

APARATURA BADAWCZA I DYDAKTYCZNA

Estimation of the Materials Reliability on Al-17wt.%Si with Cu, Ni and Mg Alloy Used in the Automotive Industry

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ABSTRACT

The results of evaluation of the reliability of hypereutectic Al-17wt.% Si with 3wt.% Cu, 1wt.% Ni and 1wt.% Mg alloy under the effect of tensile-compressive stresses were presented. AlSi alloy's used in the automotive industry in parts of pistons and cylinder heads in internal combustion engines parts. Studies were carried out on a normal-running fatigue testing machine, which was the mechanically driven resonant pulsator. The obtained results formed basis for Weibull statistical analysis, the end result of which is estimating the reliability of material (the probability survival) and establishing the motor vehicle warranty period.

Ocena niezawodności materiałowej siluminu AlSi17 z dodatkiem Cu, Ni i Mg stosowanego w przemyśle motoryzacyjnym

STRESZCZENIE

W artykule przedstawiono wyniki oceny niezawodności materiałowej siluminu nadeutektycznego AlSi17 z dodatkiem 3% Cu, 1% Ni oraz 1% Mg na podstawie wyników badań na rozciąganie i ściskanie. Stopy Al-Si stosowane są m.in. w przemyśle motoryzacyjnym na elementy tłoków i głowic. Badania przeprowadzono na normalnobieżnej maszynie wytrzymałościowej o napędzie mechanicznym z rezonansowym pulsatorem. Uzyskane wyniki stanowią podstawę do oceny niezawodności przy pomocy statystycznej analizy Weibulla, jako końcowego wyniku estymacji trwałości materiałowej (prawdopodobieństwa przetrwania) i oszacowania czasu trwania okresu gwarancji pojazdu mechanicznego.

1. INTRODUCTION

Good physical and technological properties are the reason why silumins have found wide application in various branches of the engineering industry, among others, in aviation, building industry, electronic and electro-engineering industry and, last but not least, in automotive industry. And yet, though offering numerous advantages, silumins are also characterised by a very important drawback. From the technical viewpoint this is their tendency to the formation of a coarse-grained structure, adversely affecting the mechanical properties of castings. For this reason, silumins after the refining treatment should be subjected to modification.

Of special significance are the hypereutectic silumins about 17wt.% silicon, assigned for casting of high-duty parts for automotive applications. A wide range of these alloys has been specified by Western standards. Using information given in these standards, it was the aim of the authors of this study to “enrich” the family of “slightly” hypereutectic silumins with alloying additions (Cu, Ni and Mg).

2. MATERIALS AND METHODS

To ensure safe operation of machines and equipment, the respective materials and structures are examined under the conditions of the changing loads. The conditions of fatigue testing of metallic materials have been determined, among others, by the

Polish Standard PN-76/H-04325. The said standard gives main reference terms and establishes general guidelines for preparation of the specimens and conditions under which the tests should be carried out. The performed tests most often include the tensile-compression test and bending-torsion test, made on both plain and notched specimens, and also on real items.

In statistical analysis, the calculation sheet of Excel v. 2003 made by Microsoft and programs: Statistica v. 7.1 PL offered by StatSoft and MedCalc v. 9.1.0.1 were used.

Each type of the changing load (tensile, compressive, etc.) has a corresponding form of the changing stress. Stresses of the values changing in a repetitive and continuous manner during one loading cycle form a stress cycle.

The dynamic fatigue tests were carried out applying the loads of $\sigma_{\max} = -\sigma_{\min} = 150$ [MPa], under the conditions of symmetrical tensile-compressive stresses changing in cycles. Tests were carried out on a normal-running fatigue testing machine, which in this case was the mechanically driven resonant pulsator – Figure 1.

To conduct the test properly, it was important to design a test stand in a manner such as to create the testing conditions approaching as much as possible the conditions of the real melting, casting, and solidification (Fig. 2). Maintaining the temperature, time and chemical composition constant was of key importance for further statistical analysis, and

