with the molybdenum had a noticeably lower lattice strain after the cooling. Above a temperature of 250°C, a proportional increase in the lattice parameter could be observed, with a simultaneous decrease in the strain.

4. Conclusions

Summarising the presented research, the following could be observed:

- Calculated on the basis of the chemical composition and thermal analysis, the maximum degree of the undercooling increased after increasing the molybdenum content.
- Molybdenum was the element that contributed to the smaller sizes of the primary grains of the α(Al) phase.
- The tensile properties improved after increasing the molybdenum content. The alloy that contained 0.3 wt.% Mo obtained a tensile strength of nearly 450 MPa after the heat treatment. In addition, a significant elongation was obtained.
- On the basis of the examinations that used the scanning electron microscope, it was found that the molybdenum was the element that may have contributed to the changing of the shape of the phases that contained manganese and iron. The iron-manganese phases that were present in the sample were partially coagulated. Molybdenum could be found in the iron-manganese phases and segregates inside the grains. Those phases with the more favourable shapes contributed to the improvement of the strength properties.
- The thermal stability studies showed that, during the heating, a transformation occurred at about 200°C; this was irreversible because, after the cooling of the sample, the lattice parameter did not return to its original value. Discrepancies could be observed for the sample without the addition of molybdenum and that contained 0.3 wt.% Mo in the lattice parameter. Within a temperature range of 170°-230°C, the samples differed from each other; lower latticeparameter values were obtained for the sample with molybdenum. At a temperature of 200°C, a transformation occurred in which a metastable transition phase θ' was formed (a regular structure that was partially coherent with the matrix). At higher temperatures, the transformation of θ' into θ (Al₂Cu) occurred. The intermediate precipitates of the Al₂Cu phase, which were formed within a temperature range of 150°-200°C during the artificial ageing, contributed to the high strength of the alloys from the Al-Cu system. Molybdenum had a positive effect on improving the thermal stability due to the participation of molvbdenum in the formation of the phases as well as on reducing the lattice strain.
- An excess of 1 wt.% Mo caused the formation of phases in the alloy that did not dissolve after the heat treatment.

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