Change in the Microstructure of AlSi10MgMn Alloy with Higher Iron Content Due to an Increase in the Amount of Nickel

Mária ŽIHALOVÁ, Dana BOLIBRUCHOVÁ*, Marek MATEJKA

Abstract: Recycling of the materials often requires modification of the manufacturing processes or application of the techniques to eliminate impurities that enter the material during recycling process. The main and the most detrimental impurity of the secondary (recycled) aluminium alloy is iron. Low iron solubility in aluminium leads to formation of the significant amount of harmful intermetallic phases that decrease foundry and mechanical properties of the final castings. Elimination of the detrimental iron effects might be achieved by addition of some chemical elements to change morphology of the intermetallic phase or to reduce negative effects of the phase to alloy properties. Gradual increase in the amount of nickel in the AlSi10MgMn alloy with a higher iron content is described in the presented article. From the selected additions of nickel (0,1, 0,3 and 0,5 wt.% Ni), the most favourable results were achieved by the addition of 0,5 wt. % Ni. Structural analysis of all experimental alloys (without addition and with a gradual addition of Ni) and related changes in measuring tensile and hardness properties is present.

Keywords: AI-Si-Mg alloy; correctors of iron; nickel addition; recycled materials; structural analysis

1 INTRODUCTION

In Al-Si-Mg alloys, magnesium is intentionally added (the amount is most often in the range of 0,25 to 0,45 wt. % Mg) in order to ensure the formation of an intermetallic compound Mg2Si. The formation of the intermetallic compound Mg2Si during the hardening process imparts the desired strength characteristics to the alloy with a slight decrease in ductility. The biggest customers of Al-Si-Mg alloy castings are the automotive and aerospace industries, where they are used as the highly stressed products. Quality of Al-Si-Mg alloys is besides the size, distribution, and morphology of the Si particles strongly influenced by the amount of impurities [1-3].

The presence of iron can be considered as the most common impurity in aluminum alloys. Due to its presence, iron causes the formation of harmful intermetallic phases, which negatively affect the quality of the castings. [4, 5].

Nickel is frequently recommended as iron "corrector" in the literary sources. Intermetallic compound Al9FeNi formed at the presence of Ni and Fe increases tensile strength, but also embrittlement of the castings. Simultaneous influence of Ni and Fe in Al-Si alloys with Si content above 5 wt. % expressively decreases plasticity, fluidity and corrosion resistance. A group of authors [6, 7] predicates moderate increasing of strength and elongation at indoor and elevated temperature, but only if Ni behaved as an iron modifier. Otherwise its influence, especially on the elongation is negative. Nickel reduces the coefficient of thermal expansion of Al alloys as a rule. Mutual Ni and Fe influence in Al-Si alloys enhances the alloy resistance to aggressive influence of warm water or steam [7].

Nickel is added in small amounts to Al-Si alloys in order to increase the strength properties of the casting at elevated temperatures and increase the corrosion resistance. On the other hand, the increased Ni content decreases the foundry properties of the alloy. The addition of Ni results in the formation of an Al3Ni intermetallic phase which has the solidification temperature of approximately 640 °C [8, 9].

The solubility of nickel in a solution of aluminum at a eutectic temperature is only 0,05 wt. % Ni and with decreasing temperature the solubility of Ni is at the level of 0,01 wt. % Ni. Nickel together with aluminum can, in addition to the mentioned Al3Ni phase, also form inter metallic phases AlNi, AlNi3, Al3Ni2 and Al3Ni5 [9, 10].

There is still not enough information about the nickel influence to microstructure and mechanical properties of the Al-Si-Mg alloys with elevated iron level over the tolerance. In this article, nickel influence in the amount of up to 0,5 wt. % in the AlSi10MgMn alloy with increased iron content is evaluated.

2 MATERIAL AND METHODOLOGY

Aluminium AlSi10MgMn cast alloy was used to perform experiments. Commercial aluminium alloy was firstly polluted by iron in the form of AlFe10 master alloy to obtain alloy with approximately 1 wt. % of Fe (AlSi10MgMnFe1). Increased iron level was chosen in order to get highly polluted alloy that represents secondary (recycled) material. The alloy was then poured and ingots for the next stage of experiments were prepared. Pouring temperature was 760 ± 5 °C. Such alloy was after that alloyed by nickel. The AlSi10MgMnFe1 alloy was melted in a graphite crucible using an electric resistance furnace and after the melt reached alloying temperature (780 \pm 5 °C), required amount of Ni was added. Addition of nickel in the experiment was 0,1, 0,3 and 0,5 wt. %, respectively. AlNi20 master alloy was used to nickel addition. The melts were not further purified, modified or grain refined. Prepared melts were then poured into permanent moulds preheated to 200 ± 5 °C.

