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The Influence of Selected Refining Methods of AlSi7Mg0.3 Silumin on its Quality Index

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Abstract

The publication presents the comparison of selected refining methods (gaseous and/or flux) based on mechanical properties of the obtained secondary silumin EN AC-AlSi7Mg0.3 (in accordance to the European Standard PN-EN 1706:2011). The point of reference was a similar primary alloy produced using pure batch materials. The mechanical properties measured in room temperature were used to calculate the materials quality index.

The research showed, that properly carried out refinement process of secondary (recycled) alloys can bring their quality indexes close to those of their primary materials.

The goal was to assess the efficiency of selected refining methods when applied to the examined group of casting silumins, by measuring the basic mechanical properties (in room temperature) before and after refining. The practical aspect was to choose an effective (ecologically, technologically and economically) method of refining of secondary EN AC-AlSi7Mg0.3 alloy used to cast car rims for JN METAL company in Ostrowiec Świętokrzyski (Poland).

Keywords: Cast aluminum alloys, Refining, Quality Index

1. Introduction

From the middle of 1950s the world can see a constant growth in the production of aluminum, both primary and recycled. The recycled aluminum alloys currently represent over 30% of the global production while the remaining 70% are primary ones. In high developed countries the proportion of recycled alloys is much bigger. In USA for instance, in the first decade of 21st century it exceeded the level of 60% of total aluminum used.

The whole production of aluminum castings in 2011 reached 256 112 tons [1]. Data [2] shows that 150 000 tons of secondary aluminum cast alloys were used in Poland (mainly in the production of automotive parts) in 2011. It's easy to calculate that

the proportion of recycled alloys used amounted to 58% as of 2011. According to most recent data from USA the proportion of secondary alloys used in casting reaches even 90% [2, 4-5]. Therefore a further increase in usage of recycled materials in aluminum castings should be expected in the near future.

The fall in the overall production of aluminum alloys in the years 2007-2009 can be attributed to global financial crisis. The small decrease in costs of producing aluminum alloy casts in 2014-2015 was caused by perturbations in Chinese economy.

The growth in demand for aluminum alloys is generated by the unique material properties of this metal. Aluminum alloys are in demand because they have good technological properties and are easy to recycle. The growth in demand is possible to meet because

of large natural reserves since aluminum is the third most common element in earth's crust and the most common metal.

The main advantage of recycling is that to restore aluminum waste to the state when it can be used again, it takes only 5% of the energy required to produce primary aluminum. It enables a reduction of annual world's CO₂ emissions by 90 million tons and it saves around 100 000 GWh of electric energy. The growth in demand for recycled aluminum is mainly caused by the same reasons that are behind growth in demand for primary one. That means mainly the development of transport sector and changes in ecological policies [3-5].

The relative simplicity and profitability of aluminum recycling cause that its recycling rates can reach 90-95% in transport and construction industries, and 65% for drink cans [3-4].

Primary aluminum is not produced in Poland. That is why the production of secondary aluminum alloys is currently flourishing.

The source of pollutants in liquid casting alloys (both primary and secondary) is the condition of input materials and faults in casting process.

When it comes to aluminum alloys the aim of refining process is to eliminate or reduce [6-13]:

- gaseous impurities (mainly H₂);
- solid non-metallic inclusions (mainly oxides - Al₂O₃, MgO and others);
- presence of undesirable elements (like Mg, Na, K, Ca etc.)

To remove gaseous and/or solid impurities and to reduce content of some of undesired elements various refining operations are carried out. The following refining methods are used in industry and science [8, 14]:

- ❖ depending on the type of technological process:
 - chemical;
 - physical;
- ❖ depending on the tools and means used:
 - flux refining or salt treating (ZnCl, MnCl, AlCl and others);
 - gaseous (Ar, N₂, Cl and others);
 - filtration;
 - special (void, vibration, ultrasounds and others);
 - mixed (combinations of some of the above).

The most common practical application during melting at foundries and processing plants have gaseous and flux refining of liquid alloys. Fluxes used until now contain now unacceptable or undesirable chlorine and fluorine compounds. It is the same with using chlorine and fluorine in the form of gas. That is why gaseous refining aluminum alloys of hydrogen and other non-metallic inclusions is nowadays performed using inert gases (nitrogen, argon, helium) [6, 10]. Special permits are issued to foundries or scrap aluminum processing plants allowing usage of gas mixtures containing up to 3 or 5% of chlorine together with nitrogen or argon. The mixtures of solid, refining fluxes for aluminum alloys approved in Poland are mainly nitrogen based [15-16]. However they often contain small doses of fluorine or chlorine compounds (i.e. fluorite CaF₂, MnCl, MgCl, KCl and others).