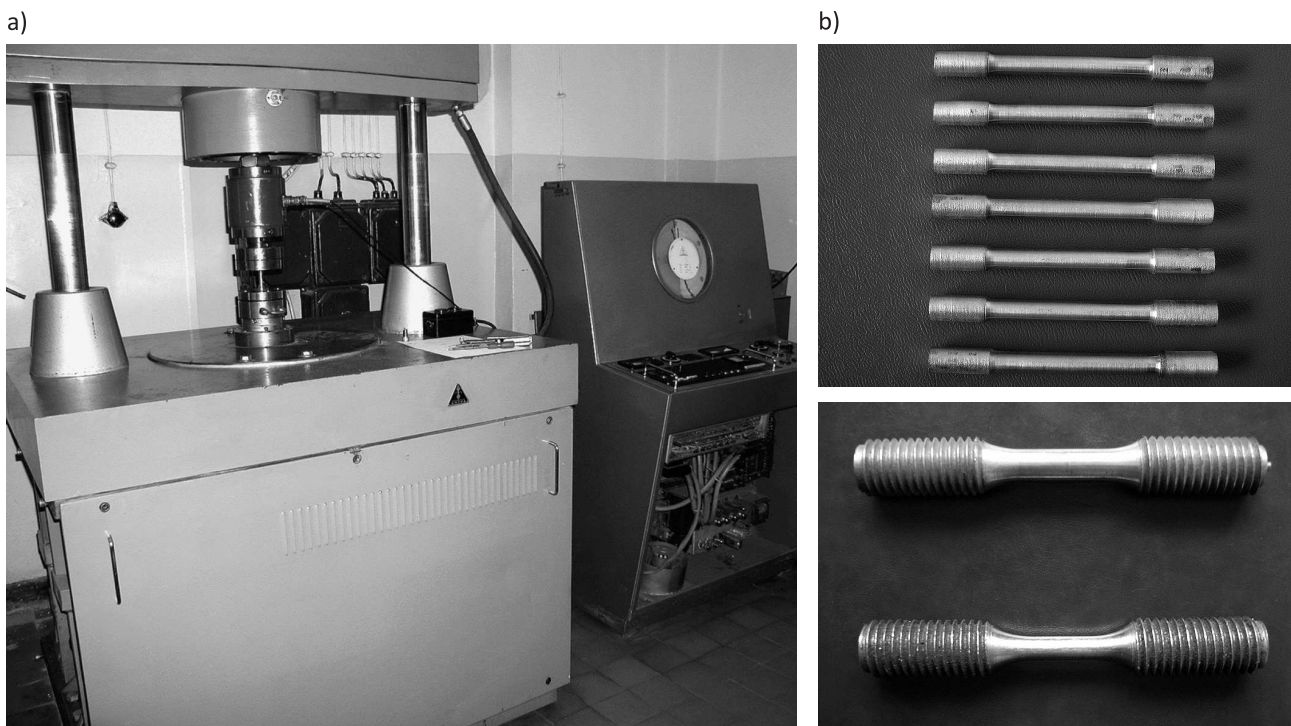


Figure 1. a) The mechanically driven resonant pulsator; b) a fatigue test specimen

for computation and correct interpretation of the obtained results.

Alloys were melted from the following charge materials: AR1 aluminium (99,96% Al), technical silicon of 98,5% purity (rest Fe and other elements), electrolytic copper (99, 98% Cu), electrolytic nickel (99, 98% Ni), cast AG10 alloy (about 10 wt.% Mg).

Melting was carried out in a 3 kg capacity magnesite crucible installed in an induction PT-600-PvG furnace, using a protective atmosphere of 2NaF and KCl (mixed in a ratio of 20 to 80%, respectively). After preheating the furnace to $\sim 820^{\circ}\text{C}$, to make

preliminary degassing of the examined alloy, the melt was refined with Rafglin-3 added in an amount of 0,3 wt.% respective of the alloy weight. The melt temperature was controlled with an NiCr-NiAl TP-202K-800-1 thermocouple immersed in the melt. Modification was carried out with phosphorus added in an amount of 0,05 wt.% in the form of a Cu-P10 master alloy ($\sim 9,95$ wt.% P). The samples were next cast into a metal mould.

The heat treatment process consisted in precipitation hardening and was basically composed of the two integral operations: solutioning at $500^{\circ}\text{C} \pm 5^{\circ}\text{C}/4$ h/, cooling in boiling water, and rapid ageing at $175^{\circ}\text{C} \pm 5^{\circ}\text{C}/8$ h/ followed by cooling with furnace.

3. THE RESULTS

3.1. ATD thermal analysis

Chemical composition of AlSi17Cu3NiMg alloy is given in Table 1.

Preserving similar melting and casting parameters, the examined AlSi17Cu3NiMg alloy were poured into a standard probe, model QC4080, plotting the temperature curves in function of time ($T=f(t)$) and a temperature derivative over time ($dT/dt = f'(t)$). Figures 3 and 4 shows an example of the plotted thermal analysis curve.

