ARCHIVES

of

FOUNDRY ENGINEERING

Published quarterly as the organ of the Foundry Commission of the Polish Academy of Sciences

ISSN (1897-3310)
Volume 7
Issue 2/2007
95 – 100

20/2

Repeatable and reproducible methodology of the AZ91 alloy structure evaluation

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Received 08.02.2007; Approved for print on: 12.03.2007

Abstract

The objective of the research conducted was to develop a comprehensive procedure for a quantitative evaluation of magnesium casting alloy's (AZ91) microstructure. The procedure encompasses: a methodology of metallographic specimens preparation, microstructure detection methods, selection of image acquisition methods, image analysis methodology and adjustment of morphological and stereological parameters to the quantitative description of volume fraction of phases and structural components of the AZ91.

Keywords: quantitative metallography, materials, metallic alloys, magnesium alloys, AZ91

1. Introduction

The increasing requirements set upon cast products come down to obtaining an appropriate macro- and microstructure of an alloy. The macro- and microscopic structure evaluation is more and more frequently introduced in the quality control of casts, equally with a chemical analysis and examination of mechanical properties [1]. So far, the casts' macro- and microstructure has been evaluated mainly based on the conventional, qualitative metallographic studies [2-6]. The results of such studies have been documented by a set of photographs presenting the cast's structure in randomly selected regions, in the casts' walls of varied thickness. They prove high diversity of the phase composition and morphology in casts. Nevertheless, based on those studies, it is difficult to equivocally evaluate the degree of diversity and non-uniformity of the macro- and microstructure of a cast in regions solidifying at a different rate [7]. Consequently, the importance of structure's quantitative evaluation by quantitative metallography methods for casts in the as-cast condition and after heat treatment is growing. This is because only at a quantitative evaluation of the macro- and microstructure, determined using the number and unit of measure, some reliable and strict dependencies can be found between the technological process parameters of the cast formation and its structure and properties. The using of mathematical statistics methods when discovering such dependencies seems to be indispensable due to the random variability of cast structures [1].

2. Factors influencing the results of qualitative and quantitative evaluation of cast structures

The contemporary knowledge of physical metallurgy and the modern quantitative metallography methods enable a correct evaluation of the dependencies between the primary and secondary structures of casts, as well as the selection of structural features having significant importance for their technological and mechanical properties, as well as durability and reliability of products made of the casts. These features include:

- the volume fraction, number, shape, size and distribution method of gaseous and shrinkage pores as well as non-metallic inclusions;
- the volume fraction of phases and structural components in two-phase and multiphase alloys;
- the shape and size of grain in granular structures;
- the volume fraction, number, shape, size and distribution method of dispersion phase;
- the non-uniformity of phase distribution and structural anisotropy [8].

A proper structure preparation to an automatic image analysis is the condition for correct identification and measurement of objects. The results of measurements in the automatic image analysis are encumbered with errors caused by different factors, among which the sample's representativeness and the metallographic specimen preparation are of great importance.

In the process of structure preparation to an automatic image analysis, the sampling of metallographic specimens should take into account the research objectives. The sampled specimen should be statistically representative. Particular attention should be paid to sampling and location of the polished section when the alloy's structure is inhomogeneous. For instance, when performing a quantitative evaluation of casts with inhomogeneous structure and axial symmetry, transverse microsections should be made. If inhomogeneity is also present at the cast's height, a few microsections need to be taken in order to average the results. In many cases, it is also necessary to prepare longitudinal microsections to obtain correct research results [1].

The fulfilment of the above-mentioned requirements by a metallographic specimen is the condition of correct identification of the object and taking measurements during an automatic image analysis. Any deviations from the microsection's flatness, its rounded edges, roughness or relief, lead inevitably to inhomogeneity of its lighting, which disrupts the course of an automatic analysis. During an automatic analysis, any scratches, pits and cracks, as well as foreign particles, can be detected to the same extent as the structural objects being of interest to the operator, e.g. the lattice of grain boundaries can be cut by scratches on a metallographic specimen.

In the process of metallographic specimens' etching for an automatic image analysis, the technique, reagent and etching conditions should be selected so as to detect only those structural elements that will be taken into consideration in the structure's quantitative analysis.