Most commonly used refining effects assessment criteria are:

- the change of hydrogen content [cm³/100g] in liquid aluminum alloy (before and after refining);
- the change in content of non-metallic inclusions:
 - methods: PoDFA, Prefil, LiMCA, K-Mold;

– DROSS test (AluSpeed Tester FMA, pressure p = 0 or 5 [mbar]);

– a metallurgical approach (quantitative and qualitative metallography of the alloy).

- the change of microstructure content in solid state (after crystallisation) based on:
 - density - ρ [g/cm³], e.g. using hydrostatic weighing;
 - gas number - $LG = \rho_{exp.} / \rho_{theor.}$, where $\rho_{exp.}$ means experimental density and $\rho_{theor.}$ is the theoretical density (determined i.e. based on alloys chemical composition);
 - porosity - $P = (\rho_{theor.} - \rho_{exp.}) / \rho_{theor.} \cdot 100$ [%];
 - density index (ger. *Dichte Index*) - $DI = [(\rho_{atm} - \rho_{80\ mbar}) / \rho_{atm}] \cdot 100$ [%], where ρ_{atm} - density of samples solidifying under atmospheric pressure, $\rho_{80\ mbar}$ - density of samples solidifying under pressure of 80 [mbar];
 - Straube-Pfeifer quality test (i.e. AluSpeed tester FMA, p = 40 [mbar]).
- the change in mechanical properties (UTS, YS, A₅, HBN, E and others) determined at room temperature in tensile testing using separate casting samples.

The quality index QI for aluminum alloys was introduced in 1980 by M. Drouzy, S. Jacob and M. Richard [17-18]. In Poland first mentions on the subject can be found in the works of A.W. Orłowicz and M. Mróz from 2004 [19]. The mathematical expression of QI combines strength and plastic properties of the material. Specifically for casting silumin AlSi7Mg0.3 it takes the form below:

$$QI = f(UTS, A_5) = UTS + d \cdot \log(A_5), \quad (1)$$

where UTS – ultimate tensile strength, A₅ – elongation at samples rupture point, d – experimentally selected proportionality coefficient (for casting silumin AlSi7Mg0.3 d = 150 [17, 19]).

QI was selected as an indicator to assess the refining efficiency because of its wide usage when assessing influence of chemical composition, modifications of stages and parameters of heat treatment on the quality of aluminum alloy castings (silumins in particular).

The analysis of specialist literature and the authors' experience shows that the physico-mechanical properties (e.g. tensile strength and plasticity) of AlSi7Mg0.3 alloys varies significantly. It is the effect of: the level of content of basic alloying additives (Si and Mg); the quantity of iron and/or zinc impurities; fragmentation of α_{Al} - solid solution or silicon eutectic modification $\alpha_{Al} + \beta_{Si}$ (e.g. Sr or Na); the speed of crystallization during casting; the state of the alloy or the method and parameters of heat treatment [20-24]. It is assumed that these properties will also depend to a great extent on the quantity of solid and/or gas inclusions. The assessment of the influence of gas and / or flux refining on the basic mechanical properties and, above all quality index of AlSi7Mg0.3 alloy at room temperature is the main subject of this publication.

2. The research methodology

The subject of the study was a standardized aluminum casting alloy EN AC-42100 (EN AC- AlSi7Mg0.3), in accordance with PN EN 1706: 2011 [26] - analogue to American 356 and A356.0 (AA or ASTM) [20, 25] (Table 1), used in low-pressure casting of car rims.

Table 1.

Percentages of content of basic alloy additives, contaminants and modifiers of AlSi7Mg0.3 alloy, according to European and American standards [20, 23, 26-27]

Designation		Source	Element content [wt. %]								
No.	Symbol		Si	Cu	Mg	Mn	Fe	Zn	Ti	Al	
356	AlSi7Mg0.3	ASTM	6.5	\leq	0.20 ⁺	\leq	\leq	\leq	\leq	\leq	rest
		Specification B26/B26M-09	7.5	0.25	0.45	0.35*	0.60*	0.35	0.25		
A356.0	AlSi7Mg0.3	ASTM	6.5	\leq	0.25 ⁺	\leq	\leq	\leq	\leq	\leq	rest
		B 108-96a (cast-metal mould)	7.5	0.20	0.45	0.10	0.20	0.10	0.20		
EN AC-42100	EN AC- AlSi7Mg0.3	PN-EN-1706: 2011	6.5	\leq	0.25 ⁺	\leq	\leq	\leq	\leq	\leq	rest
			7.5	0.05	0.45	0.10	0.19	0.07	0.25		