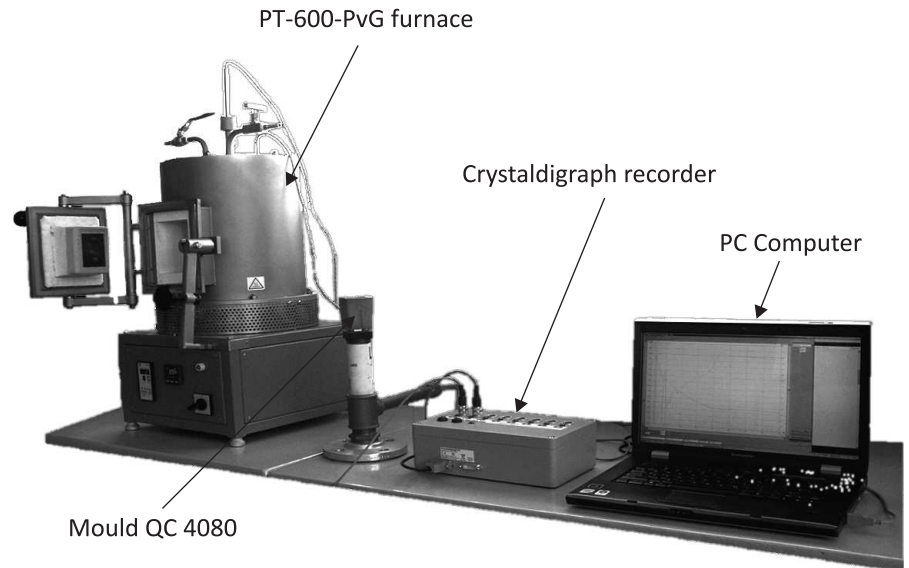


Figure 2. Test stand for melting and casting of AlSi17Cu3NiMg alloy

3.2. Statistical analysis

The Weibull probability grids were drawn, first, with a non-parametric rank - based estimation of the shape and scale parameters of a two-parametric distribution, thus enabling reading out of the characteristic value (a characteristic operational suitability time), defined as a time limit upon completing of which 63,2% of the population will have failed. This is the value of a proper parameter in scale b. From the diagrams we can also estimate the quality of fit of a regression line to the empirical data. If the quality of fit is satisfactory, we are free to proceed with the two - parametric distribution, assuming the location parameter value as equal to zero. For evaluation of the fitting quality on a probability diagram with different values of the location parameter, the determination coefficient R^2 was used. Next, the parameters of the two - and three - parametric Weibull distributions were evaluated, applying the method of maximum reliability [5]. The results of this evaluation with Hollander-Proschan and Mann-Scheurer-Ferti goodness-of-fit test are compared in Table 2.

Basing on the results of the goodness-of-fit test, it has been confirmed that, in each case, the two-parametric Weibull distribution provides a better description of the risk function than the three-parametric distribution.

Table 1. Chemical analysis of the examined silumin

Content, wt.%											
Al	Si	Cu	Ni	Mg	Fe	Mn	Ti	Zn	Cr	Pb	Sn
77,87	16,86	2,91	0,93	0,88	0,43	0,06	0,01	0,01	0,02	0,01	0,01

From evaluations obtained by the method of maximum reliability, a risk function (the damage intensity) was plotted. The lowest damage intensity and the longest operational suitability offered the AlSiCu3NiMg alloy after modification and heat treatment. In this case, the time of the operational suitability, i.e. the condition of full reliability when the component is able to perform its function in a mode consistent with the requirements, amounts to approximately 4 h. In alloy non-modified and non-heat treated, this time is nearly half as long. Also the fragment of the risk curve that illustrates the component aging time is the least steep in the case of alloy modified and heat treated (Fig. 5).

Table 3 shows the results of 50-th percentile (median) estimation of the reliability function with a 95% confidence interval.

The time corresponding to 50th percentile for the AlSi17Cu3NiMg alloy non-modified and non-heat treated amounts to about 3,65 hours with a 95% confidence interval extending from 2,86 to 4,65 hours. Hence it can be expected that 50% of all the specimens will suffer damage by the time instant $t = 3,65$ hours.

Figure 8 shows the plotted reliability diagram with a reliability function and 95% confidence intervals. The estimated values of reliability $R(t)$ (the reliability indicator) of the examined component, i.e. the probability of its failure-free operation, are compared in Table 4. The reliability index $R(t)$ is the probability that the component will be able to perform the required function under stated conditions and for a specified period of time (t_1, t_2):

$$R(t) = P(T \geq t) = 1 - F(t), \quad t \geq 0 \quad (1)$$

where: $F(t)$ – the cumulative distribution function of random variable T of the component operating time until the occurrence of damage, which is called fault (failure) of the component. The runs of function $F(t)$ are shown in Figure 5.

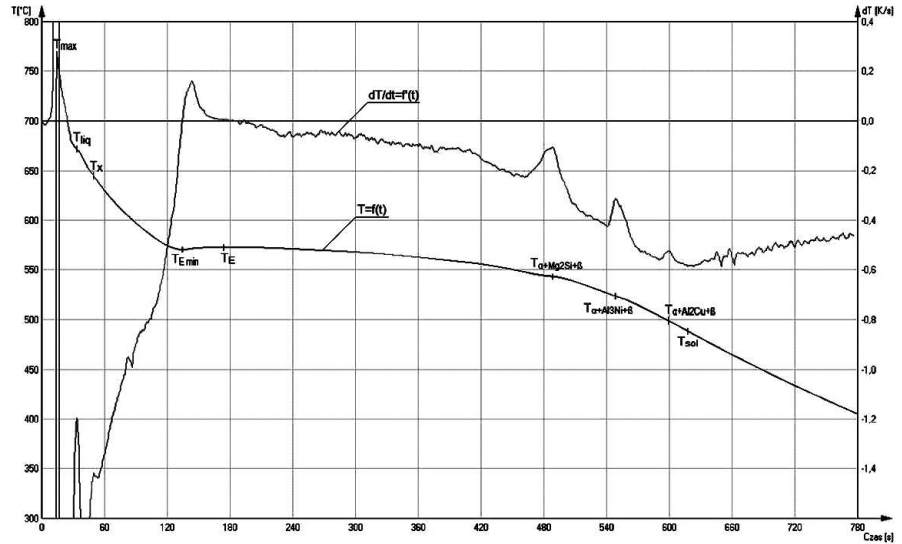


Figure 3. The ATD thermal analysis curve for AlSi17CuNiMg alloy without modification