Well-etched metallographic specimens should meet the following requirements [8, 9]:

- the etched elements of the same phase should exhibit identical contrast, with local overetching being unacceptable;
- the structural components and grains' boundaries must be continuous and clearly visible;
- different microstructure components should exhibit a varied degree of greyness.

There are two major groups of factors influencing the objectivity and accuracy of the macro- and microstructure evaluation of as-cast magnesium alloys; the factors are connected with [10]:

- the research material's internal variability,
- the research methodology properties (samples' preparation, capacities of the research equipment, operator's knowledge and experience).

The research material's inherent variability affects both the sampling method and the number of microregions analyzed in each sample. Based on the results of macro- and microscopic qualitative metallographic studies, the selection of regions from which specimens are cut out and of the microregions for an analysis on such specimens is made so that their quantitative analysis would enable presenting the characteristic features of the objects analyzed throughout their volume [9]. The factors influencing the objectivity and accuracy of material structures' quantitative evaluation results are presented in Fig. 1.

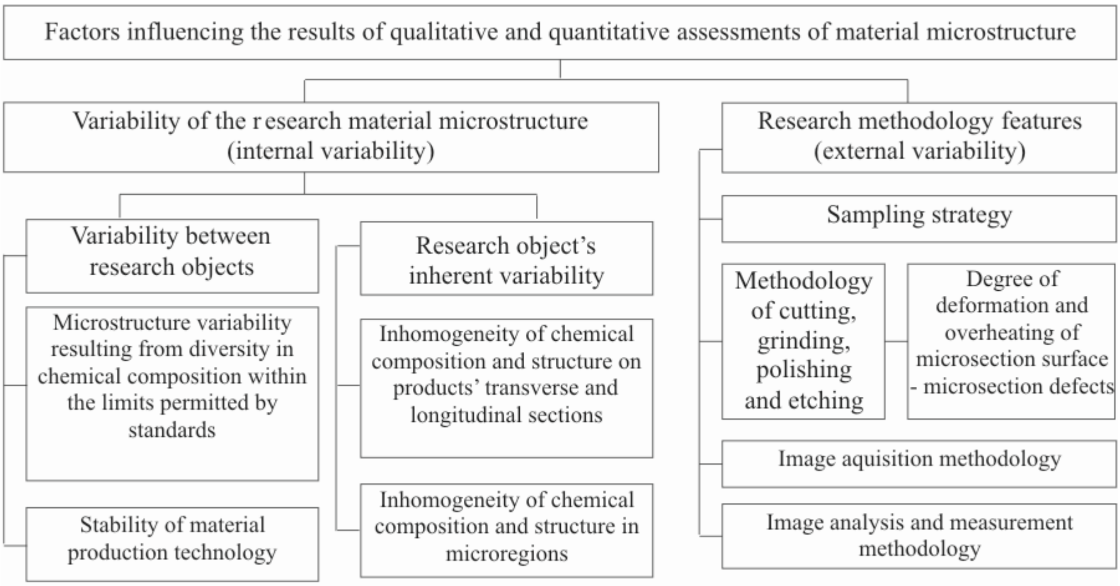


Fig. 1. Factors influencing the results of a qualitative and quantitative evaluation of material structures [8]

As the precision adopted for measurements increases, the labour-intensity and cost of research grow as well. Performing both too little and too many measurements is a methodological error.
The measurements made should ensure obtaining repeatable and reproducible results, which, every single time, requires verification with the use of appropriate statistical tests.

3. Research material

The aim of the study was to develop a comprehensive procedure for a qualitative and quantitative evaluation of the phase composition and morphology in the AZ91 alloy's structure in the as-cast state and after heat treatment. Two fragments of experimental oil pans cast from the AZ91 alloy in ZM "WSK Rzeszów" were used for the research. This area ensures observation of the macro- and microstructure on the surface of flat cross-section of walls characterized by varied thickness [11-13].

4. Research methodology and results

Comprehensive procedures have been developed for a qualitative and quantitative evaluation of the AZ91 alloy's macro- and microstructure in the as-cast state and after heat treatment.

