X-ray computed tomography of cast metal matrix composites

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The development of new cast metal matrix composites and technological processes for their production are inextricably linked with the need to improve the methods of quantitative non-destructive testing. In this work, the features of the spatial structure of cast in-situ metal matrix composites (on example of the Al – Mg_2Si system) were studied using *X*-ray computed microtomography. The parameters of tomographic scanning were optimized to identify structural components with close ranges of radioparency. The rational selection of the scanning parameters of the samples made it possible, during the subsequent computer processing of the slice sets, to clearly identify the structural constituents of the metal matrix composites depending on the gray levels in their comparison with the characteristic morphology of the observed constituents (primary Mg_2Si crystals, pseudobinary eutectic, compounds of impurity elements). The total volume of porosity in the studied samples of Al + 15 wt.% Mg_2Si composites after melt thermal-rate treatment at 900 °C did not exceed 0.05 mm^3, which corresponds to a porosity content in the sample of 0.13 vol.%. The presented results show the significant potential of tomography in the study of the structural and morphological characteristics of composite materials and internal defects in cast products made from them.

Key words: cast metal matrix composites, *X*-ray computed tomography, selection of scanning parameters, identification of structural constituents, internal defects, quantitative analysis.

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Introduction

The development and industrial implementation of new advanced materials for structural and functional purposes require a fundamental understanding of the processes of formation of their structure and its influence on the properties and characteristics of the resulting products. The appearance in recent decades of revolutionary new classes of heterophase materials, such as cast metal matrix composites, requires a change in the usual approaches to studying their structure in comparison with traditional materials [1–3]. First of all, this is due to the fact that the most important information in creating such materials and the manufacturing of products from them is data on the spatial structure of the material, the nature of the change in the structure throughout the entire volume of the product under various extreme conditions, and so forth.

The key feature of composite materials is the possibility of designing material properties at the stage of its creation [4]. At the same time, due to the inhomogeneous nature of such materials, certain problems arise in their preparation and processing, leading to defects specific to these materials, and most of these defects can be hidden. In particular, the considered defects may include delamination, clustering of reinforcing particles, various voids and inhomogeneities, cracks and other damage in the matrix material, and much more [5–7]. In this regard, the study of the structure and, in particular, the defect detection in such materials and products from them presents significant difficulties.

Therefore, the development of new composite materials and technological processes for their production are inextricably linked with the need to improve the methods of quantitative non-destructive testing. Using traditional qualitative techniques (ultrasonic inspection, *X*-ray radiography, visual control) in most cases is not enough for a complete characterization of the structural features and internal defects of composites [8, 9]. Quantitative methods of optical and electron microscopy are limited by the ability to study only two-dimensional images, except for visualization of the three-dimensional relief of the sample surface or transmission of thin foils. Many parameters, including spatial morphology, distribution, interconnectedness of defects and structural components, and much more, cannot be determined using these methods [10, 11]. At the same time, a reliable assessment of the level of technology used, prediction of the mechanical and operational properties of the materials being developed, and modeling of the behavior of materials under various conditions is impossible without accurate qualitative and quantitative data on the bulk structure of the material [12]. These limitations contributed to the emergence and development of new methods and tools that allow getting

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Table 1
Chemical composition of experimental materials

Composite	Al	Mg	Si	Fe	Mn	Cu	Zn	Ni	Ti
Al – 15Mg_2Si	Bal.	13.627	7.952	1.019	0.004	0.007	0.002	0.002	0.0006
Al – 20Mg_2Si	Bal.	9.695	5.943	0.969	0.009	0.006	0.003	0.002	0.0009

three-dimensional images of the internal structure of materials and products.

Computed tomography is a method of restoring the internal structure of an object from digital images of an object taken from different points using mathematical methods and algorithms [13]. *X*-ray computed tomography has a significant potential for obtaining three-dimensional quantitative information necessary for the creation of new functional materials and technologies for their production [14]. Tomographic techniques make it possible to accurately determine the size and location of voids, foreign inclusions, to identify areas with reduced density, cracks, and other discontinuities [15]. The information obtained is very useful in the categorization and evaluation of defects in products made from various functional materials, including cast metal matrix composites. At the same time, in many cases, the structural components of metal matrix composites have a close range of radioparency, which significantly complicates their identification and requires the development and optimization of scanning modes for specific conditions.

The purpose of this work is to study the features of the spatial structure of cast in-situ metal matrix composites (on example of the Al – Mg_2Si system) using *X*-ray computed tomography.

Materials and Methods

Experimental materials were prepared in an alundum crucible in a resistance electric furnace (GRAFICARBO S.R.L., Zorlesco, Italy) by direct melting of pure components: aluminum (99.99% Al), magnesium (99.9% Mg), silicon (96.0% Si). Silicon was added into the aluminum melt at 750 ± 5°C, magnesium at 720 ± 5 °C. After complete dissolution of the charge components, the melt was manually mixed and overheated to a temperature of 900 ± 5 °C. This procedure was followed by isothermal holding for 30 min and subsequent rapid cooling to the pouring temperature to carry out the melt thermal-rate treatment. Using the technique of thermal-rate treatment during the manufacturing of in-situ Al – Mg_2Si composites has a significant positive effect on the structural and morphological characteristics of materials [16]. The temperature regime of the melting process was controlled during the entire experiment with a K-type thermocouple. After holding, the melt was poured at a temperature of 720 ± 5 °C into steel molds with a wall thickness of 30 mm to get ingots with a diameter of 5 mm and a length of 50 mm. A more detailed description of the technological features of the manufacturing Al –Mg_2Si composites was reported earlier [17, 18]. The chemical composition of the samples was determined on an *X*-ray fluorescence spectrometer ARL ADVANT'X (Thermo Fisher Scientific, Waltham, MA, USA) and averaged over at least five measurements for each sample. The results of chemical analysis are shown in **Table 1**.