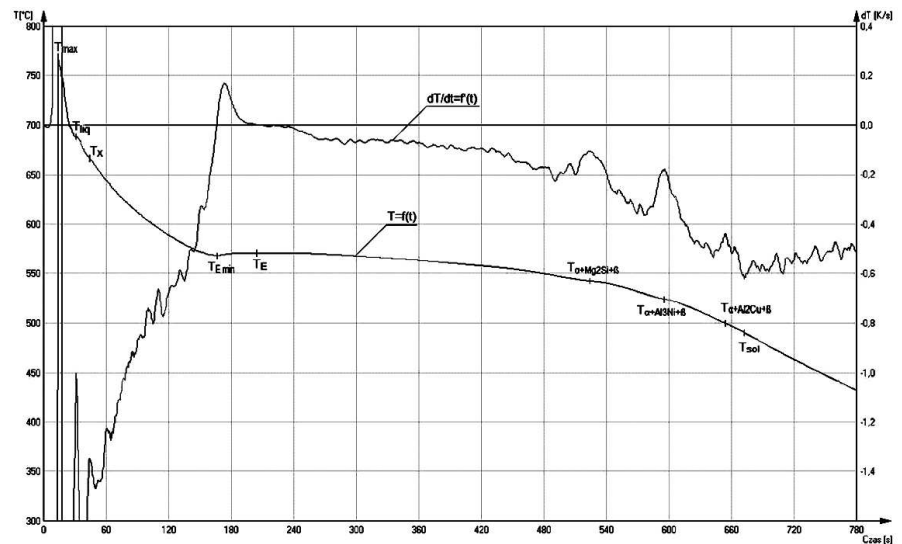


Figure 4. The ATD thermal analysis curve for AlSi17CuNiMg alloy after modification with 0,05% P

Using fitted Weibull distribution, the percentiles of reliability function with 95% confidence intervals (LCL and UCL) were computed by the method of maximum reliability (Tab. 5).

The information comprised in this table is particularly useful in determination of the expected fraction of components suffering failure after certain period of time. For example, it can be stated that 75% of non-modified and non-heat treated alloy specimens will suffer failure after the period of 4,65 hours, while for the specimens of modified and heat treated alloy this time will be prolonged to approximately 6,92 hours.

Table 2. Evaluation of parameters for the two- and three-parametric Weibull distributions and the results of Hollander-Proschan and Mann-Scheurer-Ferti goodness-of-fit test

