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# THE PRECIPITATION STRENGTHENING OF DIRECTIONALLY SOLIDIFIED Al Si Cu ALLOYS

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## ABSTRACT

The structure of directionally solidified eutectic alloys is generally composed of matrix and reinforcing phases. The second phase has a fibre or flocks form and is embeddded in the matrix. Both phases reveal a sharp, fibre texture with its axis of low indiced crystallographic direction parallel to the grow direction. The reinforcing phase of Al-Si alloys is built of silicon-rich and the matrix of aluminium- rich solid solutions. The growth direction of the both phases is near <200>. The mechanical properties of the directionally solidified Al-Si alloys are relatively low. The increase of these properties was investigated in an Al-Si hypoeutectic alloy with additions of 2 wt.% and 4 wt.% copper. These alloys were solution treated at 530$^{o}$C for an hour, quenched in water and aged at 200$^{o}$C for up to above 40 hours. The large precipitates of $Al_2Cu$ present in D.S. samples partly dissolved and after the ageing they precipitated in the form of small platelets, which significantly increased the mechanical properties. Hardness changes were measured in the course of ageing. The structure was investigated by means of scanning microscopy ( with Link SEM analysis), x - ray phase analysis and lattice parameter measurements.

## STRESZCZENIE

Struktura kierunkowo krystalizowanych stopów charakteryzuje się obecnością głównie dwu faz: umacniającej i osnowy. Faza umacniająca posiada postać włókien lub płatków otoczonych osnową. Obie fazy charakteryzują się silną orientacją uprzywilejowaną typu osiowego o nisko wskażnikowym kierunku krystalograficznym równoległym do kierunku wzrostu kompozytu. Faza umacniająca jest zbudowana z bogatego w krzem roztworu stałego z aluminium podczas gdy osnowa, z bogatego w aluminium roztworu stałego z krzemem. Orientacja uiprzywilejowana obu faz jest bliska <200>. Mechaniczne własności kierunkowo krystalizowanych stopów Al-Si są relatywnie niskie. Podwyższenie ich własności przebadano na przykładzie pod-eutektycznych stopów Al-Si z dodatkami 2 cięż.% i 4 cięż.% miedzi. W stopach tych, po kierunkowej krystalizacji miedź tworzy duże wydzielenia. Stopy te były ujednoradniane w 530$^{o}$C w czasie 1 godz., przesycane w wodzie i następnie starzone w 200$^{o}$C w czasie powyżej 40 godz. Duże wydzielenia miedzi $Al_2Cu$ w próbkach po kierunkowej krystalizacji zostały częściowo rozpuszczone i po starzeniu wydzielały się w postaci małych płytek podnoszących znacznie własności mechaniczne badanych stopów. Zmiany twardości mierzono na próbkach po różnych czasach starzenia. Badania struktury wykonano metodami mikroskopii skanningowej (z analizą SEM) oraz rtg. analizy fazowej i pomiaru parametru sieciowego osnowy badanych stopów..

