Z. PĘDZICH, K. GAWDZIŃSKA, E. PAMUŁA

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FLAWS DESCRIPTION IN AlSi11 ALLOY-BASED COMPOSITE FIBRES REINFORCED CASTS

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In the paper the application of different methods: profilography, atomic force microscopy, scanning electron microscopy and mercury porosimetry for description of flaws in composites based on AlSi11 (AK11) alloy with fibrous reinforcement is presented.

Key words: *composites, flaws, SEM, AFM, profilometry, mercury porosimetry*

Opis nedostataka u odljevcima ojačanim kompozitnim vlaknima na bazi legure AlSi11. U članku je predstavljena primjena različitih metoda: profilografije, mikroskopija atomske sile, skening elektronske mikroskopije i živine porozimetrije za opis nedostataka u kompozitima na bazi legure AlSi11 (AK11) ojačanjem na bazi vlakana.

Ključne riječi: *kompoziti, nedostaci, SEM, AFM, profilometrija, živina porozimetrija*

INTRODUCTION

Composites based on aluminium alloys matrix belong to a group of very promising materials. Thanks to a skilful selection of reinforcement and matrix phases it is possible to obtain a vide variety of composite materials having favourable mechanical and thermal properties [1, 2].

The manufacturing of composite casts differs distinctively from traditional casting. The presence of reinforcement phase may cause creation of many types of flaws not existing in the "pure" matrix material. This is especially evident in the case of composites produced by saturating of the reinforcement structure with liquid metal under pressure.

Flaws in composite materials are often in form of isolated voids (pores), unconnected with themselves and with the external atmosphere. The investigation of such type of defects is quite difficult. The ultrasonic defectoscopy does not always identify the voids in an explicit way [3]. Other more sophisticated methods, for instance nuclear magnetic resonance, are rather expensive [4]. Moreover, those methods have the resolution of the size of several micrometers. This could be not enough to identify all possible flaws in composite casts, where a significant number of flaws could be smaller than one micrometer.

The aim of this study was to check the use of different analytical techniques for the characterization of isolated voids in fibre/alloy composites.

Z. Pędzich, E. Pamuła, Faculty of Materials Science and Ceramics AGH - University of Science and Technology, Cracow, Poland, K. Gawdzińska, Institute of Basic Technical Sciences Maritime University of Szczecin, Szczecin, Poland

STUDY DESCRIPTION

Three types of materials were examined:

- a cast of AlSi11 alloy (AK11),
- a composite of AK11 matrix and short non-oriented aluminasilicate fibres (Sibral) (AK11/AlSi),
- a composite of AK11 matrix and short non-oriented graphite fibres (AK11/C).

Samples were made by saturating short fibre reinforcing profiles with liquid alloy (AlSi11) under the pressure of 15 MPa for 300 seconds considering all requirements of the technology of casts production [5]. The investigations were performed on polished cross-sections of casts. Samples were cut off from casts as cuboids with dimensions of 5 mm × 5 mm × 12 mm. The external surface was polished with diamond paste, finally with the 1 micrometer one.

For investigations different methods were applied:

- the profilography (Hommelwerke T500 equipment),
- the atomic force microscopy (Explorer ThermoMicroscopes equipment with Vecco SPMLab 5.01 software),
- the mercury porosimetry (Quantachrome Poremaster 60 equipment).

The investigations were complemented with standard scanning electron microscopy (Jeol 5400 and Jeol JSM 6100 equipments).

RESULTS OF INVESTIGATIONS

Scanning electron microscopy

The microscopic defects visible on the polished cross-section are shown in Figures 1. - 5.

In the case of the matrix material using SEM only voids in the order of few micrometers could be detected (Figure 1.).

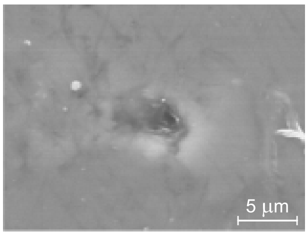

Figure 1. **A typical SEM image of flaw in AK11 alloy cast**
Slika 1. **Tipična SEM snimka nedostatka u odljevku od legure**

The microscopic observation of composite cross-sections show that the voids have a complex morphology.