Non-modified, non-heat treated AISiCuNiMg alloy								
Evaluation of the highest reliability for two-parametric distribution	Parameter values	Asymptotic std. errors	-95,0% LCL	+95,0% UCL	Covariance	Goodness-of-fit test	Test Value	p
Location	0,000					Hollander-Proschan	-0,1057	p=0,9158
Shape	2,866	0,804	1,654	4,965		Mann-Scheuer-Ferti	0,4229	p>0,25
Scale	4,149	0,525	3,238	5,317	-0,094			
Evaluation of the highest reliability for three-parametric distribution	Parameter values	Asymptotic std. errors	-95,0% LCL	+95,0% UCL	Covariance	Goodness-of-fit test	Test Value	p
Location	-4,287	1,442	-7,113	-1,461		Hollander-Proschan	-0,1496	p=0,8810
Shape	7,325	1,951	4,346	12,347		Mann-Scheuer-Ferti	0,5154	p>0,25
Scale	8,444	0,413	7,672	9,294	-0,132			
Modified, non-heat treated AISiCuNiMg alloy								
Evaluation of the highest reliability for two-parametric distribution	Parameter values	Asymptotic std. errors	-95,0% LCL	+95,0% UCL	Covariance	Goodness-of-fit test	Test Value	p
Location	0,000					Hollander-Proschan	0,2957	p=0,7674
Shape	6,310	1,798	3,609	11,032		Mann-Scheuer-Ferti	0,4573	p>0,25
Scale	5,653	0,308	5,080	6,290	-0,136			
Evaluation of the highest reliability for three-parametric distribution	Parameter values	Asymptotic std. errors	-95,0% LCL	+95,0% UCL	Covariance	Goodness-of-fit test	Test Value	p
Location	2,788					Hollander-Proschan	0,4507	p=0,6522
Shape	1,969	0,597	1,087	3,566		Mann-Scheuer-Ferti	0,3027	p>0,25
Scale	3,013	0,539	2,122	4,278	-0,104			
Non-modified, heat treated AISiCuNiMg alloy								
Evaluation of the highest reliability for two-parametric distribution	Parameter values	Asymptotic std. errors	-95,0% LCL	+95,0% UCL	Covariance	Goodness-of-fit test	Test Value	p
Location	0,000					Hollander-Proschan	-0,0772	p=0,9385
Shape	3,441	0,940	2,015	5,876		Mann-Scheuer-Ferti	0,5336	p>0,25
Scale	4,406	0,460	3,591	5,406	-0,076			
Evaluation of the highest reliability for three-parametric distribution	Parameter values	Asymptotic std. errors	-95,0% LCL	+95,0% UCL	Covariance	Goodness-of-fit test	Test Value	p
Location	1,605					Hollander-Proschan	0,0665	p=0,9370
Shape	1,444	0,424	0,812	2,568		Mann-Scheuer-Ferti	0,3831	p>0,25
Scale	2,850	0,722	1,735	4,681	-0,078			
Modified, heat treated AISiCuNiMg alloy								
Evaluation of the highest reliability for two-parametric distribution	Parameter values	Asymptotic std. errors	-95,0% LCL	+95,0% UCL	Covariance	Goodness-of-fit test	Test Value	p
Location	0,000					Hollander-Proschan	0,3726	p=0,7094
Shape	4,766	1,294	2,799	8,115		Mann-Scheuer-Ferti	0,6086	p>0,25
Scale	6,464	0,462	5,619	7,435	-0,121			
Evaluation of the highest reliability for three-parametric distribution	Parameter values	Asymptotic std. errors	-95,0% LCL	+95,0% UCL	Covariance	Goodness-of-fit test	Test Value	p
Location	1,137	0,750	-0,334	2,608		Hollander-Proschan	0,3742	p=0,7083
Shape	3,691	1,015	2,154	6,326		Mann-Scheuer-Ferti	0,5795	p>0,25
Scale	5,330	0,493	4,446	6,389	-0,108			

3.3. Weibull analysis

For the estimation of Weibull modulus “m” the authors used the function allowing for a relationship between the “survival” probability p (i.e. the cumulative probability that the examined property will exceed the adopted threshold limit, counted from its maximum value), the selected property of material (σ (sigma)

– that is R_m) and modulus m, given by M. Ashby and D. Jons [6]:

$$p = \exp \left[- \left(\frac{\sigma}{\sigma_0} \right)^m \right] \quad (2)$$

where: σ_0 (σ_0) is the value for which 37 % of samples exceed this value in respect of the examined property.

Table 3. The values of 50-th percentile of the reliability function with a 95% confidence interval for the examined AISi17Cu3NiMg alloy

AISi17Cu3NiMg alloy	Time, t [h]	-95,0% LCL	+95,0% LCL
Non-modified and nonheat treatment	3,65	2,86	4,66
Modified and nonheat treatment	5,33	4,80	5,19
Non-modified and heat treatment	3,96	3,23	4,85
Modified and heat treatment	5,98	5,21	6,86

Table 4. The reliability $R(t)$ values as estimated by the method of maximum reliability and by a non-parametric method

Non-modified and non-heat treatment		Modified and non-heat treatment		Non-modified and heat treatment		Modified and heat treatment	
Time to failure	R(t)	Time to failure	R(t)	Time to failure	R(t)	Time to failure	R(t)
1,18	0,97	2,94	0,98	1,78	0,95	2,88	0,97
1,22	0,97	3,61	0,94	1,96	0,94	3,92	0,91
2,19	0,85	4,22	0,85	2,34	0,89	4,82	0,78
2,69	0,74	4,82	0,69	2,88	0,79	4,91	0,76
3,44	0,55	5,03	0,62	3,51	0,63	5,23	0,69
3,67	0,49	5,19	0,55	3,93	0,50	5,49	0,63