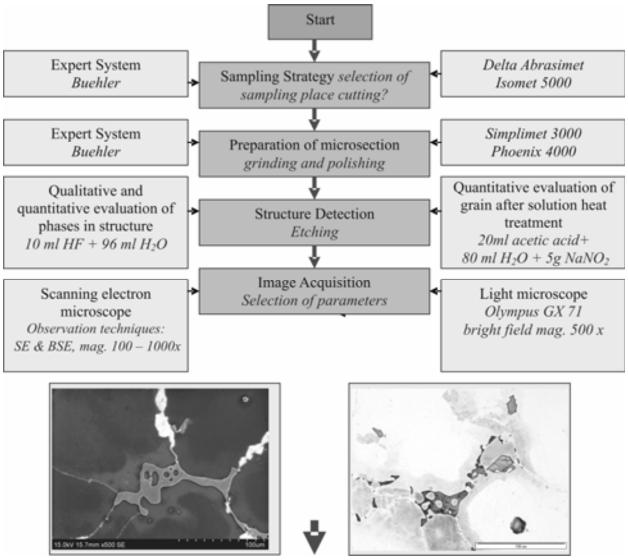
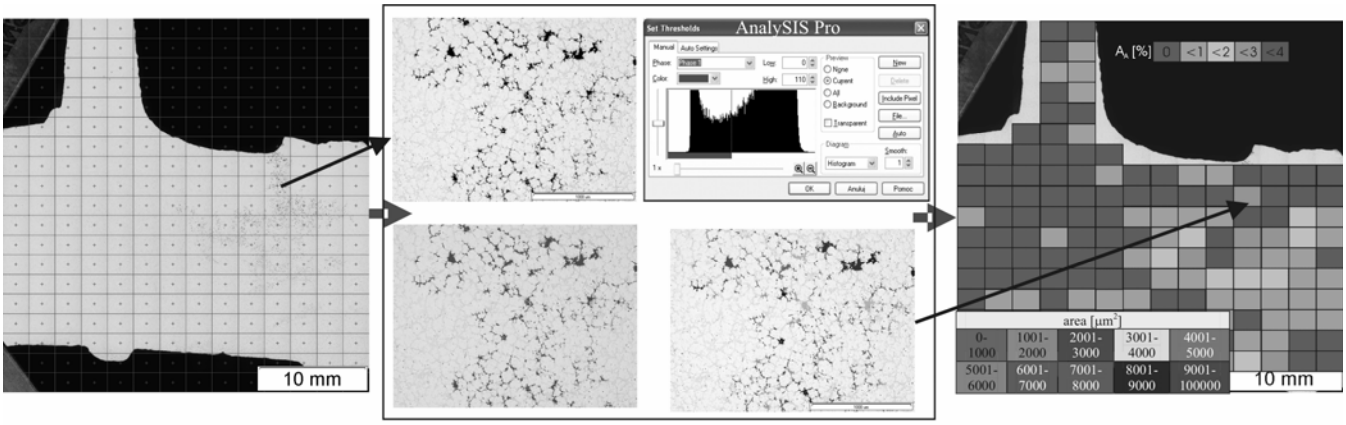


Fig. 2. Chart of the AZ91 alloy structure evaluation procedure, part I: Structure detection [14, 15]



Parametr	pores	
	mean	v [%]
A_A [%]	0,20	248
N_A [1/mm²]	7,43	226
$\overline{A}$ [μm²]	431	122
$\overline{P}$ [μm]	79,9	89,7
$\overline{d}$ [μm]	38,5	55,5
$\overline{\hat{\imath}} = 4\pi A/P^2$	1,97	31,2
$\overline{\delta} = d_{max}/d_{min}$	0,838	27,7

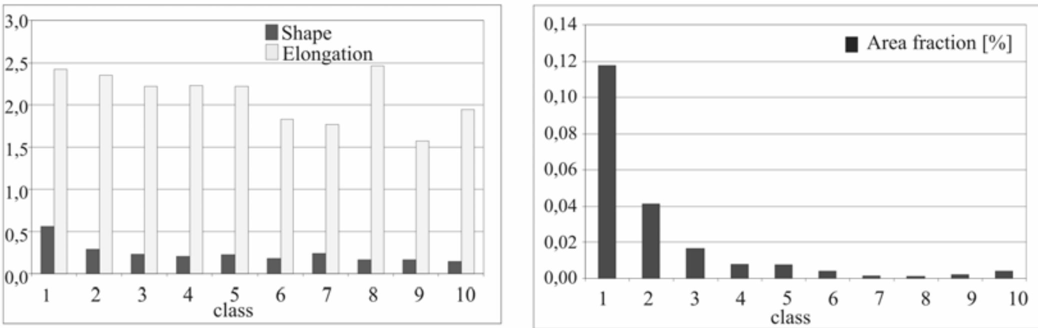


Fig. 3. Chart of the AZ91 alloy structure evaluation procedure, part II: Quantitative description of porosity