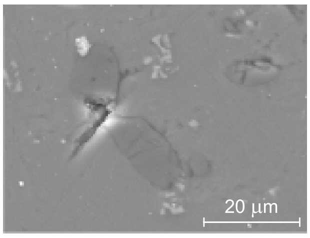

Figure 2. **A typical microstructure of AK11/AlSi composite cast**
Slika 2. **Tipična mikrostruktura odljevka od kompozita AK11/AlSi**

Figures 2. and 3. illustrate typical flaws detected in AK11/AlSi casts. The flaws had dimensions mainly in the order of magnitude of 10 micrometers, a size related with the diameter of fibres. Also smaller cracks having the size in the order of 1 micrometer were detected.

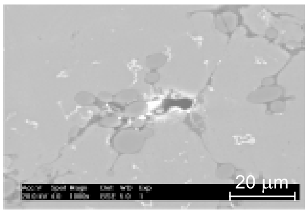

Figure 3. **A typical microstructure of AK11/AlSi composite cast**
Slika 3. **Tipična mikrostruktura odljevka od kompozita AK11/AlSi**

A similar size range of voids was detected in AK11/C graphite composite, but as shown in Figures 4. and 5., the morphology of the voids was distinctly different.

Microscopic observations showed qualitatively that voids present in the casts had quite a wide range of size distribution. The microscopy could not provide detailed information about void size and volume distribution due to a wide range of changes of these parameters. The image analysis of objects differing in size for two or even three orders of magnitude is very difficult and labour-consuming. A systematic "scanning" of sample surface by high magnification showed that such analysis was unpractical.

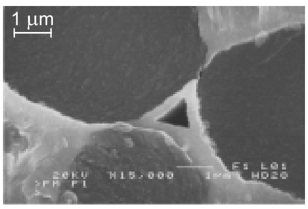

Figure 4. **A typical microstructure of AK11/C composite cast**
Slika 4. **Tipična mikrostruktura odljevka od kompozita AK11/C**

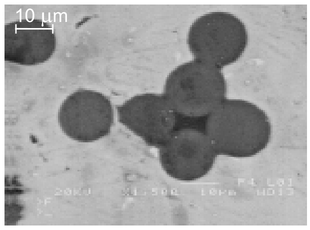

Figure 5. **A typical microstructure of AK11/C composite cast**
Slika 5. **Tipična mikrostruktura odljevka od kompozita AK11/C**