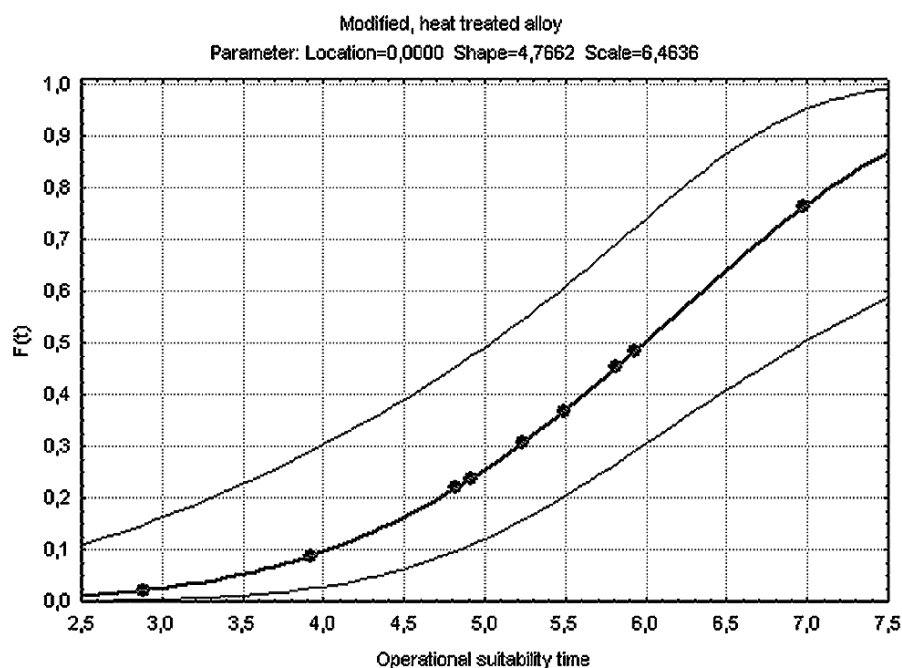


Figure 5. Plotted cumulative distribution function $F(t)$ (fault) with a confidence interval for parameters evaluated by the method of maximum reliability

Table 5. Percentiles of reliability function with 95% confidence intervals computed by the method of maximum reliability

Percentiles	Non-modified and non-heat treatment			Modified and non-heat treatment		
	Time, t	-0,95% LCL	+0,95% LCL	Time, t	-0,95% LCL	+0,95% LCL
25	2,69	1,98	3,64	4,64	4,06	5,30
50	3,65	2,87	4,65	5,33	4,81	5,92
75	4,65	3,55	6,09	5,95	5,29	6,69
Percentiles	Non-modified and heat treatment			Modified and heat treatment		
	Time, t	-0,95% LCL	+0,95% LCL	Time, t	-0,95% LCL	+0,95% LCL
25	3,07	2,37	3,96	4,98	4,17	5,94
50	3,96	3,23	4,85	5,99	5,22	6,87
75	4,85	3,89	6,03	6,92	5,95	8,06

Function (2) was reduced to its linear form by double two-sided logarithmic operation done with a natural logarithm:

$$\ln \left[\ln \left(\frac{1}{p} \right) \right] = m \ln \frac{\sigma}{\sigma_0} \quad (3)$$

Using the obtained empirical pairs of values (x_i ; y_i), parameters of the model of linear function of the type $y = a \cdot x$ were estimated. For this purpose an Excel -from the packet of tools – Data Analysis called Regression was used [7, 8]. To estimate the coefficients of the function of regression, the Tool uses a method of optimisation based on minimalisation of the sum of least squares (SLS) of the deviations of empirical points from a model curve. The results of the estimation of the coefficient a of the function of regression, which at the same time is the searched value of modulus “ m ” for the examined cast alloy, are shown in Figure 6.

Analysis of regression and correlation				
Linear model $y=ax$: ($\ln(\ln(1/p))=\ln(\text{Sigma}/\text{Sigma0}) \cdot m$)				
Regression statistics				
Multiple	0,9756			
R square	0,9517			
Sdjusted R square	0,8608			
Standard error	0,3890			
Observations	12			
	Coefficients	Standard error	t Stat	p
Modul Weibulla (m):	69,8362	4,08756	17,085	0,0000
	Lower 95%	Upper 95%		
	60,83958	78,83288		

Figure 6. Determination of Weibull modulus value “ m ” for the examined alloy ($\sigma_0 = 197$)

The obtained model of the searched function of regression for the examined alloy is as follows:

$$\ln \left[\ln \left(\frac{1}{p} \right) \right] = 69,84 \ln \frac{\sigma}{197} \quad (4)$$

A graphic representation of the model obtained in double logarithmic system is shown in Figure 7.

Table 6 shows the effect of chemical composition of the examined cast AlSi17Cu3NiMg alloys after modification and casting into metal mould on the values of Weibull modulus “ m ” determined for the tensile strength. The results of the investigations indicated that the highest value of Weibull modulus “ m ” had AlSiCuNiMg alloys [7]. Therefore these alloys were subjected to further examinations

to estimate the effect of process history on their properties.

Each of the indices should have the highest value possible and as such can serve as a criterion in the choice of material best matching the assumed operating conditions. Table 7 shows the effect of process history during manufacture of the examined cast alloys on the value of Weibull modulus “ m ”. It has been observed that the highest values of Weibull modulus and of the calculated indices had the samples of the modified AlSi17Cu3NiMg alloy cast into metal mould and subjected to heat treatment, which decided this alloy was selected for further investigations of the thermal fatigue behaviour and was proposed as a material for industrial applications.

3.4. Structure examinations

Metallographic sections were prepared according to standard procedure on Struers polishing machine.

The structure on the specimen surface was examined and recorded under an Olympus GX-71 microscope.

The morphology of powders and local chemical composition of alloys were determined on a Hitachi microscope with EDX attachment made by Norah using a Voyager software.