## INTRODUCTION

The structure of directionally solidified near eutectic alloys consists of a matrix and reinforcing phases. According to models of eutectic growth the morphology of the reinforcing phase and its growth during solidification is related to undercooling, solidification rate and alloy composition. At small solidification rates the silicon precipitates appear as flakes, but at high rates (near of 1000 μm/s) as fibre. [1,2,3].The shape of the reinforcing phase has a great influence on the mechanical properties of the D.S. composites. According to Steen et al. [4] the flake structure causes a strong increase of ductility in comparison with fibre structure, which gives higher UTS of the D.S. alloys. The type of crystallographic orientation and crystallographic relationship of phases is very important for the properties of these composites. In the Al-Si alloys this relationship is dependent on the growth parameter of directional solidification (growth rate) and alloy composition (active additions). According to Shamzuzzoha [5] and Kobayashi [6] the most frequent orientation relationship is as follows: [110] Si II [100] Al $\alpha$ although Eskin [7] reports a different relationship [111] Si II [010] Al $\alpha$. The [100] effective growth direction is obtained at intermediate rate of twinning in Si - phase and relationship of the type [100] Si II [001] Al $\alpha$ [8] takes place. The Al - Si composites reveal a limited tensile strength. Higher mechanical properties could be achieved by strengthening of the matrix in the precipitation hardening process. According to Eskin [9] the highest UTS can be obtained for the alloy with 4 wt.% Cu and 10 wt.% Si ( about 460 MPa). The $Al_2Cu$ precipitates appear in the D.S. Al - Si alloy in very disperse form after heat treatment considerably increasing the alloy strength [9,10]. The precipitation of $\Theta''$ and $\Theta'$ due to the presence of copper [7,10] has been reported. The $\Theta'$ particles precipitate in a plate or needle shape. After heat treatment only the binary $\Theta'$ phase and no ternary, silicon containing phases in the Al - Si - Cu alloys were found [11], despite Gowiy et al. [10] stated that copper in Al - Si alloy was present not only as $Al_2Cu$ but also as complex precipitates with silicon. Copper and other alloying elements enter either in the matrix solid solution or form the intermetallic compounds during solidification [11, 12]. The object of the present study is to investigate the influence of copper addition in the Al - Si alloy on the mechanical properties of the directionally solidified hypo- eutectic Al - Si alloy after heat treatment.

## 2. EXPERIMENTAL PROCEDURE

Two aluminium alloys of composition: AlSi10Cu2 (1) and AlSi10Cu4 (2) - (all in wt. %) were chosen for the present study. They were melted and cast in an argon atmosphere in a Balzers furnace and directionally solidified at 28 μm/s. The alloys were homogenised in argon atmosphere at 530°C during an hour and quenched into RT water. The ageing was carried out in argon atmosphere at 200°C for up to above 40 hours. Hardness was measured using Vickers method at 5 KG load. The scanning microscope examinations were performed using PHILIPS XL-30 apparatus. The quantitative analyses of solid solution and precipitates were carried out with EDS method in Link-Isis apparatus. Texture investigations were performed on PHILIPS PW 1710 diffractometer using texture goniometer PW 3020 in back reflection region on cross sections of the D.S. bars. The preferred orientations were recorded as pole figures for Si precipitates and the matrix. The conventional x - ray analysis was carried out on PHILIPS PW 1710 diffractometer. The lattice parameters of the matrix of both investigated alloys ($a_0$) were obtained on the basis of phase analysis (at smaller`angle velocity) and calculated in the computer way using DHN - PDS program.

## 3. RESULTS AND DISCUSSION

### 3.1. Results of hardness measurements

The hardness changes of the alloys aged at 200°C were tested with Vickers method. Fig.1 shows the plot of hardness versus ageing time up to above 40 hours. It can be seen that the alloy with 2 % copper addition attains much smaller hardness than the another (4 % copper). The hardness maximum for the alloy with smaller copper content is only 88 KG/mm$^2$ while the alloy with higher copper content has about 117 Kg/mm$^2$ after 8 and 10 hours of ageing, respectively. Then, for longer ageing times, above 20 hours, the hardness decreases.