Table 1 Chemical compositions of all AISi10MgMn experimental alloys

Elemente	Si	Fe	Ni	Mg	Mn	Cu	Zn	Ti
Elements	/ wt. %							
AlSi10MgMn	10,22	0,448	0,005	0,277	0,108	0,047	0,029	0,046
AlSi10MgMnFe1	9,73	0,980	0,016	0,313	0,118	0,048	0,026	0,041
AlSi10MgMnFe1 (0,1 wt. % Ni)	12,24	2,182	0,309	0,540	0,250	0,087	0,068	0,068
AlSi10MgMnFe1 (0,3 wt. % Ni)	9,93	2,153	0,273	0,297	0,100	0,043	0,082	0,032
AlSi10MgMnFe1 (0,5 wt. % Ni)	9,64	1,697	0,621	0,283	0,105	0,042	0,085	0,033

Tab. 1 shows the chemical composition of all experimental alloys. From the obtained castings, specimens to tensile testing, hardness testing and microstructure evaluation were prepared.

The microstructure analysis of the experimental alloys was performed by a NEOPHOT 32 (optical microscope) and a VEGA LMU II (scanning electron microscope) with EDX analysis of Brucker Quantax EDX analyzer. Analysis of the impact of wt. % Ni for crystallization of experimental alloys structural components was performed by thermal analysis (K-type thermocouple - NiCr-Ni).

3 RESULTS OF EXPERIMENTS

3.1 Crystallization Process and Microstructural Analysis

Based on the recorded values from the measurement of the thermal analysis, cooling curves and their first derivatives were created and the individual phase transition temperatures of the AlSi10MgMnFe1 alloy with a graded amount of Ni were determined. The nucleation temperatures of the individual structural components are given in Tab. 2.

Table 2 Characteristic transformation temperatures of structural components of AISi10MgMnFe1 alloy with a graded amount of Ni

AlSi10MgMnFe1	T _{lig} ∕ °C	$T_{\text{Fe-rich}} / ^{\circ}\text{C}$	T _{eutectic} ∕ °C
without the addition of Ni	591	575	566
0,1 wt. % Ni	587	573	552
0,2 wt. % Ni	591	573	557
0,5 wt. % Ni	588	576	551

The liquidus temperatures and nucleation temperatures of the iron-rich phases did not change significantly in all experimental alloys due to the increase in the amount of Ni and were in a narrow interval (\pm 3 °C). A more significant change occurred at the nucleation temperatures of eutectic Si, where a decrease was noted with each addition of Ni. Addition of 0,5 wt. % Ni caused a 15 °C decrease in the nucleation temperature of eutectic Si from 566 °C (without the addition of Ni) to 551 °C.

A light microscope image of the microstructure of the AlSi10MgMnFe1 alloy is shown in Fig. 1. It can be seen that platelets (needles in micrograph) of β -Al5FeSi phases are present in the interdendritic regions as well as precipitated along with the eutectic Si. Needles seem to be evenly distributed in the alloy and also length does not vary significantly.



Figure 1 Microstructure of AlSi10MgMnFe1 alloy, etch. 20 ml H₂SO₄ + 100 ml H_2O

Point EDX analysis of the needle-like phase is shown in Fig. 2. The analysis shows presence of Al, Si, Fe and low amount of Mn, which means that it is a phase β -AlSiFe, probably Al5FeSi.



Dimensions of the needle-like particles started to vary after the addition of 0,1 wt. % of Ni (Fig. 3). Formation of thicker needle-like phases was observed but along with them also high number of short and thin particles can be seen.



Figure 3 Microstructure of AlSi10MgMnFe1 alloy with addition of 0,1 wt. % of Ni, etch. 20 ml H_2SO_4 + 100 ml H_2O

After addition of 0,3 wt. % of Ni (Fig. 4), thickness and length of the iron intermetallics seem to decrease and after addition of the 0,5 wt. % (Fig. 5) the effect is even more evident.



Figure 4 Microstructure of AlSi10MgMnFe1 alloy with addition of 0,3 wt. % of Ni, etch. 20 ml H₂SO₄ + 100 ml H₂O



Figure 5 Microstructure of AlSi10MgMnFe1 alloy with addition of 0,5 wt. % of Ni, etch. 20 ml H₂SO₄ + 100 ml H₂O

Measuring of the needles length was performed on the samples to confirm the assumption. Results are shown in Tab. 3. Average length of the particles decreased after nickel addition and minimal average length was measured in the alloy with 0,5 wt. % of Ni.