Examples of AlSi17Cu3NiMg alloy microstructures in as-cast condition and after modification 0,05wt.% phosphorus (CuP10 master alloy) are shown in Figure 8.

4. SUMMARY AND CONCLUSIONS

As follows from the characteristic values of the solidification temperature of AlSi17Cu3NiMg alloy modified with CuP10, the temperature T_{max} was similar in all experiments, (780°C) proving that similar

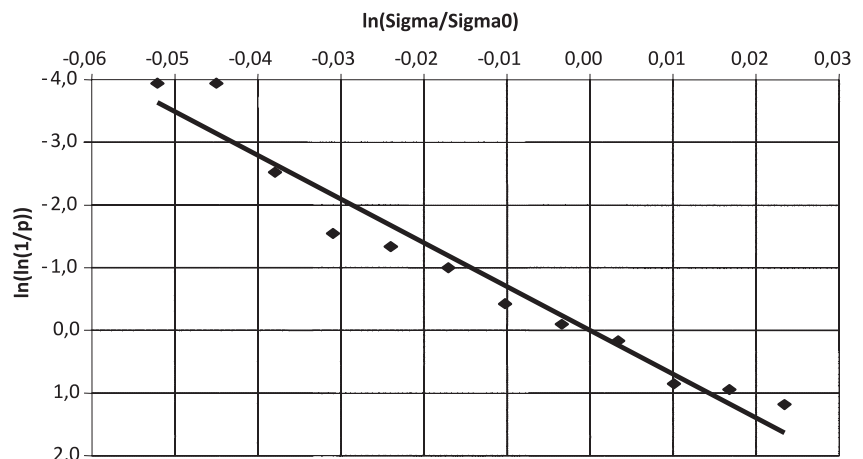


Figure 7. Graphic representation of a model of the survival function plotted in double logarithmic system

Table 6. Effect of the chemical composition of cast AlSi17Cu3NiMg alloys after modification and casting into metal mould on the values of Weibull distribution m determined for R_m

Rm [MPa]	Metal mould and modification process				
	AlSi17	AlSi17Cu3	AlSi17Cu3Ni	AlSi17Cu3Mg	AlSi17Cu3NiMg
Mean	147,79	165,98	166,69	175,21	215,92
Weibull modulus	42,86	74,75	67,48	71,54	82,58
σ_0	149	167	168	176	217
SD	4,03	3,77	2,83	3,10	3,04

Table 7. Effect of the chemical composition of cast AlSi17Cu3NiMg alloy on the values of Weibull modulus “m” determined for the tensile strength R_m

Rm [MPa]	Non-modified and non-heat treatment		Modified and non-heat treatment		Non-modified and heat treatment		Modified and heat treatment	
	Sand mould	Metal mould	Sand mould	Metal mould	Sand mould	Metal mould	Sand mould	Metal mould
Mean	16,38	188,33	195,38	215,92	192,50	207,57	221,75	235,26
Weibull modulus	59,71	68,03	69,83	82,58	67,84	70,78	105,79	116,22
σ_0	178	190	217	217	193	209	222	236
SD	3,75	3,17	3,78	3,04	3,84	4,15	2,84	2,56

conditions of melting and casting of the examined alloy were maintained. The crystallisation temperature of the silicon crystals of α phase (T_{liq}) assumed the highest value of 690°C for the AlSi17Cu3NiMg alloy after modification phosphorus. This fact is well proved by data given in literature [9-10] and is consistent with the phase equilibrium diagram of Al-Si system [11].

The crystallisation temperature of a binary α (Al)- β (Si) eutectic assumed similar value and amounted to 568°C, preceded by a two-step temperature drop (T_{minE} 566°C). When the α (Al)- β (Si) eutectic crystallisation ended, an exothermic effect was observed on the ATD solidification curve; most probably it originated from crystallisation of a ternary eutectics containing the intermetallic: α +Mg₂Si+ β , α +Al₃Ni+ β and α +Al₂Cu+ β phase. There eutectics solidifies at a temperature of 543°C, 528°C and 500°C after modification with CuP10 a considerable drop of this temperature. The end of crystallisation is observed to take place within the temperature range of 480 to 489°C, which is characteristic of the temperature T_{sol} .

The images of metallographic structures indicate that the structure of casting containing an addition of phosphorus has undergone some modifications. The precipitates are fine and distributed evenly in the matrix. The modification with CuP type master alloys also refines the silicon crystals and distributes

them evenly in the matrix of aluminium solution, but in this case, besides the commonly encountered fine silicon crystals, other crystals of slightly different morphology appear as well. It is quite possible that these uncommon crystals are responsible for the appearance of the additional heat effect. In other words, they crystallise as secondary precipitates, and the appearance of thermal effect is a consequence of this situation (T_x). It is also worth noting that the second additional thermal effect is less intense than the effect caused by the precipitation of the crystals of primary silicon. Perhaps the secondary crystallisation of silicon characterised by different morphology results in a lower rate and volume of the heat evolution. The traditional metallographic examinations can contribute little to the identification of other intermetallic phases.

