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Processing and properties of Al–Si–Fe–Cr *in situ* composites

INTRODUCTION

The concept of cost effective production of composite material by *in situ* precipitation of the reinforcement in the metal matrix has been developed during the last decade. The fabrication consists in mixing the liquid or solid alloy with selected solid metal or non-metal powders, which results in exothermic chemical reaction between the alloy and the powders leading to the precipitation of reinforcing phases. The new alloy may have the advantages of MMC (strong, stiff, abrasion resistant) without their weaknesses (in *in situ* reinforcement is thermodynamically stable, material is cheap and recyclable) [1–5]. The *in situ* reinforcement has very clean surfaces enabling better wetting and bonding between them and the matrix. The sub-micron size of the precipitates is possible which do not embrittle the material and may constitute the effective reinforcement once their properties (e.g. stiffness) and the morphology (e.g. aspect ratio) are well selected.

In situ particulate-reinforced aluminium-based composites were successfully fabricated by hot pressing and reaction sintering or reaction pressing and extrusion of Ti, Al and B powders [6]. In [7] the TiB has been obtained as *in situ* reinforcement of Al–Cu (4.5 % Cu). Without subjecting the composite to secondary processing such as extrusion, *in situ* TiB reinforcement (10 vol %) resulted in tensile strength increase of 21 % whereas the *in situ* TiB₂ reinforcement (3.3 vol %) improved the tensile strength by 5.6 %. Alternative technique for fabrication of *in situ* composites is Reactive Gas Injection (RGI) method, in which chemical reaction occurs between the reactive (C or N containing) gas and liquid alloy [8]. During crystallization products of the reaction form the reinforcing phase. Synthesis of TiC carbides in Al and Cu matrices using methane as reactive gas was described in [9]: The TiC particles of the mean diameter $1 \div 4 \mu\text{m}$ and volume fraction up to 12 % were formed. Analysis of the potential of RGI method shows that it can be used to the production of wide range of composites with matrices of Al, Ti, Cu, NiAl, NiTi, TiAl reinforced with particles of carbides, (TiC, TaC, B₄C, SiC) or nitrides (Si₃N₄, AlN, BN) [9]. In [10] liquid metal processing SHSB (self-propagating high temperature synthesis bath) has been used to Al–TiC synthesis (4–10 % TiC). The compression strength of the composites increased by 400 % as compared with pure Al. The important increase in abrasion resistance has been demonstrated.

The group of potential reinforcement for aluminum alloys are aluminum rich intermetallic compounds. In ordinary Al alloys they usually form during solidification or during subsequent thermo-mechanical processing in aluminum based alloys, either from unwanted impurities (e.g. Fe and Si) or by intentionally added elements [11]. The intermetallic compounds in ingot–cast aluminum alloys are reasonably understood. However, the limited solid solubility of transition metals (TMs) such as Fe, Mn, Cr etc. and the nature of the Al-TM phase diagrams limit the volume fraction of intermetallic compound that can be achieved (in a traditional way) to provide appreciable dispersion strengthening. The progress on

nonequilibrium processing of aluminum powder alloys using gas atomisation, [12], mechanical alloying [13] has led to new class of commercially significant aluminum alloys strengthened by dispersoids of aluminum rich intermetallic compounds. These alloys derive their excellent mechanical and corrosion properties from a number of metastable intermetallic compounds that exhibit unique structures, thermal stability (up to 450 °C) and desirable properties in aluminum matrix. Attention is paid to the Al-Fe-Si intermetallic compounds. The focus is on the type and crystallographic data of intermetallics depending on the cooling rate and the processing route [11].

The present work is the continuation of the study on fabrication of *in situ* Al-Si matrix composites reinforced with intermetallic Al-Fe-Si or Al-Fe-Cr-Si compounds obtained by the reaction of liquid Al–Si alloy with Fe or Cr powders [4]. The Fe powders were selected for low cost, low processing temperature, reactivity with Al which should facilitate precipitation of reinforcement *in situ* and hexagonal crystallographic lattice of Al-Fe-Si compound which promotes the formation of elongated („fibre”) precipitates. Chromium was supposed to form the precipitates of improved hardness and abrasion resistance which could be predicted from the data of microhardness of chromium silicides [4, 11]. However, *in situ* precipitates of Al₄FeSi obtained in [4] did not exhibit important microhardness and the tensile properties were low due to the large size of the precipitates and porosity of the alloy. The fabrication of Al-Si composites with Cr powders proved time consuming yet microhardness of Al₁₃Cr₄Si₄ precipitates was promising. Consequently, in order to take advantage of the beneficial characteristics of both sources the fabrication of composites with source of both Fe and Cr has been attempted. Some results of microstructural examination of the composites obtained have been presented in [5]. In the present study the previous work has been continued and the effect of reinforcement on the mechanical behaviour as well as abrasion resistance has been studied.