Profilography

Profilography is the simplest method for pro-

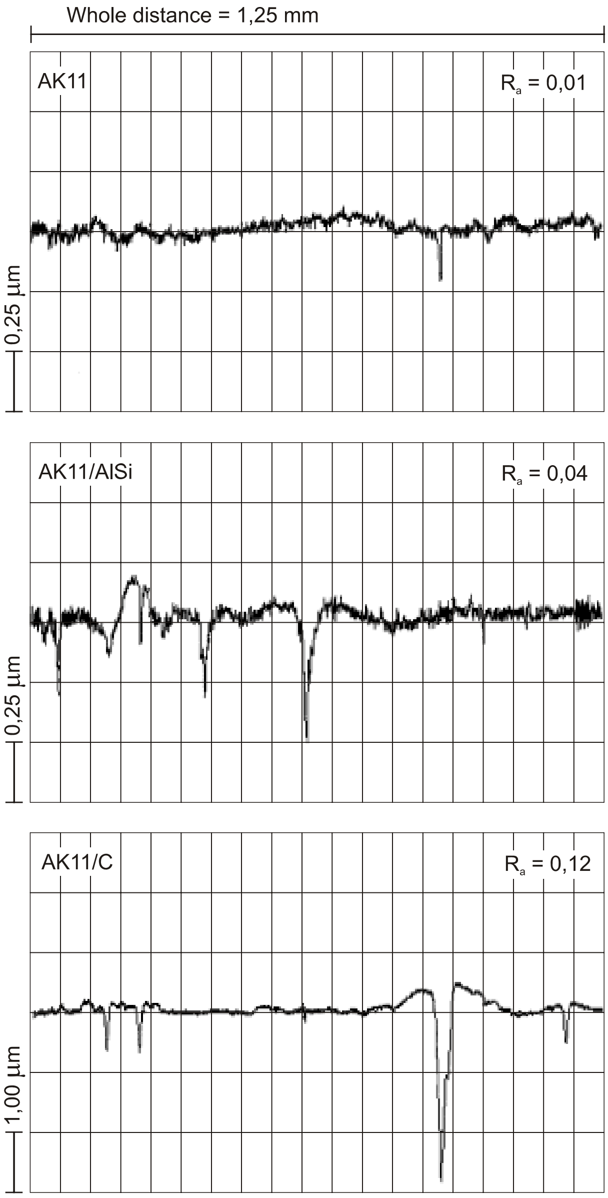

Figure 6. **Typical profiles of polished cross-sections: AK11 alloy, AK11/AlSi and AK11/C composites**
Slika 6. **Tipični profili poliranih poprečnih presjeka: legura AK11, kompozita AK11/AlSi i AK11/C**

viding statistical information about the size of the flaws. The signal collected on the relatively long distance (1250 micrometers) allowed to register voids present on the cross-section surface and to estimate their depth. Determination of flaw depth was limited by geometry of measuring system.

Figure 6. shows typical profilographs for all investigated materials. The results correspond to SEM observation.

The surface roughness of investigated materials was different. The differentiation registered for investigated samples could be described in terms of the R_a (roughness average - the arithmetic average of the absolute values of the measured profile height deviations) difference. The resolution of profilography is limited but performed investigation allowed to establish that the voids in AK11/C composite were greater than those registered for AK11/AlSi. The information from profile measurements could be collected and elaborated fast for relatively great area of the sample but the results are very sensitive to the surface state. Moreover, the measurements require very careful and repeatable preparation of the samples. The results of profilography measurements did not distinguish the individual voids and have only a comparative value.

Atomic force microscopy

The atomic force microscopy (AFM) used in this study is the more accurate profilography operating in three dimensions. The precision and resolution of measurements is much higher but determination of the flaws "depth" is still limited by the geometry of the measuring system (tip).

Figures 7. and 8. show selected AFM pictures of composite casts. The surface image in AFM is similar to that

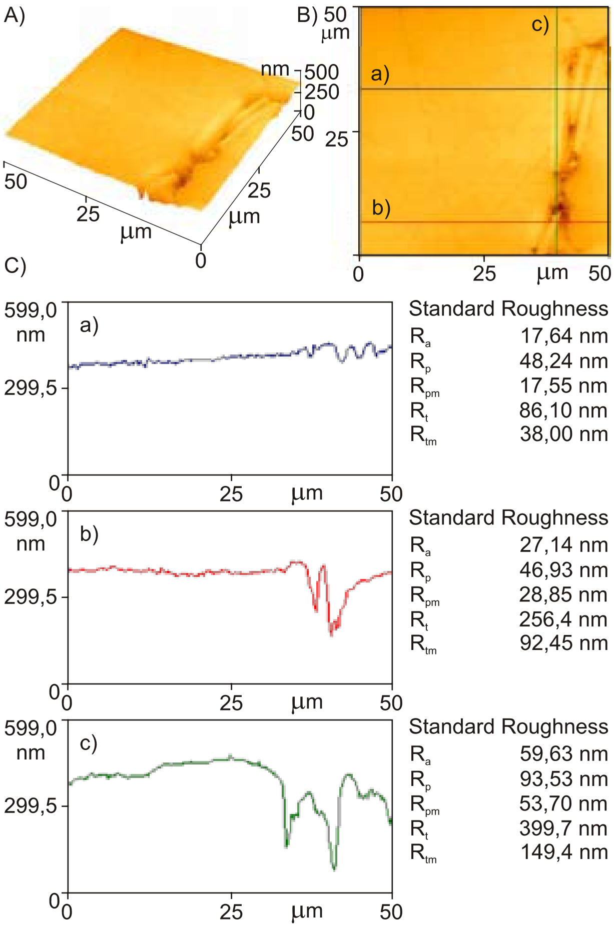

Figure 7. **Results of AFM investigation of selected area of AK11/AlSi composite: A) three dimensional visualization of the scanned area; B) two dimensional visualization of the scanned area with marked profile analyze line; C) profiles of selected lines**

Slika 7. **Rezultati AFM ispitivanja odabranog područja kompozita AK11/AlSi: A) trodimenzionalna vizualizacija skeniranog područja; B) dvodimenzionalna vizualizacija skeniranog područja s označenom linijom profilne analize; C) profili odabranih linija**