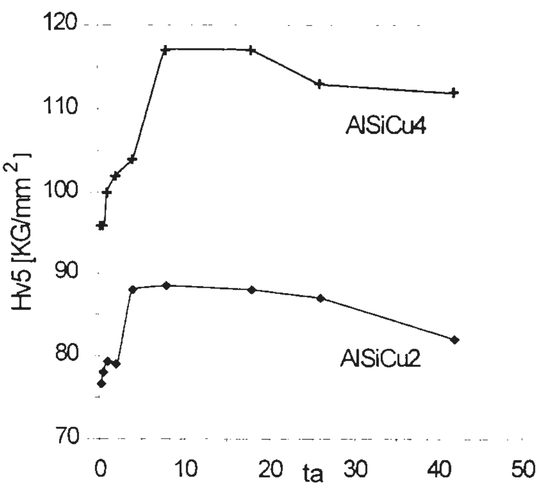


Fig.1. Hardness of alloys 1 and 2 as a function of the ageing time at 200°C

Rys.1. Zmiany twardości stopów 1 i 2 w funkcji czasu starzenia w 200°C

### 3.2. X - ray phase analysis

According to the x - ray phase analysis the D.S. AlSiCu alloy is composed of three phases: Al- solid solution as the matrix, Si - precipitates as the reinforcing phase and aluminium - copper (Θ') preciptates. In the as grown state these Θ' precipitates form large particles, frequent on the boundaries of the matrix and reinforcing phase. In their diffraction pattern, the {200} Al - diffraction line (d = 2.03 A) has a very high intensity, which suggests the sharp fibre <200> preferred orientation of the matrix. The diffraction lines of the reinforced Si - phase do not reveal such significant intensity differences as the matrix and it is not possible to observe its texture only from the phase analysis.

The changes of lattice parameter distincly show the formation of $Al_2Cu$ - phase (Fig.2). In the as quenched state the matrix solid solution reaches the values of the lattice parameter typical for very high content of copper: 1.85 wt.% and 3.60wt.% copper for alloys 1 and 2, respectively. The rate of precipitation is higher in the alloy 1 and reveal a maximum just after 8 hours of ageing, which is slightly under the value of pure aluminium after the longest ageing time of investigation. The

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Philips Analytical PC-APD, Diffraction software

Sample identification: Pr.12(C)/90h/200°C
Data measured at: 20-mar-1996 11:00:00

Diffractometer type: PW1710 BASED
Tube anode: Co
Generator tension [kV]: 30
Generator current [mA]: 40
Wavelength Alpha1 []: 1.78896
Wavelength Alpha2 []: 1.79285
Intensity ratio (alpha2/alpha1): 0.500
Divergence slit: 1/2°
Receiving slit: 0.2
Monochromator used: YES

Start angle [°2θ]: 30.010
End angle [°2θ]: 109.970
Step size [°2θ]: 0.020
Maximum intensity: 20967.04
Time per step [s]: 1.000
Type of scan: CONTINUOUS

Minimum peak tip width: 0.02
Maximum peak tip width: 4.00
Peak base width: 9.00
Minimum significance: 0.75
Number of peaks: 28

| Angle [°2θ] | d-value α1 [] | d-value α2 [] | Peak width [°2θ] | Peak int [counts] | Back. int [counts] | Rel. int [%] | Signif. | |
|---|---|---|---|---|---|---|---|---|
| 31.035 | 3.3434 | 3.3507 | 0.480 | 30 | 7 | 0.1 | 2.37 | |
| 33.140 | 3.1365 | 3.1433 | 0.160 | 666 | 7 | 3.2 | 16.09 | Si |
| 34.300 | 3.0334 | 3.0400 | 0.080 | 53 | 7 | 0.3 | 1.10 | Al2Cu |
| 35.090 | 2.9672 | 2.9737 | 0.200 | 10 | 7 | 0.0 | 0.75 | |
| 41.575 | 2.5204 | 2.5258 | 0.480 | 12 | 7 | 0.1 | 1.20 | |
| 42.715 | 2.4561 | 2.4614 | 0.160 | 15 | 7 | 0.1 | 0.82 | |
| 44.200 | 2.3734 | 2.3786 | 0.120 | 34 | 7 | 0.2 | 1.74 | Al2Cu |
| 44.905 | 2.3421 | 2.3472 | 0.200 | 894 | 7 | 4.3 | 24.67 | Al |
| 47.140 | 2.2369 | 2.2418 | 0.160 | 36 | 7 | 0.2 | 1.05 | |
| 49.355 | 2.1424 | 2.1471 | 0.240 | 32 | 7 | 0.2 | 1.71 | Al2Cu |
| 52.365 | 2.0272 | 2.0316 | 0.180 | 20967 | 7 | 100.0 | 84.27 | Al |
| 52.495 | 2.0226 | 2.0270 | 0.060 | 11946 | 7 | 57.0 | 1.11 | |
| 55.500 | 1.9211 | 1.9253 | 0.140 | 445 | 7 | 2.1 | 7.01 | Si |
| 55.645 | 1.9165 | 1.9206 | 0.080 | 303 | 7 | 1.4 | 0.83 | Al2Cu |
| 62.060 | 1.7352 | 1.7390 | 0.640 | 10 | 7 | 0.0 | 1.44 | |
| 66.235 | 1.6372 | 1.6407 | 0.080 | 119 | 7 | 0.6 | 1.08 | Si, Al2Cu |
| 71.010 | 1.5402 | 1.5435 | 0.240 | 4 | 7 | 0.0 | 0.93 | Al2Cu |
| 75.915 | 1.4543 | 1.4574 | 0.400 | 41 | 7 | 0.2 | 1.22 | Al2Cu |
| 77.215 | 1.4335 | 1.4366 | 0.080 | 216 | 7 | 1.0 | 0.94 | Al |
| 77.475 | 1.4294 | 1.4326 | 0.120 | 100 | 7 | 0.5 | 0.80 | |
| 78.890 | 1.4079 | 1.4109 | 0.060 | 35 | 7 | 0.2 | 1.99 | Al2Cu |
| 82.475 | 1.3570 | 1.3599 | 0.100 | 66 | 7 | 0.3 | 1.41 | Si, Al2Cu |