* If the iron content is greater than 0.45%, then manganese should not be less than half of the iron value

Two experimental (model) AlSi7Mg0.3 alloys were used in the study: a) recycled (secondary alloy - Table 2) from JN METAL from Ostrowiec Świętokrzyski (Poland); b) primary alloy (Table 3) made in the Institute of Non-Ferrous Metals, Light Metals Division (IMN OML) in Skawina from technically pure input materials.

Table 2.

Chemical composition of experimental secondary AlSi7Mg0.3 output alloy (JN METAL company)

Symbol	Content of alloy additives and aluminum [wt. %]								
	Si	Mg	Cu	Fe	Mn	Ni	Cr	Zn	Ti
JN METAL	7.46	0.17	0.00	0.24	0.01	0.00	0.00	0.01	0.15
EN AC-42100 (EN AC- AlSi7Mg0.3)	Sr	P	Na	Ga	V	Li	Ca	B	Al
	0.01	0.00	0.00	0.01	0.00	0.00	0.00	0.00	rest

Table 3.

Chemical composition of experimental primary AlSi7Mg0.3 output alloy (IMN OML Skawina)

Symbol	Content of alloy additives and aluminum [wt. %]								
	Si	Mg	Cu	Fe	Mn	Ni	Cr	Zn	Ti
IMN OML	7.37	0.25	0.00	0.11	0.00	0.00	-	0.01	0.10
EN AC-42100 (EN AC- AlSi7Mg0.3)	Sr	P	Na	Ga	V	Li	Ca	B	Al
	0.01	-	-	-	-	-	-	-	rest

The secondary (JN METAL) alloy originated from recycled elements of a gating system (ger. *angus*) containing iron filtering nets used for low-pressure casting of car wheel rims. The recycling of AlSi7Mg0.3 alloy was carried out at JN METAL in a medium frequency induction furnace with a crucible capacity of

approximately 3 tons of aluminum alloy. Experimental secondary alloy was not subjected to additional refining. It was only kept for several minutes at a temperature of 720-760°C. Then the slag (dross) was removed from the surface and alloy was poured into metal moulds.

The processes of smelting and refining were carried out in a graphite-chamotte crucible (with a capacity of ~14 kg of aluminum alloy) of an electric resistance furnace made by CZYLOK company (Poland). Upon loading the metal into the furnace it was first being pre-heated, then melted and overheated (about 110-130°C over the melting point). The temperature of liquid metal was measured using 2xNiCr K-2 thermocouple (accuracy of $\pm 5^\circ\text{C}$). The temperature was in range of 720-740°C when casting.

Two gas and two solid refiners have been selected for the purpose of the study. Their characteristics are presented in Table 4. The main criteria for selecting the refiners (fluxes) were ecological considerations. Mainly nitrogen based compounds with minimal quantity of elements such as chlorine or fluorine were used from among the solid refiners.

The selection of refiners was mainly determined by the plan of experimental casting shown in Table 4.

Table 4.

The plan of experimental smelting

No.	Smelt characteristics
1	Recycled alloy (from JN METAL) - casting of rods for strength tests - before refining and after refining with nitrogen (N₂) (7 minutes refining).
2	Recycled alloy (from JN METAL) - casting of rods for strength tests - before refining and after refining with argon (Ar) (7 minutes refining).
3	Recycled alloy (from JN METAL) - casting of rods for strength tests - before refining and after refining with a Desydral N71P solid (powder) refiner (0.35% of the input mass).
4	Recycled alloy (from JN METAL) - casting of rods for strength tests - before refining and after refining with a Affigral DN11P solid (powder) refiner (0.35% of the input mass).
5	Recycled alloy (from JN METAL) - casting of rods for strength tests - before refining and after refining with a Desydral N71P solid (powder) refiner (0.35% of the input mass) + refining with argon (Ar) (7 minutes refining).
6	Primary alloy (from IMN OML Skawina) - casting of rods for strength tests - before refining and after refining with argon (Ar) (7 minutes refining).