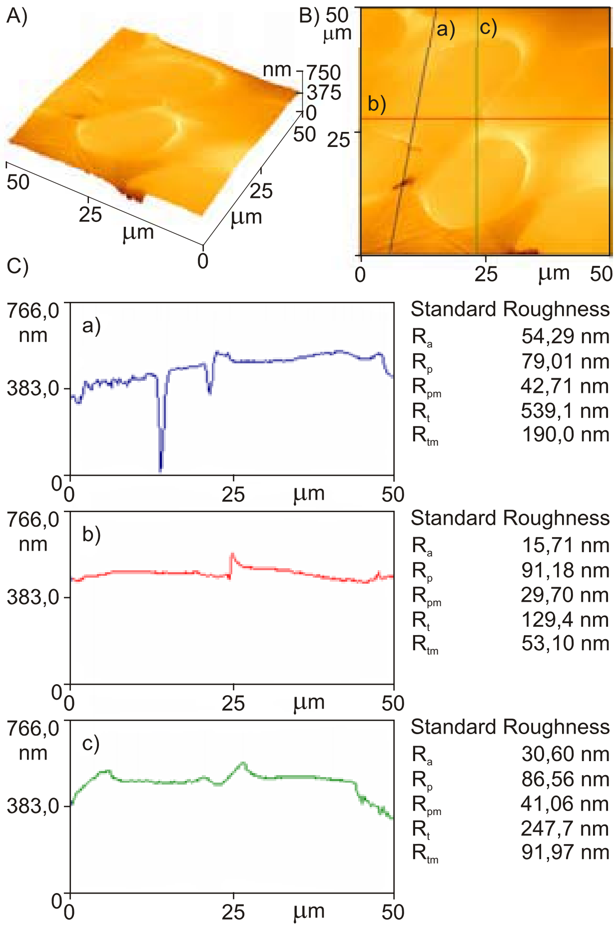

Figure 8. **Results of AFM investigation of selected area of AK11/C composite: A) three dimensional visualization of the scanned area; B) two dimensional visualization of the scanned area with marked profile analyze line; C) profiles of selected lines**

Slika 8. **Rezultati AFM ispitivanja odabranog područja kompozita AK11/C: A) trodimenzionalna vizualizacija skeniranog područja; B) dvodimenzionalna vizualizacija skeniranog područja s označenom linijom profilne analize; C) profili odabranih linija** attained with SEM. The advantage of AFM is the possibility of advanced data treatment. The data collected by AFM could be analyzed in detail.

The AFM investigations were performed for different scan areas of 20 μm × 20 μm, 50 μm × 50 μm and 100 μm × 100 μm. The comparison of results allowed to conclude that for studied materials the observations at 50 μm × 50 μm scan areas were the most useful. This was related to the fact that the majority of voids had dimensions close to the size of single fibers or their agglomerates.

By a decreased of scan area the great defect became "invisible" for this method.

The AFM software allowed to treat obtained profiles statistically for the whole scan area and to analyze them in selected regions or lines (Figures 7B and 8B).

The software allowed also to determine many detailed parameters describing the sample surface (Figures 7C and 8C). It is possible to determine with accuracy many profilographic parameters (e.g. R_p - maximal height of the profile above the mean line, R_t - maximum peak-to-valley height for the profiles or their mean values - R_{pm}, R_{tm}). However, these data could not be used directly in determining voids volume and distribution.