EXPERIMENTAL PROCEDURE

Materials

The hypereutectic and eutectic Al-Si alloys (25 and 11 % Si) were used as matrix materials. The particles of reinforcement were obtained by precipitation *in situ*, as a result of self propagating chemical reaction between the liquid Al-Si alloy and selected precursor elements for intermetallics, introduced in the form of powders or alternatively as a metallic stirrer „dissolving” in the liquid.

Chemical composition of materials used is given in table 1.

Composites fabrication

The *in situ* composites were made by stir-casting technique, using the following procedures. The powder was gradually added inside the funnel formed in the liquid aluminum alloy while stirring the mixture continuously (265 rev/min.). In order to avoid oxidation of substrate powder particles the use of high chromium steel stirrer as

an alternative source of Fe and Cr was investigated. The stirrer, (10 wt % of Al alloy) was supposed to „dissolve” totally during agitation process. The weight of the stirrer was 10 % of the liquid Al alloy. The extent of the reaction between the source material and the liquid alloy has been controlled by varying the time of the process. In the present work the mechanical agitation time varied from 15 min. to 2 hours. The constant temperature of 680 °C was adopted. The cylindrical samples $\Phi 20 \times 30$ mm were moulded by gravity casting in metallic moulds. Data on fabrication of composites are given in table 2.

Table 1. Chemical composition of materials used

Tabela 1. Skład chemiczny materiałów użytych do wytwarzania kompozytów

Matrix materials		
1	AlSi11	Al, 11 % Si
2	AlSi25	Al, 25 % Si
Source materials		
1	Fe powders (diameter of the particles A) 0.04-0.07 mm, B) max. 0,45 mm	
2	Cr powders (max. diameter of the particles 0.2 mm)	
3	Alloy steel Fe, 12% Cr 1.65% C, 0.35% Mn 0.35% Si, 0.7% V	

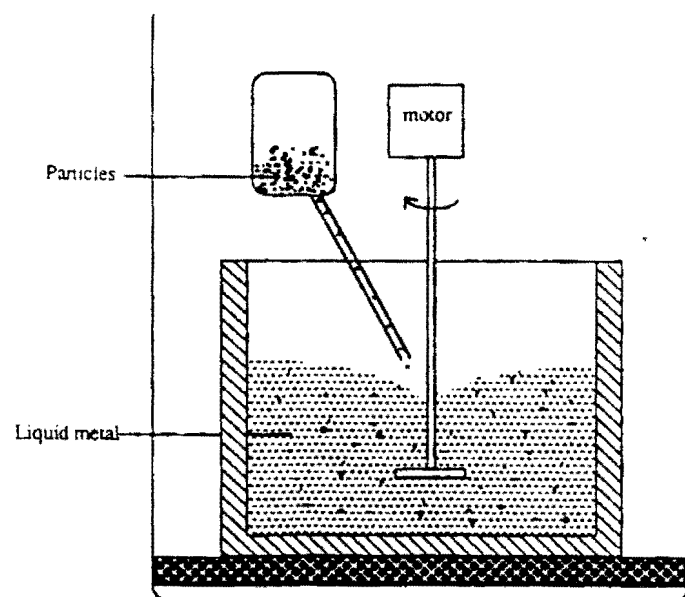


Fig. 1. Scheme of experimental set-up for composites fabrication

Rys. 1. Schemat stanowiska do wytwarzania kompozytów

Table 2. Data on fabrication of composites

Tabela 2. Dane o sposobie wytwarzania kompozytów

	Matrix alloy	Source material	Temperature	Max. stirring time [min.]	Moulding
1	Al-Si(1)	Fe(A)5 wt % + Cr5 wt %	680 °C	120	warm metallic mould
2	Al-Si(1)	steel (12 % Cr) 10 wt %	680 °C	60	warm metallic mould
3	Al-Si(2)	steel (12 % Cr) 10 wt %	680 °C	120	warm metallic mould

Composites characterization

In order to identify and characterize the composites the morphology of the materials obtained by stir-casting has been examined using scanning electron microscope coupled with x-ray diffraction microprobe analyser EDX. The maps of distribution of the elements have been made followed by approximate identification of phases. The volume fractions of precipitates have been determined by quantitative image analysis.