Gaseous refining was carried out using a lance (ceramic tube sealed at the bottom and with side openings, resistant to temperatures up to 1300°C). The stream of gas was introduced at a height of about 50 [mm] from the bottom of the crucible. The liquid metal's surface was calm and showed only slight vibration without interception (occlusion) of air. The time of refining was 7 minutes.

Shredded solid refiner (flux) wrapped in aluminum foil (with clearances) was dried for about 2 hours in a furnace-dryer at 100°C prior its introduction into the liquid metal. It was introduced on the bottom of the crucible with a steel casting spoon covered with a separator and then mixed with liquid metal. After about 7-10 minutes the dross was removed from the liquid metal's surface and casting was carried out.

Smelting was conducted in a controlled atmosphere (under cover). Temperature, relative humidity and atmospheric pressure were monitored using 4-WB213 weather station (made in Germany).

The rods for strength tests were cast in iron mould coated on the inside with a graphite-based colloidal coilite and heated to a temperature of 240-260°C. For each refining variant 5 samples were cast. The results of the measurements were thus statistically averaged.

The shape and dimensions of the strength samples were in accordance with PN-EN ISO 6892-1: 2010 standard. The samples were tested in the F state (Fabrication) - i.e., after casting.

Static tensile strength tests (according to PN-EN ISO 6892-1: 2010 [28]) were carried out at the Centre for Material Testing and Mechatronics of the Motor Transport Institute in Warsaw. An Instron 8802 (250 kN) testing machine was used. Measurements were made at 20°C. The humidity (LB700 hytherograph) ranged from 39% to 45%. The elongation speed was 0.40 mm/min.

HBN Brinell hardness testing (according to PN-EN ISO 6506-1: 2014 [29]) was carried out at the Foundry Institute in Cracow, using the Zwick/Roell ZHU 250 universal hardness tester. The measurements were made on cross-sections of the "heads" of the strength samples (3 measurements for each of the 5 specimens of each cast). Diameter of tungsten bead was 5.0 mm, the load was 2452 N and nominal load time - 12 sec.

Based on the tensile strength and hardness tests, mechanical properties were determined at room temperature, as shown in Table 5.

Table 5. Mechanical properties of secondary and primary AlSi7Mg0.3 alloys - before and after refining

Name	Ultimate Tensile	Elastic Limit	Yield Strength	Elongation
Symbol	UTS	EL	YS	A*
Unit	[MPa]	[MPa]	[MPa]	[%]
Name	Elongation	Necking	Young's Modulus	Brinell Hardness
Symbol	A _s **	N	E	HBN
Unit	[%]	[%]	[GPa]	[HB]

A* – relative elongation, in relation to displacement of machine jaws;
 A_s** relative elongation, in relation to displacement measured with a strain gauge (length 50 mm, sample diameter – Ø10 mm)

3. Measurements and discussion

A summary of the measured mechanical properties and the QI quality index (calculated using equation 1) for secondary and primary experimental AlSi7Mg0.3 alloys, before and after refining are shown in Table 6.

Table 6. Summary of mechanical properties of AlSi7Mg0.3, secondary and primary alloys, before and after refining

melt sample no.	Alloy description: its origin and state	Mechanical properties					
		UTS [MPa]	YS [MPa]	A _s [%]	N [%]	HBN [HB]	QI [MPa]
1	Recycled alloy (JN METAL). before gas refining with nitrogen (N ₂)	160.5	78.4	5.4	6.1	54.3	270.4
	Recycled alloy (JN METAL). after gas refining with nitrogen (N ₂)	172.4	76.9	7.1	7.6	56.7	299.7
2	Recycled alloy (JN METAL). before gas refining with argon (Ar)	158.9	78.2	4.3	4.5	56.7	253.9
	Recycled alloy (JN METAL). after gas refining with argon (Ar)	177.3	78.7	9.1	8.6	57.7	321.2
3	Recycled alloy (JN METAL). before flux refining with Desydral N71P	154.6	77.4	4.5	6.2	54.6	252.6
	Recycled alloy (JN METAL). after flux refining with Desydral N71P	165.5	76.9	7.2	7.2	55.5	294.1
4	Recycled alloy (JN METAL). before flux refining with Affigral DN11P	158.0	78.0	4.7	5.6	55.2	258.8
	Recycled alloy (JN METAL). after flux refining with Affigral DN11P	178.1	75.5	9.3	11.3	57.4	323.4
5	Recycled alloy (JN METAL). before refining with Desydral N71P + argon (Ar)	157.5	77.5	4.3	5.0	56.7	252.5
	Recycled alloy (JN METAL). after refining with Desydral N71P + argon (Ar)	172.9	79.5	8.6	8.8	56.5	313.1
6	Primary alloy (IMN OML). before gas refining with argon (Ar)	181.8	87.8	8.8	7.9	58.1	323.5
	Primary alloy (IMN OML). after gas refining with argon (Ar)	183.3	85.6	7.0	6.7	59.9	310.1

Linear dependencies of selected pairs of mechanical properties (for all obtained results- Table 6) are presented in Figures 1 and 2.