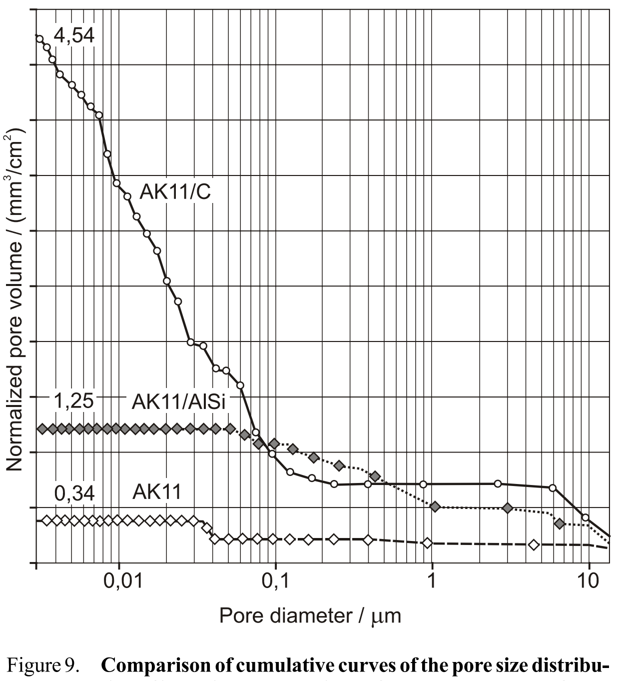

Figure 9. **Comparison of cumulative curves of the pore size distribution of investigated materials referred to samples surface**
Slika 9. **Usporedba kumulativnih krivulja distribucije veličine pora ispitivanih materijala obzirom na površinu uzoraka**

It is worth to notice, that measured profile "depth" in standard profilography and AFM is different (compare Figures 6, 7C and 8C). It is due to the geometry of measuring system and it limits the credibility of voids depth determination (results achieved with both methods are different).

Mercury porosimetry

The mercury porosimetry is not a conventional method for the determination of voids in composite casts practically devoid of open porosity.

Assuming that the sample cross-section is representative for its whole volume [6], it is possible to deduce the void size distribution in the investigated material. However, it is not allowed to relate the data obtained by porosimetry to the fundamental stereological dependence $V_V = A_A$.

The method of collection of the measured signal in mercury porosimetry does not limit the depth of mercury penetration. As a result of porosimetry measurement and data recalculation, it is possible to obtain an overall information about voids size (diameter) and size distribution. It allows to describe the full volume of pores in contact with surface cross-section.

As it was shown in Figure 2. - 5., the voids present in composites with fiber reinforcement could be no isotropically distributed. The pores were often parallel to fibers or were connected with fibers agglomerates (Figures 4. and 5.). The image analysis can describe such "channels" as two-dimensional object. The porosimerty does not probe the shape of such voids but it can precisely establish their volume and compare it with other voids present in the sample.

Figure 9. shows the voids size distribution in all investigated materials. Pores bigger than 15 μm were not detected. In all investigated materials there was some number of pores in the range of few microns and a much greater volume of pores below one micrometer. The mercury porosimetry gave information on the voids volume (Figure 9.) and their size

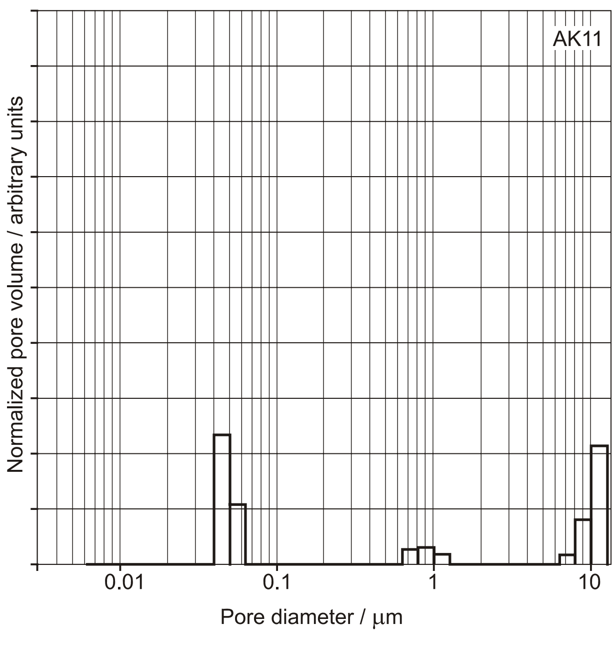

Figure 10. **Histogram of pore size distribution in AK11 cast**
Slika 10. **Histogram distribucije veličine pora u odljevku Ak11**