For more complete explanation of the proeutectic crystallisation it is necessary to carry out additional examinations by RTG, SEM, and – especially – by calorimetry. These additional investigations are expected to enable more complete identification of the phase that crystallises in hypereutectic silumins between the temperature T_{liq} and the solidification point of α (Al)- β (Si) eutectic mixture.

Only the results of the fatigue tests which allow for the time of loading should be considered a rational and efficient tool in evaluation of the operating reliability of the responsible parts of machines and

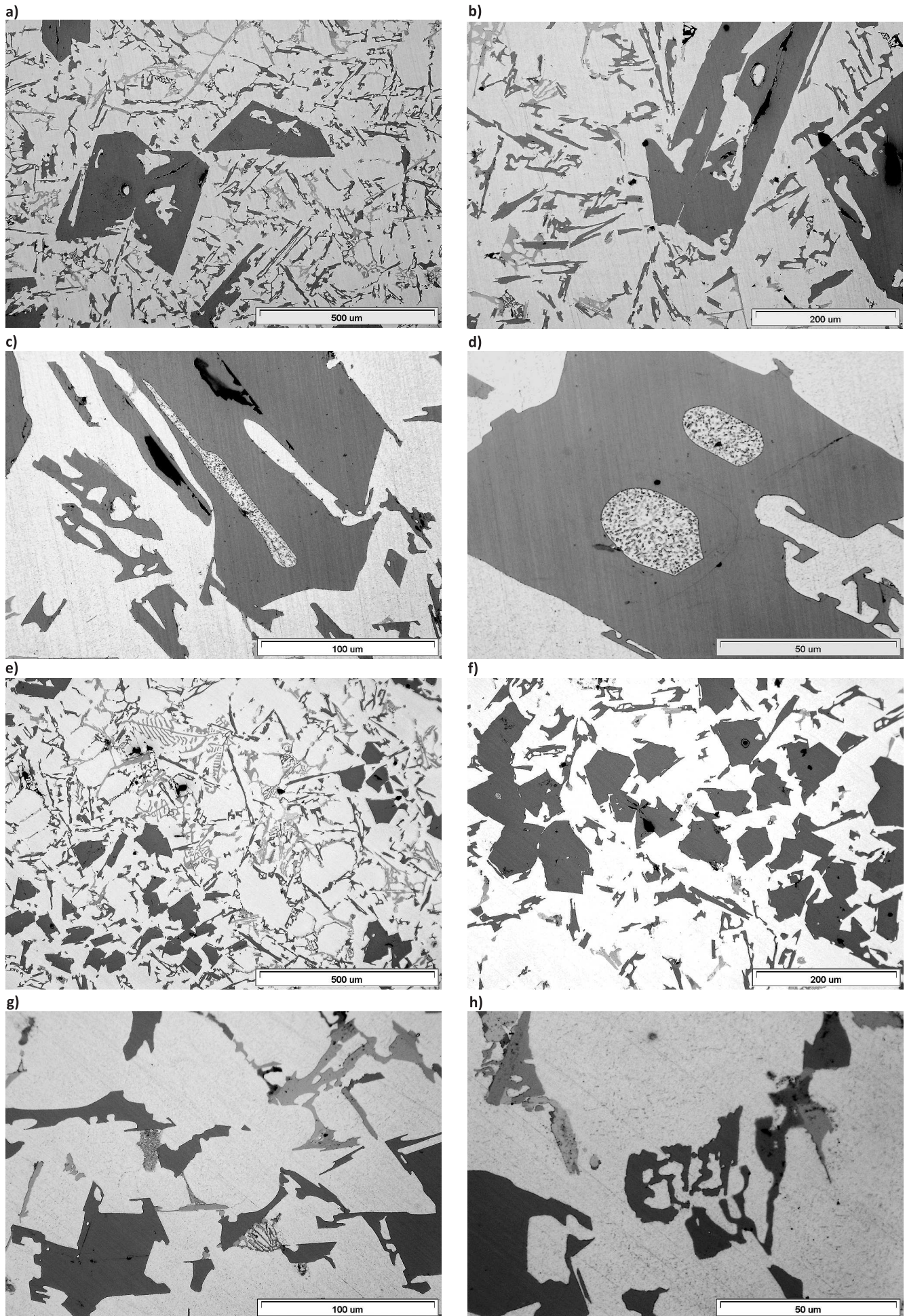


Figure 8. The microstructure of AlSi17Cu3NiMg alloy: a-d) no modified, e-h) after modification

equipment. The method based on analysis and on the two- and three-parametric Weibull distributions, evaluating parameters by the method of maximum reliability and by a non-parametric method based on ranks, provides the reliable and complex information

on, among others, the up time in function of the failed components percent fraction, the cumulative risk in function of up time, the reliability function with estimated percentiles and confidence intervals, and the probability function of reliability with a cumulative distribution function of this probability.

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