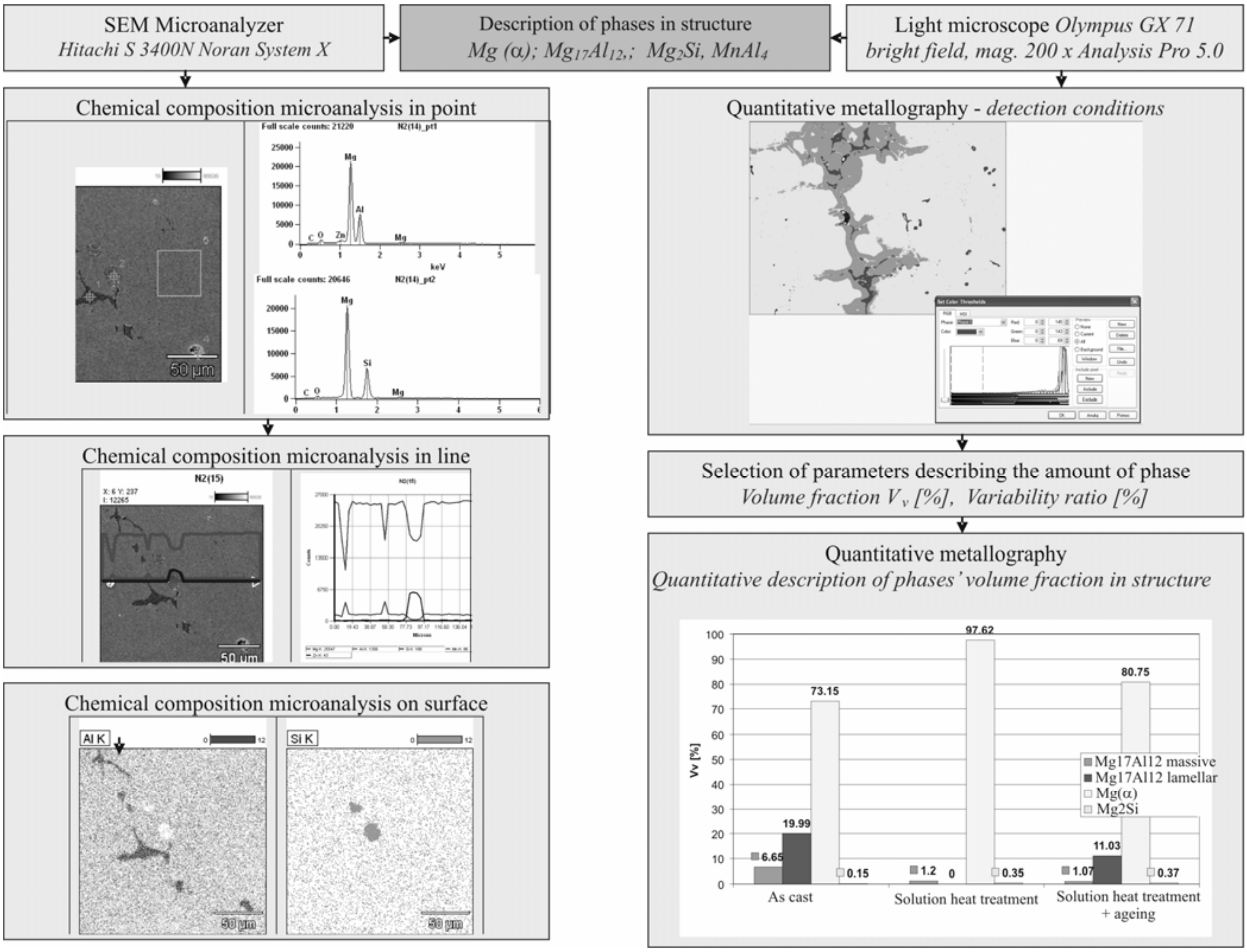


Fig. 4 Chart of the AZ91 alloy structure evaluation procedure, part III: Qualitative and quantitative description of phases

Methodological conditions were determined for obtaining accurate and repeatable results of a quantitative evaluation of the AZ91 alloy's macro- and microstructure in the as-cast state and after solution heat treatment.