Table 3 Average length of iron based needles after nickel addition					
Nickel addition / wt. %	0,1	0,3	0,5		
Average length of needles / µm	147	87	48		

Analysis of the chemical composition by point EDX analysis of the needle-like phases after nickel addition is shown in Figs. 6 to 8.



Figure 6 Point EDX analysis of needle particles iron phase in AlSi10MgMnFe1 (0,1 wt. % of Ni) alloy, SEM



Figure 7 Point EDX analysis of needle particles iron phase in AlSi10MgMnFe (0,3 wt. % of Ni) alloy, SEM

Presence of nickel in needle-like phase in alloy with addition of Ni in amount of 0,1 wt. % was not detected.

After addition of 0,3 and 0,5 wt. % of Ni, point EDX analysis shows nickel presence in the needle-like phases. Higher amount of Ni has been measured after nickel addition 0,5 wt. % (Fig. 8).



Figure 8 Point EDX analysis of needle particles iron phase in AlSi10MgMnFe1 (0,5 wt. % of Ni) alloy, SEM

3.2 Mechanical Properties

The results from static tensile test and hardness measuring are shown in Tab. 4. Tensile strength after Ni addition of 0,1 wt. % decreased. The same effect has been observed in the elongation. Higher nickel addition leads to increasing of the alloy tensile strength and also elongation. Tensile strength reached after addition of Ni values was higher than AlSi10MgMnFe1 alloy but elongation of the alloys was always lower.

Table 4 Mechanical properties of AISi10MgMnFe1 alloy with different Ni addition					
Nickel addition /	Tensile strength	Tensile strength Elongation			
wt. %	<i>Rm</i> / MPa	$A_5 / \%$	HB		
0	171	1,1	81		
0,1	145	0,7	78		
0,3	173	0,9	81		
0,5	176	1,0	79		

Measuring of the Brinell hardness does not show significant differences between results after nickel addition. The highest hardness was measured in alloy AlSi10MgMnFe1 and the same value was after addition of Ni in the amount of 0,3 wt. %.

4 DISCUSSION

Nickel influence was examined in AlSi10MgMn alloy with increased iron level. Addition of iron in commercial alloy causes formation of high number of intermetallic phases evenly dispersed in the microstructure. Point EDX analysis of the needle-like particles shows increased presence of Al, Si, Fe and also small amount of Mn. Manganese is present in the commercial alloy to decrease iron effect. In such high iron level the formation of intermetallics Al15(Fe,Mn)3Si2 with script-like morphology is low, as the Mn to Fe ratio is only 0,12 (0,5 is recommended [11]).

Addition of nickel in amount 0,1 wt. % leads to uneven distribution of iron intermetallics. Intermetallic phases present in the alloy were mainly with platelet morphology. Formation of longer and thicker phases was observed together with short and thin particles. This distribution possibly leads to decreasing of tensile strength and elongation of the final castings. Presence of Ni in plateletlike particles after addition of 0,1 wt. % of Ni was not detected by EDX analysis. Solid solubility of Ni in aluminium can be up to 0,24 wt. % [12-14]. Because of this fact, Ni in amount of 0,1 wt. % does not have to act as an iron "corrector" and tensile properties of such alloy might decrease.

Increasing of the nickel level to 0.3 and 0.5 wt. % positively influenced tensile strength and also elongation of the alloy. Better results were observed in tensile strength that reached higher values than alloy without nickel. Differences in distribution of iron-based intermetallics were not significant, but shortening of average length of needles was evident. Chemical analysis shows presence of the Ni in intermetallic phases. Presence of the Ni in ironbased phases might decrease negative influence of the particles to tensile strength and also reduce its length. Elongation of the alloy after nickel addition does not reach values of the alloy with increased iron level. Similar behaviour of the alloys after Ni addition was observed by other authors [11, 15, 16]. Elongation of the Al-Si alloys with higher iron level is mainly determined by shape and the amount of iron-intermetallics. As the distribution of platelets in the alloy with 0,5 wt. % of Ni corresponds to the alloy after iron addition, nickel influence to elongation is not significant.

5 CONCLUSION

Several conclusions can be stated from experimental measures of nickel addition to AlSi10MgMn alloy with increased iron level as follows:

- 1) Nickel addition to the alloy in an amount lower than nickel solubility might lead to decrease of mechanical properties and unfavourable microstructure.
- 2) Presence of Ni in platelet-like particles can be detected after addition of Ni in the amount 0,3 wt. % and higher.
- 3) Nickel addition in the amount 0,3 wt. % and higher increases tensile strength but elongation might decrease.
- Optimal nickel addition as the iron "corrector" is 0,5 wt. %.