Mechanical behaviour of composites was studied by means of compression tests performed on cylindrical samples (10 mm \times 15 mm) using Instron hydraulic testing machine. Elastic modulus and compression strength as well as yield strength and fracture strain have been estimated.

As Al-Si *in situ* composite materials are expected to have improved wear resistance abrasion tests were performed. Dry sliding wear tests were carried out under unlubricated conditions at room temperature in a wear testing machine using the pin on-reciprocating-plate mode for wear testing. Cylindrical samples (6 mm \times 20 mm) cut from the moulded composite cylinders, were used as pins sliding against the reciprocating plate of hardened tool steel. Before the tests the surface of the sample was polished using a 3 μ m diamond paste. The wear rate was studied at fixed velocity 0.24 m/s over a sliding distance of 1200 m and the pressure between the sliding surfaces was 1 MPa. The wear rate was calculated by measuring the weight loss. The samples were weighted before and after each wear test to 0.01 mg. Each test was repeated three times and the mean value was used for the analysis of the results. Finally, Rockwell (scale B) hardness of composites and microhardness of the reinforcement phases were estimated.

RESULTS AND DISCUSSION

Morphology of the composites

The SEM images shown in Fig. 2 and 3 illustrate the evolution of microstructures of the composites (Al-Si + 5 wt. % Fe + 5 wt. % Cr) at different stages of the fabrication process: after incorporation of Cr powder (Fig 2), after the incorporation of Cr + Fe powders and stirring the mixture for 60 (Fig. 3a) and 120 minutes (Fig. 3b). Chemical composition of the precipitates (Fig. 1b) as well as the maps of Cr and Fe distribution in the final composite illustrate changes taking place in the material during the process. Larger, round Al-Cr rich phase which is in reaction with Fe and smaller, elongated Al-Fe rich precipitates are observed (chemical composition relevant to the maps has been given in [5]). The increasing brightness of the initially very dark precipitates of both Al-Cr rich and Al-Fe rich phases indicate the gradually progressing diffusion and reaction of Cr and Fe leading to the complex intermetallic compounds (the chemical composition indicates that they are the nonequilibrium phases of the type $Al_{18}Cr_2Fe$ and $Al_{19}Fe_2Si_2Cr$). Microhardness of the intermetallic phases seen in Fig. 3 was $HV_{50} = 450 \div 530$. The volume fraction of the precipitates estimated by image analysis was $V_f = 20 \div 22$ %.

These results have been next compared with the previous study [4] in which alternatively 10 wt. % Fe or 10 wt. % Cr powders were incorporated in hypereutectic AlSi25 alloy. In the first case the elongated, acicular intermetallic precipitates of $Al_{4,5}SiFe$ ($V_f = 16 \div 18$ %) have been identified by XRD (Fig. 4ab). The ternary phase diagram (Al-Si-Fe) confirmed that the Al_4SiFe or $Al_{4,5}SiFe$ compounds appear between 25 to 600 °C. The crystallographic analysis showed that the monoclinic $\beta-Al_{4,5}SiFe$ crystal exhibit a strong anisotropy in the c direction. The microhardness of the platelets was 270 HV0.02

In the case of Cr source powder [4] (Fig. 4c) after stirring for 11 hours large precipitates of cubic $Al_{13}Cr_4Si_4$ phase ($V_f = 30 \div 35$ %) were identified (570 HV0.02).

Since the incorporation of source materials in the form of powders was difficult and time consuming an attempt has been made to introduce Fe and Cr by „dissolving” the chromium steel blades of the stirrer. However, the processing proved longer and it was difficult to „dissolve” all the material in short time, consequently it was impossible to control the amount of Fe and Cr introduced into the liquid alloy. Microstructure of composite obtained by reaction of the steel stirrer with liquid hypereutectic alloy is shown in Fig. 4d. Two distinct phases are seen, round particles and thin needles ($V_f = 23 \div 25$ %). Point analysis of both the needle-like phase and particles as well as EDX maps of distribution of elements

in the composite structure showed that both Al-Si-Fe phases differ only slightly in Fe, Si and Cr contents [5].