precipitation process is not finished. The lattice parameter of alloy 2 increases less significantly than in alloy 1, and reaches $a_o$ = 4.0489 after about 30 hours. It corresponds to the content of 0.20 wt. % Cu and attains the value of pure aluminium after 62 hours of

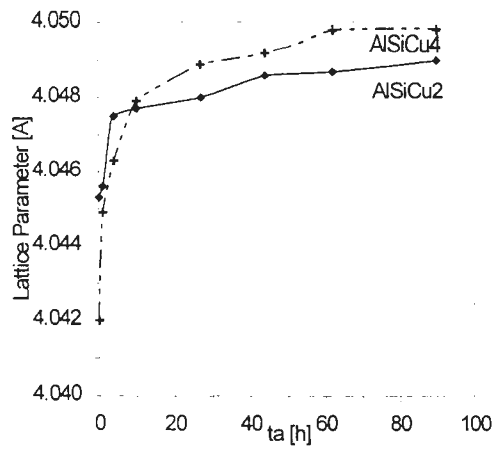


Fig.2. Lattice parameter as a function of ageing time at 200°C in the alloys 1 and 2

Rys.2. Zmiany parametru sieciowego stopów 1 i 2 w funkcji czasu starzenia w 200°C

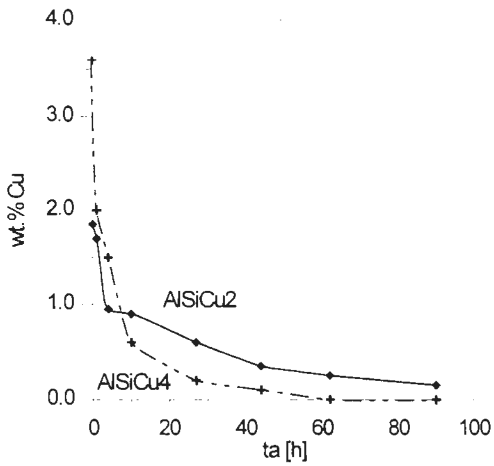


Fig.3. The dependence of copper content in the matrix vs ageing time at 200°C in alloys 1 and 2

Rys.3. Zależność zmian zawartości miedzi w osnowie w funkcji czasu starzenia w 200°C w stopach 1 i 2.

ageing (Fig.3). The above was calculated on the basis of the results of Elwood and Silcock [14]. The given above is in a good agreement with the hardness results.

**3.3. Preferred orientation analysis**

Both phases Al - matrix and reinforcing Si - precipitates are well crystallographically aligned. The preferred orientations are of fibre type with the fibre axis nearly parallel to the growth axis. The crystallographic relationship of Al- and Si - phases, according to the present investigation are like in [8]. The texture of the matrix is sharp and the deviations of the <200> orientation from the growth axis is of about 10°. In the reinforcing, silicon phase the deviations of <200> crystallographic direction from the growth axis are smaller than in the matrix but with a small contribution of the <111> crystallographic direction which agrees with [7]. It is known from [13] That the texture perfection of the matrix in the annealed sample slightly improves. The <200> component slightly decrease, while the <111> component increases in the reinforcing phase and for the ageing time of about 1 hour. For longer ageing times (about 20 hours at 530°C), the texture changes to more weaker and more distorted.