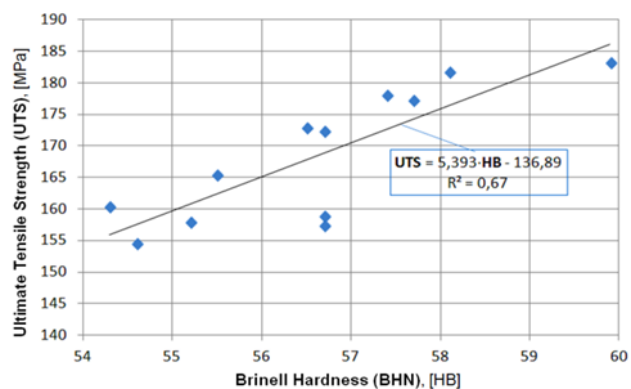


Fig. 1. Linearity between Brinells hardness (HBN) and ultimate tensile strength (UTS) for all experimental AlSi7Mg0.3 alloy variants (based on Table 6)

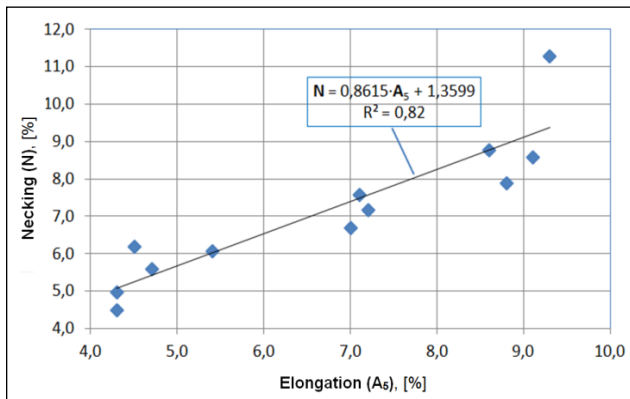


Fig. 2. Linearity between elongation (A5) and necking (N) for all experimental AlSi7Mg0.3 alloy variants (based on Table 6)

The dependencies shown in the above figures indicate that their correlation is typical for casting silumins. At the same time the values of elongation (A5) and necking (N) in F-state (after casting) are similar to those mentioned in Seifeddine's work [30].

High plasticity of AlSi7Mg0.3 alloy in these conditions is mainly the result of:

- low iron content ($\leq 0.25\%$),
- α_{Al} solid solution grain fragmentation with titanium
- silicon eutectic modification ($\alpha_{Al} + \beta_{Si}$) with strontium.

In the states after heat treatment (T4, T6 or T7), the relative elongation of a given group of alloys (AlSi7Mg0.3-0.6, or A356 - A357 – according to American standards AA or ASTM) may reach 20% or more [21].

An example of the evaluation of refining effects (Desydral N71P + Ar) for the recycled alloy (JN METAL) from smelt No. 5 based on mechanical properties and quality index is presented in Table 7.

Table 7.

Evaluation of the effects of refining of the secondary alloy (JN METAL) from the smelting No. 5 on the basis of changes in mechanical properties and QI

Melt no. 5 – recycled alloy JN METAL									
Alloy description	Sample no.	Mechanical properties							
		UTS [MPa]	EL [MPa]	YS [MPa]	A [%]	A ₅ [%]	N [%]	HBW [HB]	E [GPa]
before refining with a Desydral N71P + argon (Ar)	5.1	157,5	55,8	77,7	4,5	4,3	5,0	56,7	75,2
after refining with a Desydral N71P + argon (Ar)	5.2	172,9	61,8	79,5	9,1	8,6	8,8	56,5	72,3
Property change [%]		+9,8	+10,8	+2,6	+102,2	+100,0	+76,0	-0,4	-3,9
Quality index (QI), [MPa]		$QI(5.1) = UTS + 150 \cdot \log_{10}(A_5) = 157,5 + 150 \cdot \log_{10}(4,3) = 252,5$ $QI(5.2) = UTS + 150 \cdot \log_{10}(A_5) = 172,9 + 150 \cdot \log_{10}(8,6) = 313,1$							

The results of the geometric analysis of tensile strength (UTS) and plasticity (A5) of the tested alloys (secondary JN METAL and primary IMN OMN) are shown in Figure 3, in relation to the refining method used.