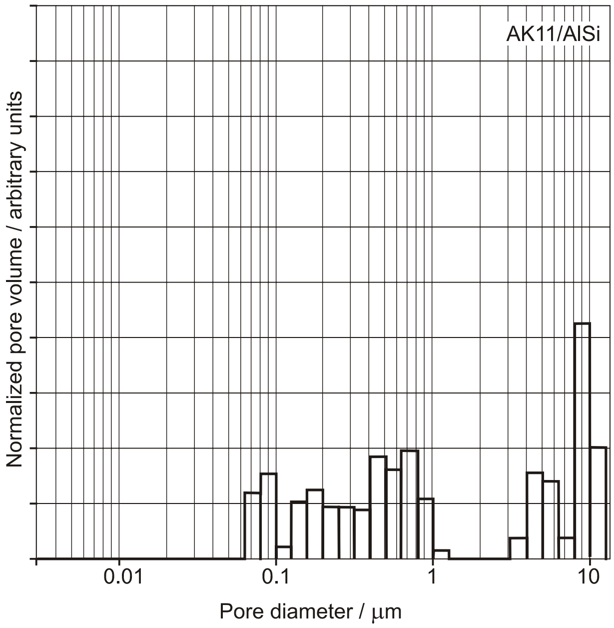

Figure 11. **Histogram of pore size distribution in AK11/AlSi composite**
Slika 11. **Histogram distribucije veličine pora u kompozitu AK11/AlSi**

distribution (Figures 10. - 12.). These data were achieved relatively easily from the whole sample surface.

The porosimetry confirmed the conclusion obvious from SEM observations and profilography measurements, that in AK11 alloy cast (matrix material) the number of voids was small (Figures 9. and 10.). Much more interesting is the observation that the number of voids in AK11/AlSi and AK11/C composites was significantly different. The volume of voids in composite reinforced with graphite fibres was more than three times bigger (Figures 9.) and also the voids size distributions were different. In AK11/AlSi composite pores in the range of 0,1 - 1,0 μm dominated (Figure 11.), while in AK11/C material the dominating pore fraction was much smaller and enclosed in the range of 0,3 - 0,004 μm (Figure 12.).

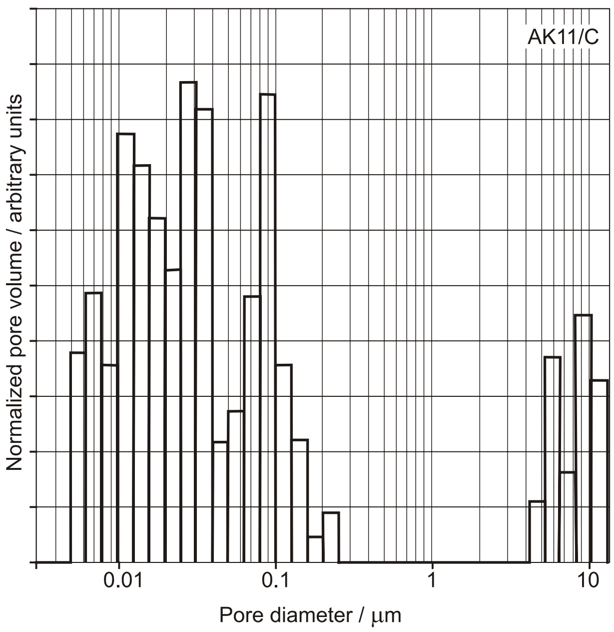

Figure 12. **Histogram of pore size distribution in AK11/C composite**
Slika 12. **Histogram distribucije veličine pora u kompozitu AK11/C**

CONCLUSIONS

Different analytical methods were used for the characterization of voids in composite casts. The data collected with profilography, AFM, SEM and mercury porosimetry were complementary.

The results obtained by profilography were the most "raw". The information achieved was averaged and it was not possible to distinguish individual voids. The results had a mainly comparative value.

The AFM analysis was more precise due to higher resolution and the possibility of numerical treatment of acquired data. Nevertheless, the analysis of individual voids and their classification was time and work consuming.

Both mentioned methods were limited by a specific way of data collection (tip interaction with sample surface) which did not allow to penetrate the volume of each void.

The application of the mercury porosimetry to voids characterization enabled the accurate determination of their volume and size distribution. This method gave a fast information about whole pore population without necessity (and without possibility) of individual objects analysis. It is worth to notice, that with porosimetry it is possible to detect pores in the range between several nanometers and hundreds of microns. The results obtained by porosimetry confirmed microscopic observations and added same statistical generalization.

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