**3.4. Scanning microscopy analysis**

The fields of aggregates of silicon flakes, the precipitation free zones of the disordered distribution and large, stable $Al_2Cu$ precipitations can be seen in the microstructure of the investigated D.S. alloys in as grown state (Fig.4) After homogenisation and quenching the shape of the silicon precipitates is unchanged, but

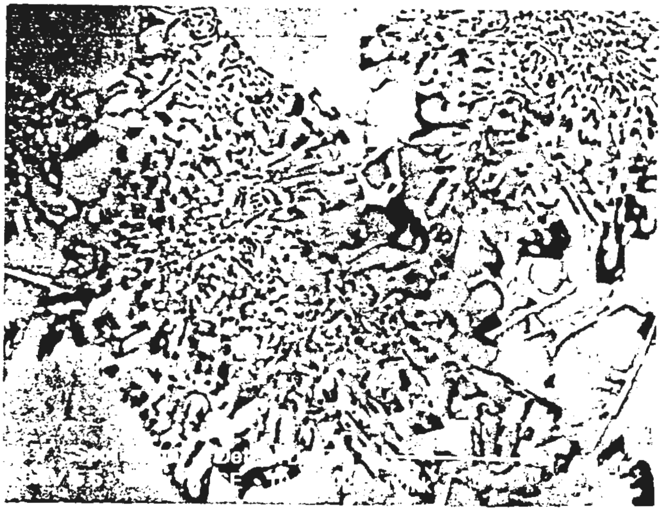

Fig.4. Scanning electron microstructure of directionally solidifiedalloy 2

Rys.4. Mikrostruktura skanningowa stopu 2 po kierunkowej krystalizacji

the $\Theta$ - phase dissolves and copper forms solid solution with the aluminium matrix. A small amount of this phase remains in the unchanged form, particularly in alloy 2 with larger copper content, where only 3.6% copper dissolved in the matrix (Fig.6). In the course of ageing the $\Theta$ and $\Theta'$ phases appear through the precipitation of GPI I/II zones. The $\Theta'$ precipitate in the plate-like form, which grows with ageing time.