Comprehensive procedures have been developed for a qualitative and quantitative evaluation of the AZ91 alloy's macro- and microstructure in the as-cast state and after heat treatment. Methodological conditions were determined for obtaining accurate and repeatable results of a quantitative evaluation of the AZ91 alloy's macro- and microstructure in the as-cast state and after solution heat treatment.

From the point of view of quality control of products cast from the AZ91 alloy, the developed procedure contains the most important elements of metallographic studies. They encompass: the methodology of structure detection (Fig. 2), quantitative description of porosity (Fig. 3), quantitative and qualitative description of phases present in the structure (Fig. 4) and quantitative description of the grain size in the MG-α phase after solution heat treatment (Fig. 5).

New elements in this procedure are, inter alia:

- selective phase etching for the sake of quantitative measurement in the initial state, after solution heat treatment and after full heat treatment (Fig. 2),
- a quantitative evaluation of porosity, performed throughout the specimen's cross-section and not on single measurement fields; determination of the location of the worst field based on the results' matrix from the whole element's cross-section, and not on a subjective visual evaluation; structural maps constituting a clear interpretation of research results; and differentiating between gaseous pores and shrinkage pores based on shape indicators (Fig. 3),

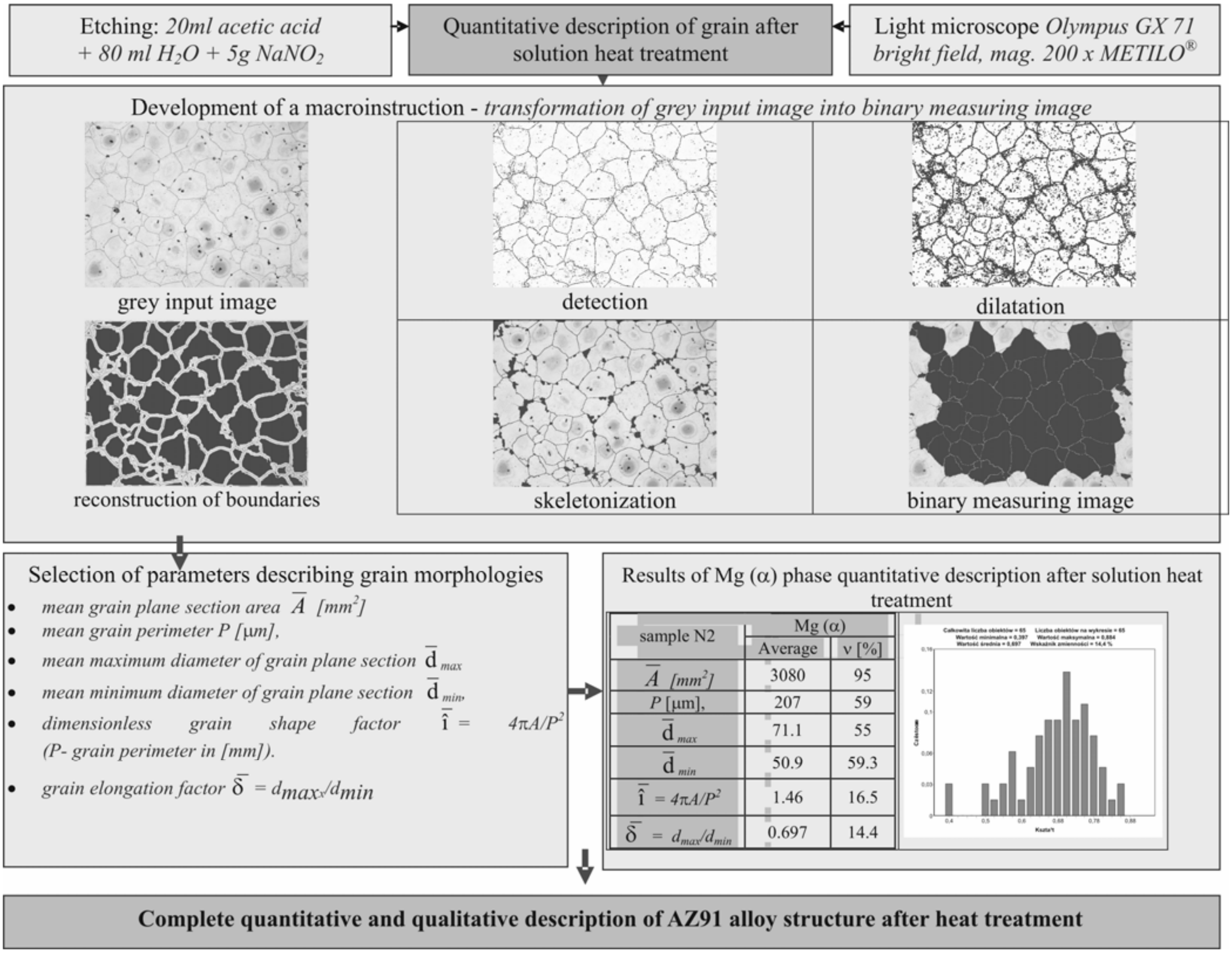


Fig. 5. Chart of the AZ91 alloy structure evaluation procedure, part IV: Quantitative description of Mg(α) phase grain after solution heat treatment

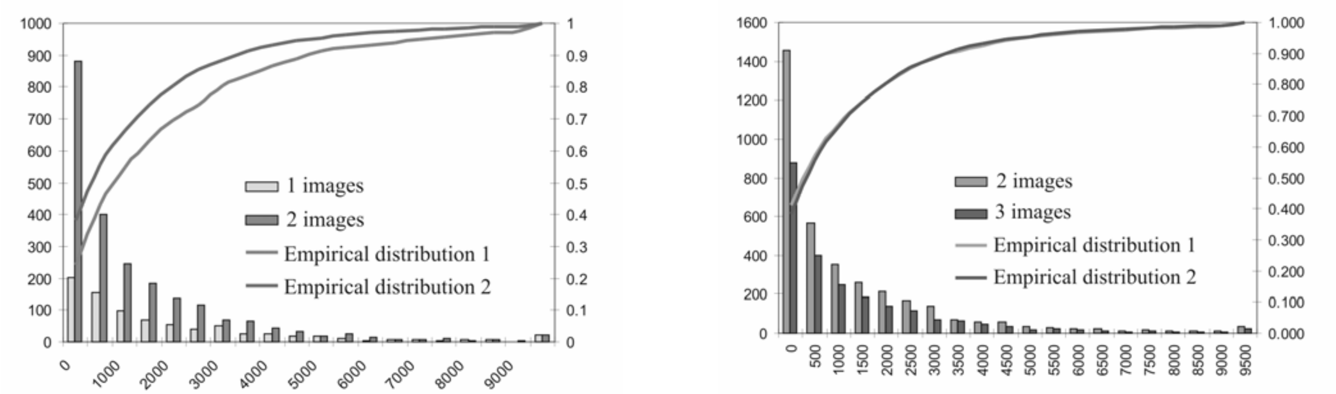


Fig. 6. Distributions of frequency as a function of the grain plane section area, determined for one and two and three measurement fields, and the corresponding empirical distribution functions for the investigated specimen

- microanalysis of the chemical composition in a point, line and on the surface; automatic detection of all phases in the structure (Fig. 4),
- measurement of the Mg-□phase grain size after solution heat treatment, being an important cast quality criterion (Fig. 5),
- application of non-parametric tests of significance (including the Kolmogorow-Smirnow test) for an evaluation of the necessary number of measurements as well as repetitiveness and reproducibility of research results (Fig. 6).

5. Final remarks

The procedures developed may become new and original tools in the studies of dependencies in the cause-effect chain: chemical composition – technology – AZ91 alloy's structure and properties.

Acknowledgment

The work was supported by the Ministry of Science and Higher Education under the research project No 6 ZR7 2005 C/06609.

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