Morphology of composites obtained by dissolving the steel stirrer in eutectic AlSi11 alloy is demonstrated in Fig. 5. After mixing the alloy for 90 minutes the volume fraction of precipitates was $V_f = 12 \div 15 \%$.

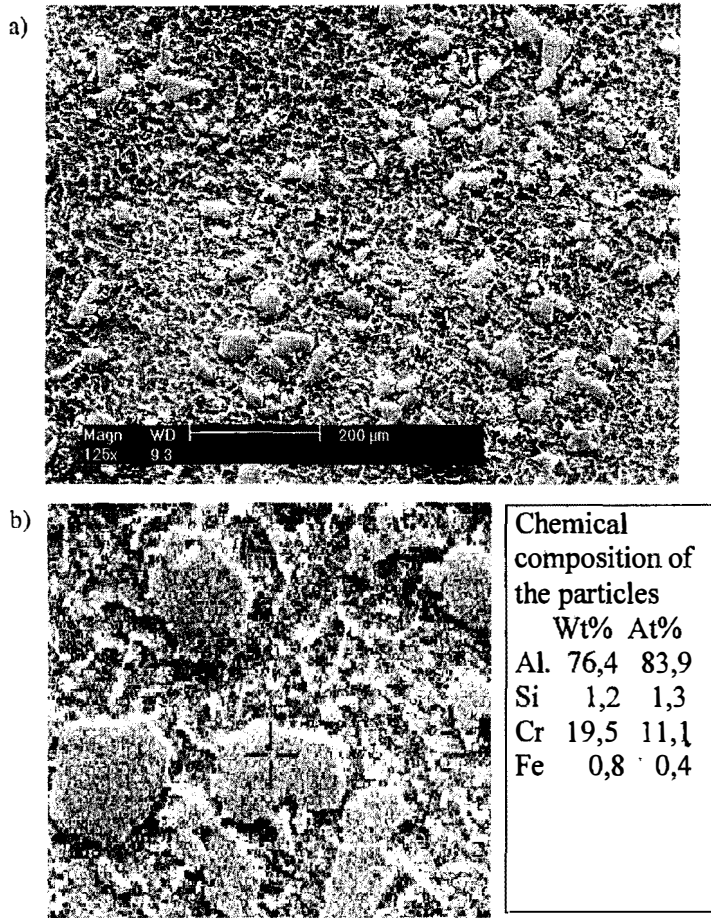


Fig. 2. SEM micrograph of eutectic Al-Si matrix composites after incorporation of Cr powder (mixing the liquid for 15 minutes) a) magnification 125 \times , b) detail of the micrograph shown in Fig. 2a, indicating the chemical composition of the "+" marked precipitate
Rys. 2. Mikrofotografia SEM kompozytu o osnowie stopu eutektycznego Al-Si po wprowadzeniu proszku Cr (czas mieszania 15 min.). a) powiększenie 125 \times , b) szczegół struktury pokazanej na rys. 2a, skład chemiczny wydzielenia zaznaczonego krzyżem

Abrasion tests results

As the potential application of the composites fabricated in this study are wear resistant elements some results of abrasion tests have been analysed in order to compare the behaviour of composite materials with pure Al-Si11 alloy. As is shown in Fig. 6 illustrating the weight loss over a distance of 1200 m, at 1 MPa pressure and the sliding velocity of 0,24 m/s for all fabricated composites unreinforced Al-Si11 alloy suffered higher weight loss than *in situ* composites based on Al-Si11 obtained in this study. The lowest weight loss was observed for hypereutectic matrix composite (+ Fe) the highest (among the composites) for AlSi11 Fe+Cr. The improved performance of hypereutectic matrix composite proves the importance of matrix properties. The effect of harder matrix seems to be stronger than that of the precipitates.

Mechanical characterization

In Fig. 7 plots of Brinell hardness of the composites have been shown. It is evident that *in situ* reinforcement has beneficial effect