Acknowledgements

The article was created as part of the VEGA grant agency project: 1/0494/17. The authors thank the agency for their support.

6 REFERENCES

- Zhang, Y. et al. (2014). Efficient use of iron impurity in Al-Si alloys. *Journal of Alloys and Compounds*, 615, 594-597. https://doi.org/10.1016/j.jallcom.2014.06.114
- [2] Blatnicky, M., Saga, M., Dizo, J., & Bruna, M. Application of Light Metal Alloy EN AW 6063 to Vehicle Frame Construction with an Innovated Steering Mechanism. *Materials*, 13(4). https://doi.org/10.3390/ma13040817
- [3] Pastirčák, R. (2014). Effect of low pressure Application during Solidification on Microstructure of AlSi Alloys. *Manufacturing Technology*, 564, 397-400. https://doi.org/10.21062/ujep/x.2014/a/1213-2489/MT/14/3/397

- [4] Medlen, D. & Bolibruchova, D. (2012). The Influence of Remelting on the Properties of AlSi6Cu4 Alloy Modified by Antimony. *Archives of Foundry Engineering*, 12(1), 81-86. https://doi.org/10.2478/v10266-012-0016-y
- [5] Ji, S. et al. (2013). Effect of iron on the microstructure and mechanical property of Al-Mg-Si-Mn and Al-Mg-Si die cast alloys. Materials Science & Engineering A, 564, 130-139. https://doi.org/10.1016/j.msea.2012.11.095
- [6] Shabestari, S. G. (2004). The effect of iron and manganese on the formation of intermetallic compounds in aluminiumsilicon alloys. *Materials Science & Engineering A*, 383, 289-298. https://doi.org/10.1016/S0921-5093(04)00832-9
- [7] Gao, J. W. et al. (2007). Effects of Na2B4O7 on the elimination of iron from aluminum melt. *Scripta Materialia*, 57, 197-200. https://doi.org/10.1016/j.scriptamat.2007.04.009
- [8] Cao, X. & Campbell, J. (2006). Morphology of β-Al5FeSi Phase in Al-Si Cast Alloys. *Materials Transactions*, 47(5), 1303-1312. https://doi.org/10.2320/matertrans.47.1303
- [9] Šerák, J. et al. (2008). Snížení obsahu železa ve slitinách AlSiCuMgFe. *Metal*, 1-6.
- [10] Petrík, J., Szarvasy, P., & Špeťuch, V. (2004). Vlastnostisilumínu s obsahomželeza, niklu a mangánu. Acta Metallurgica Slovaca, 10(2), 73-79.
- [11] Petrík, J. & Horvath, M. (2011). The iron correctors in Al-Si alloys. Annals of Faculty Engineering Hunedoara. *International journal of engineering*, 9(3), 401-405.
- [12] Taylor, J. A. (2012). Iron-containing intermetallic phases in Al-Si based casting alloys. *Procedia Materials Science*, 1, 19-33. https://doi.org/10.1016/j.mspro.2012.06.004
- [13] ASM Handbook (1992). Volume 3. Alloy Phase Diagrams. 1st. ed. USA : ASM International.
- [14] Michna, Š. et al. (2005). Encyklopedie hliniku. 1. vyd. Prešov, Slovakia : Adin.
- [15] Martinec, D., Pastircak, R., & Kantorikova, E. (2020). Using of Technology Semisolid Squeeze Casting by Different Initial States of Material. *Archives of Foundry Engineering*, 20(1), 117-121. https://doi.org/10.21062/mft.2020.012
- [16] Richtárech, L., Bolibruchová, D., Brůna, M., & Caiss. J. (2015). Influence of Nickel Addition on Properties of Secondary AlSi7Mg0.3 Alloy. *Archives of Foundry Engineering*, 20(2), 95-98. https://doi.org/10.1515/afe-2015-0046

Contact information:

Mária ŽIHALOVÁ, Ing., PhD University of Žilina, Faculty of Mechanical Engineering, Department of Technological Engineering, Univerzitná 1, 010 26 Žilina E-mail: maria.zihalova@fstroj.uniza.sk

Dana BOLIBRUCHOVÁ, prof. Ing., PhD (Corresponding author) University of Žilina, Faculty of Mechanical Engineering, Department of Technological Engineering, Univerzitná 1, 010 26 Žilina E-mail: danka.bolibruchova@fstroj.uniza.sk

Marek MATEJKA, Ing., PhD University of Žilina, Faculty of Mechanical Engineering, Department of Technological Engineering, Univerzitná 1, 010 26 Žilina E-mail: marek.matejka@fstroj.uniza.sk