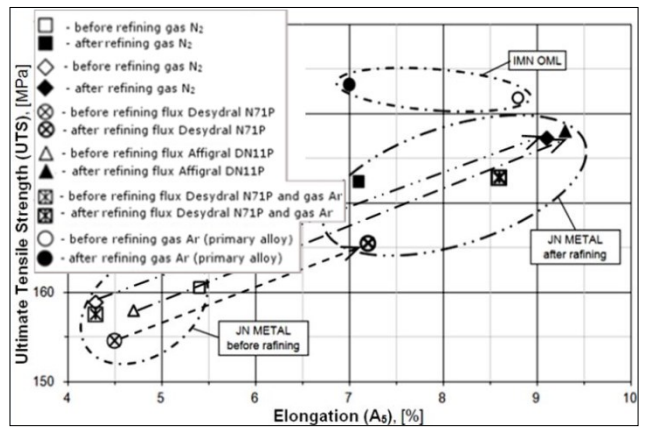


Fig. 3. Changes in mechanical properties (UTS and A5) of experimental silumin (primary IMN OML and secondary - JN METAL) in relation to the refining method

Figure 4 shows the same data with A5 in logarithmic scale. Constant values of QI (175 to 325, with a spacing of 25 units) were marked, calculated using equation (1).

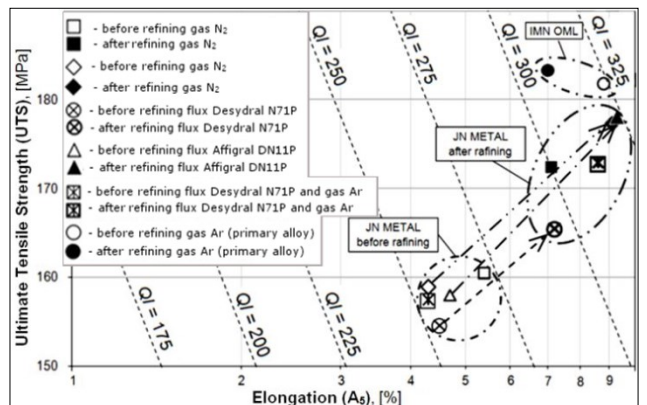


Fig. 4. Effect of refining method on QI quality index change of silumins: primary IMM OML and recycled - JN METAL

The data presented in Tables 6 and 7 and in Figures 3 and 4 show that the refining processes of secondary alloy reach the desired effect of improving its quality measured by QI. The effect of positive comprehensive influence (on strength and plasticity parameters) depends on the refining method used. At the same time it should be noted that the refining of technically pure (primary, metallurgical) alloys (casting silumins) can not always be technically and economically justified.

4. Summary

The conducted studies show that:

- The QI can be a simple, relatively inexpensive and effective way to comprehensively evaluate the quality of casting aluminum alloys, particularly the silumins.

- The gaseous, flux as well as the mixed (flux-gas) refining of the recycled casting silumin AlSi7Mg0.3 allow a significant improvement of its basic mechanical properties (UTS, A₅), and as a result improve its quality index.
- The final refining effect depends on the refiner and refining method. The experiments show a slightly better effect of refining with argon compared to nitrogen. Flux refiners should be carefully selected not only because of overall quality improvement (e.g. QI) but also for ecological and economic reasons.
- If the use of a solid medium (flux) gives unsatisfactory results, the final effect can be significantly improved by additional refining with inert gas (N₂ or Ar). Due to the similar prices of both of these gases recently, priority should be given to Ar.
- Environmentally friendly refiners nowadays contain more nitrogen (N₂) and have limited content of chlorine and fluorine compounds.
- Refining of primary, metallurgical alloys is not always desirable. The primary aluminum alloy in many cases may not require refining prior to casting, especially if it is melted rapidly (e.g. in induction furnaces).
- Proper refining can bring the quality of the recycled aluminum alloy very close to the parameters of the primary (metallurgical) alloy.

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