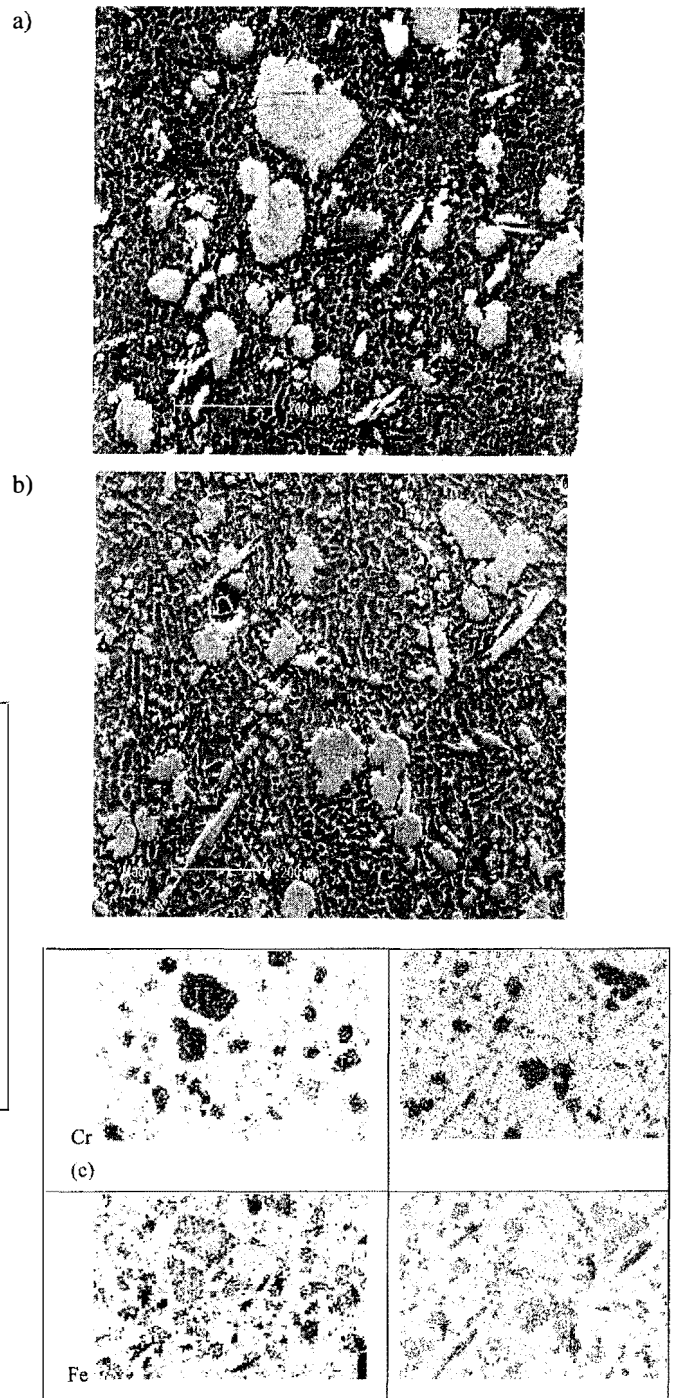
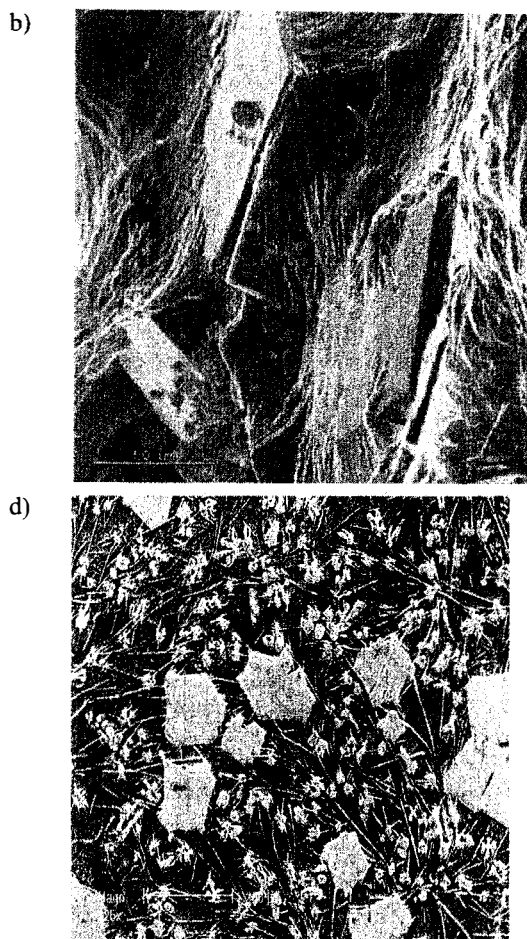
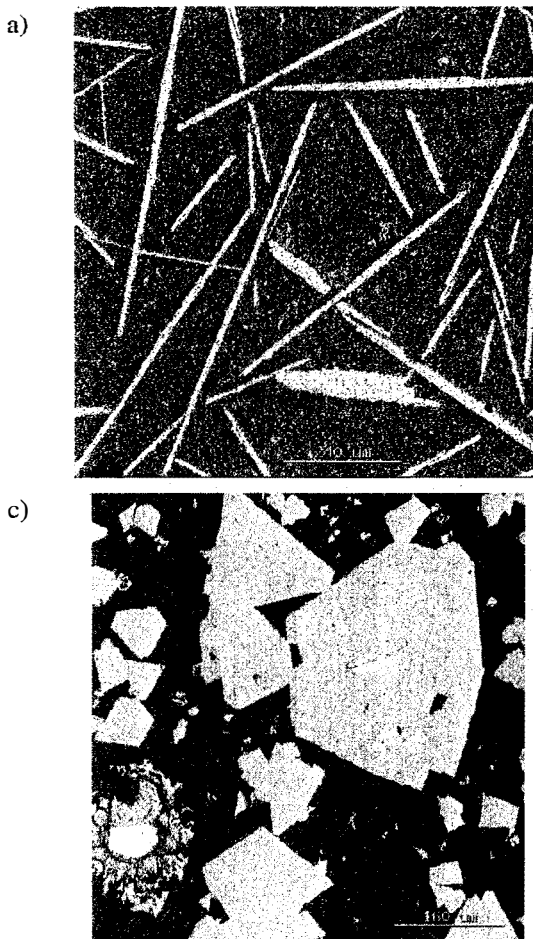