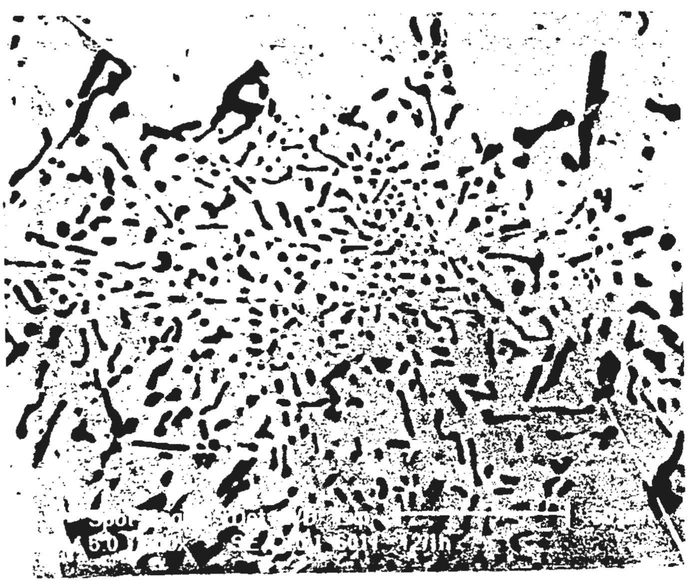

Fig.5. Scanning electron microstructure of alloy 2 aged 1 hour at 200°C

Rys.5. Mikrostruktura skanningowa stopu 2 starzonego 1 godzinę w 200°C

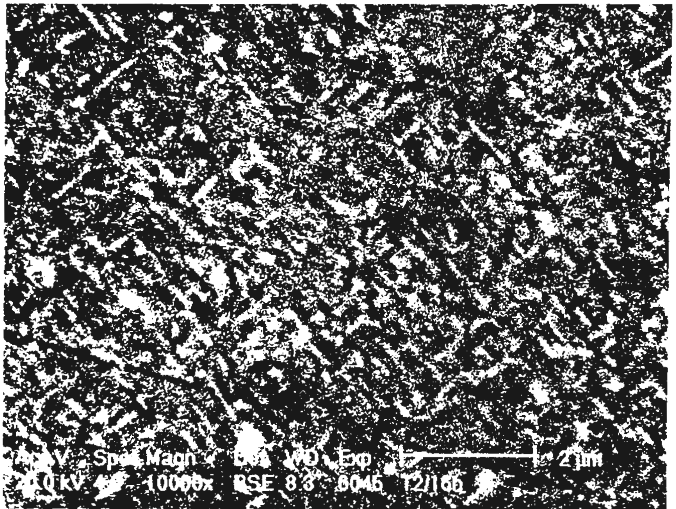


Fig.6. Scanning electron microstructure of alloy 2 matrix aged 16 hours at 200°C

Rys.6. Mikrostruktura skanningowa osnowy stopu 2 starzonej 16 godzin w 200°C


Fig.7. Scanning electron microstructure of alloy 2 matrix aged 44 hours at 200°C

Rys.7. Mikrostruktura skanningowa osnowy stopu 2 starzonej 44 godziny w 200°C

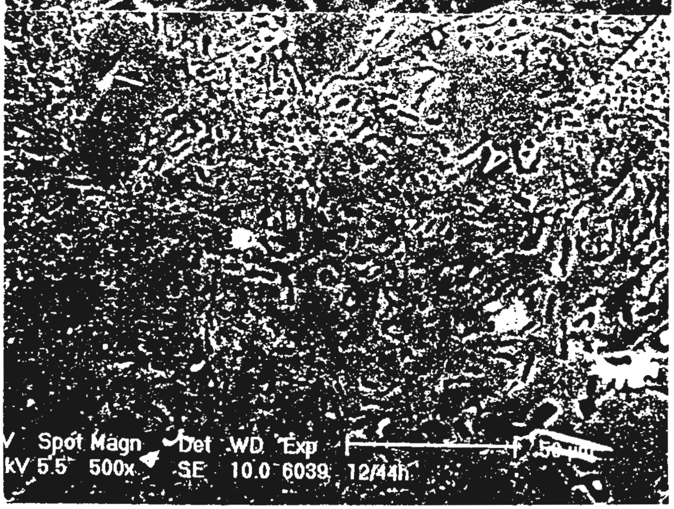


Fig.8. Scanning electron microstructure of the alloy 2 aged 44 hours at 200°C

Rys.8. Mikrostruktura skanningowa stopu 2 starzonego 44 godzin w 200°C

In Fig.6 these precipitates can be seen, but in smaller amount than in Fig.7 after 16 hours and 44 hours of ageing, respectively. Hardness maximum is observed in alloy 2 above 10 hour ageing and after above 30 hours decreases due to the formation of rather large, stable $Al_2Cu$ phase particles, which can be observed in Fig. 8. The results given above and the dependence of the lattice parameter changes vs. ageing time corresponds well with the results of the EDS analysis. In the as quenched state the alloys 1 and 2 contain slightly under 2%Cu and 4% copper, respectively. After 44 hours of ageing only about 0.1% Cu and 0.0% Cu in the alloy 1 and 2, respectively was found.

## 4. Conclusions

1. Large precipitates of the stable $Al_2Cu$ phase in the directionally solidified alloys were found.
2. The composition of the quenched and aged aluminium matrix changes with the ageing time due to the precipitation of the Θ' phase
3. The increase of hardness corresponds very well to the amount of the Θ' precipitates.Despite the reports of [10] there is no evidence of ternary silicon phases
4. The decrease of hardness is bound with the Θ' phase growth and the transition of the $Al_2Cu$ phase to the large, stable precipitates

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