Fig. 3. SEM micrographs of eutectic Al-Si matrix composites after incorporation of Cr+Fe powders and stirring a) 60 minutes, b) 120 minutes c) maps of distribution of Cr and Fe in the above composites
Rys. 3. Mikrofotografie SEM kompozytu o osnowie stopu eutektycznego po wprowadzeniu proszku Cr i Fe i mieszaniu przez a) 60 min, b) 120 min, c) mapy rozkładu Cr i Fe w przedstawionych wyżej mikroobszarach

on the hardness of Al-Si alloys. However, small differences in Brinell hardness indicate that this parameter is not sensitive enough so as to be used in assessing the effect of a given type of reinforcement. The results of microhardness tests showed that *in situ* reinforcements obtained in this study differed importantly, (microhardness ranging from 270 HV0.02 for Fe-rich phases to 650 HV0.02 for Cr-rich phases) However these important differences had little effect on abrasion resistance expressed by weight loss, investigated in this study (Fig. 6).

In order to compare the behaviour of composites under compression loading graphs of compression modulus, strengths and fracture strains have been plotted for five composites and pure AlSi11 alloy. As can be observed the compression modulus of



Rys. 4. Mikrofotografie SEM przedstawiające a) mikrostrukturę kompozytu AlSi25 + Fe (strzałka wskazuje fazę międzymetaliczną $Al_{4,5}FeSi$ powstałą *in situ*), b) płytkowa budowa fazy międzymetalicznej $Al_{4,5}FeSi$, c) mikrostruktura kompozytu AlSi25 + stal (12% Cr), czas mieszania 120 min. Widoczne liczne wydzielania faz Al-Fe-Si-Cr o lekko zróżnicowanym składzie

Fig. 4. SEM microphotographs showing a) microstructure of the composite AlSi25 + Fe (the arrow is pointing to the intermetallic *in situ* formed phase $Al_{4,5}FeSi$), b) morphology of the intermetallic phase $Al_{4,5}FeSi$, c) microstructure of the composite AlSi25 + Cr (bright precipitates of $Al_{13}Si_4Cr_4$), d) microstructure of the composite AlSi25 + steel (12% Cr), mixing time – 120 minutes. Numerous precipitates of Al-Fe-Si-Cr of slightly varying composition



Fig. 5. SEM microphotograph showing microstructure of the composite after mixing the AlSi11 alloy with steel (12% Cr) stirrer for 60 minutes. Precipitates of *in situ* formed reinforcements against the eutectics modified by Cr and Fe can be observed

Rys. 5. Mikrofotografia SEM przedstawiająca mikrostrukturę kompozytu powstałego na skutek mieszania stopu AlSi11 za pomocą stalowego (12% Cr) mieszadła przez 60 minut. Widoczne cząstki utworzone *in situ* na tle eutektyki zmodyfikowanej przez Cr i Fe

composites are highest for hypereutectic matrix composites. The effect of volume fraction and shape of the reinforcement is also noticeable. In sample AlSi25 + Fe, exhibiting the highest modulus, the precipitates were long and „short fibre”-like. Yet, microstructure of second hypereutectic alloy based composite (steel stirrer) was dominated by the particulate precipitates (Fig. 4d). Although the volume fraction of the reinforcement obtained for this material

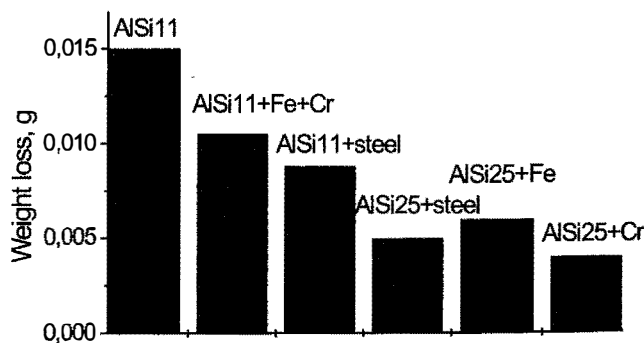


Fig. 6. Weight loss over a distance of 1200 m in dry sliding wear tests using the pin on-reciprocating-plate mode for wear testing (pressure 1 MPa, 0,24 m/s)

Fig. 6. Zużycie ścierny na dystansie 1200 m w próbie tarcia suchego przy zastosowaniu urządzenia z ruchem posuwisto zwrotnym (nacisk 1 MPa; 0,24 m/s)

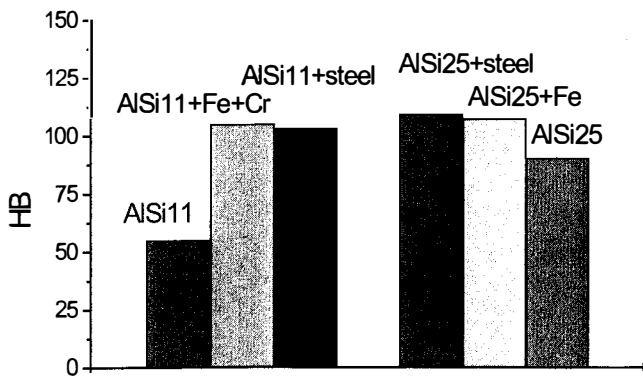


Fig. 7. Comparison of Brinell hardness for different composites
Rys. 7. Porównanie twardości Brinella dla badanych kompozytów

($V_f = 23 \div 25\%$) was higher as compared with the former composite ($V_f = 16 \div 18\%$) the form of the reinforcements seem to play more important role in improving the compression modulus of the composite.

The lowest modulus obtained for eutectic composite 2 (steel) can be explained by lower volume fraction of the reinforcement ($V_f = 12 \div 15\%$) as compared with Fe + Cr samples ($V_f = 20 \div 22\%$).

Ultimate compression strength and yield strength (Fig. 8) are similar for the composites tested with notable, 400% rise as compared with matrix material AlSi11. The compression fracture strain was highest for AlSi11 ($\epsilon_f = 35\%$) as compared with $\epsilon_f = 7\%$ for brittle, hypereutectic alloy reinforced with coarse needles. Then the considerable difference is observed in the behaviour of other composites. Except the properties of the matrix this is due to the size, form and volume fraction of the precipitates. Small, round particles proved more favourable than the coarse „fibers”.

The observation of fracture surface confirmed the difference in the behaviour of the composites. Those based on AlSi-rich matrix fractured by multiple, brittle fracture parallel to the compression sample axis, the eutectic alloy-based composites exhibited ductile mode of fracture (at 45° to the sample axis).

From the results of compression behaviour it may be concluded that the most interesting are the properties of eutectic alloy reinforced with intermetallic compounds resulting from the reaction of liquid AlSi11 alloy with steel stirrer, since this material possesses the combination of strength stiffness and plasticity. This is also in agreement with abrasion results for the matrix of eutectic alloy. The composites based on hypereutectic Al-Si alloy exhibited lower weight loss, because of the effect of the matrix rather than precipitates.

Further studies performed for this kind of materials indicated that it is the refinement of microstructure of the composites that plays a major role in improvement of their mechanical behaviour.

CONCLUSIONS

1. Incorporation of combination of Fe, and Cr into the Al-Si alloy leads to the formation of intermetallic phases constituting the effective reinforcement of Al alloy.
2. The source materials for *in situ* reaction in liquid aluminum may be both in the form of powders as well as solid rod gradually “dissolving” due to reaction with the liquid. However, the second type is less efficient, requires longer times or gives lower volume fractions of reinforcement
3. The *in situ* reinforcements of Al-Si alloys constitute the complex intermetallic phases, e.g. $Al_{4.5}FeSi$ or $Al_{13}Fe_4Cr_4$ of medium hardness ranging from 270 HV0.02 for Fe-rich phases to 650 HV0.02 for the Fe-Cr-Si phases.
4. The volume fractions of the reinforcements obtained ranged from $V_f = 12 \div 35\%$.

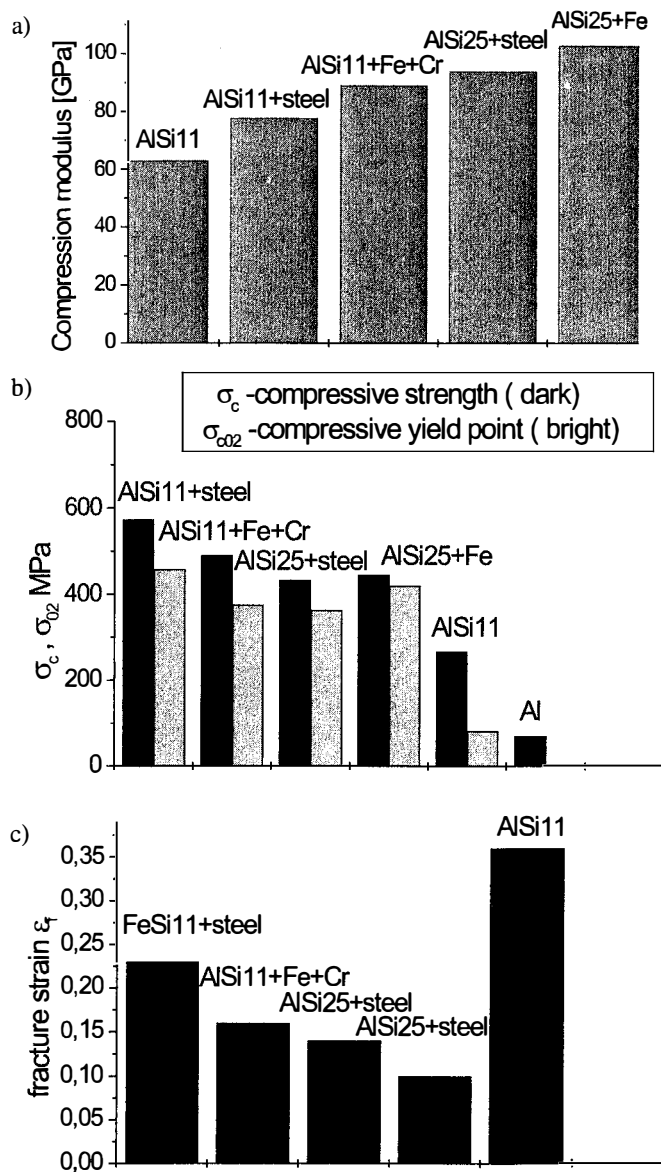


Fig. 8. Compression tests results for different composites: a) elastic modulus, b) compression ultimate and yield strengths, c) fracture strain
Rys. 8. Wyniki próby ściskania dla otrzymanych kompozytów: a) moduł sprężystości, b) wytrzymałość na ściskanie i umowna granica plastyczności, c) wydłużenie przy zniszczeniu

5. The *in situ* composites produced in this study exhibited improved compression behaviour, as compared with AlSi11 alloy i.e. modulus – by $30 \div 60\%$, compression strength – by $60 \div 250\%$, yield point $\sigma_{0.02}$ by $300 \div 400\%$, fracture strain lower by about 30% for AlSi11 + steel.
6. All composites exhibited improved abrasion resistance as compared with pure AlSi11 alloy.
7. The most important improvement in the behaviour of the AlSi11 alloys as far as the combination of mechanical properties in compression and wear resistance are concerned was obtained for composites produced using the chromium steel stirrer as a source of Fe and Cr.
8. In order to obtain further improvements in the mechanical behaviour of composites the refinement of *in situ* precipitates must be achieved.

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