Table 2
Inspection parameters for cast metal matrix composites of the Al – Mg_2Si system

Parameter	Value
Geometric magnification, times	61.74
Voxel size, μm	3.2
Number of projections, pcs	2400
Scan time, min	160
Accelerating voltage, kV	80
Current strength, μA	170
Exposure / Averaging	1 s / 4 shots per frame

During *X*-ray tomographic studies of cast metal matrix composites, a metrological computed tomography scanner Phoenix V|tome|x m300 (GE Measurement & Control GmbH, Germany) with two sources of ionizing radiation was used to perform non-destructive testing, research and metrological measurements with an accuracy of linear measurements up to 4 μm + L (mm) / 100. The inspection was carried out using a nanofocus radiation source with a maximum accelerating voltage of 180 kV. The inspection parameters were worked out by the exhaustive search method in such a way as to ensure the identification of not only structural defects but also the identification of various structural components. Samples of compositions Al + 15 wt.% Mg_2Si and Al + 20 wt.% Mg_2Si were studied. To optimize the scanning modes, a composite material of the composition Al + 20 wt.% Mg_2Si was used due to the rather large number of primary particles Mg_2Si (compared to the composition Al + 15 wt.% Mg_2Si) with their relatively small average sizes (in contrast to the composition Al + 25 wt.% Mg_2Si, in which the particles are formed as large dendritic complexes [19]). The optimized parameters of the inspection of samples are given in **Table 2**.

During the inspection, the test samples were mounted on an *X*-ray transparent glass stand and fixed in a three-jaw chuck of the manipulator. To reduce the required computational resources for reconstruction and data analysis, the reconstruction area was limited to 450 slices (the height of the area along the sample axis is 1.44 mm, or ~40 mm^3). The Phoenix Datos|x software was used for automated data collection and tomographic reconstruction. Analysis and computer processing of the results were performed using the VGStudio MAX software package (Volume Graphics GmbH, Germany).

Results and Discussion

Samples of metal matrix composites with a diameter of 5 mm with different content of reinforcing phases were subjected to comparative studies. During tomographic investigations, the distribution of *X*-ray density over the sample was recorded. **Fig. 1** shows representative tomographic sections of samples of Al + 20 wt.% Mg_2Si composites in the as-cast state.

Both in 2D sections and in 3D images, structural constituents morphologically corresponding to the primary Mg_2Si phase are clearly revealed. The difference in the density of the observed phases is insignificant, which substantially complicates the qualitative tomographic identification and quantitative determination of the structural and morphological characteristics of the phases in this radioparency range. For the least dense phase, the gray levels are in the range of $13...15 \cdot 10^3$ GV, the main phase is $16...16.5 \cdot 10^3$ GV, the densest phase is $18...19 \cdot 10^3$ GV. These parameters vary from the center of the sample to the periphery. Rational selection of the parameters of tomographic scanning of the samples made it possible to clearly identify the structural constituents of the metal matrix composites depending on the gray levels in their comparison with the characteristic morphology of the observed structural elements during the subsequent computer processing of the tomographic scans. In particular, inclusions of the least dense phase can be identified as primary Mg_2Si crystals, the main phase is a pseudobinary eutectic ($\alpha + Mg_2Si$), while the densest phase can be associated with iron-containing compounds segregated at grain boundaries.

The structural features observed in tomographic sections correlated with the previously obtained results of metallographic studies for these samples in relation to the distribution of reinforcing particles in different parts of the ingot [20]. Overall, the results of *X*-ray tomographic control of the samples obtained under various modes of the melt thermal-rate treatment indicate that the distribution of the Mg_2Si reinforcing phase over the entire volume of the ingot is not quite uniform, revealing zones where the concentration of particles is increased. One of the future tasks in the direction of optimizing the method of tomographic scanning of the materials under study seems to be to increase the scanning resolution (reduce the voxel size to the submicron level), which will make it possible to get precision data on the 3D morphology of individual primary Mg_2Si crystals.

Attention should be paid to the fact that published information regarding *X*-ray tomographic 3D visualization of the microstructure of cast metal matrix composites based on aluminum alloys is still limited. This is largely due to the close values of the *X*-ray attenuation coefficients of the structural constituents of most aluminum matrix composites, and therefore a significant difference in the resulting gray values cannot be observed on *X*-ray scans. It was noted [21] that a substantial increase in phase contrast for the identification of the structural components of aluminum alloys is possible in the methods of synchrotron tomography, characterized by the use of *X*-rays with high spatial coherence. However, such methods are very expensive and are limited by the need for access to a

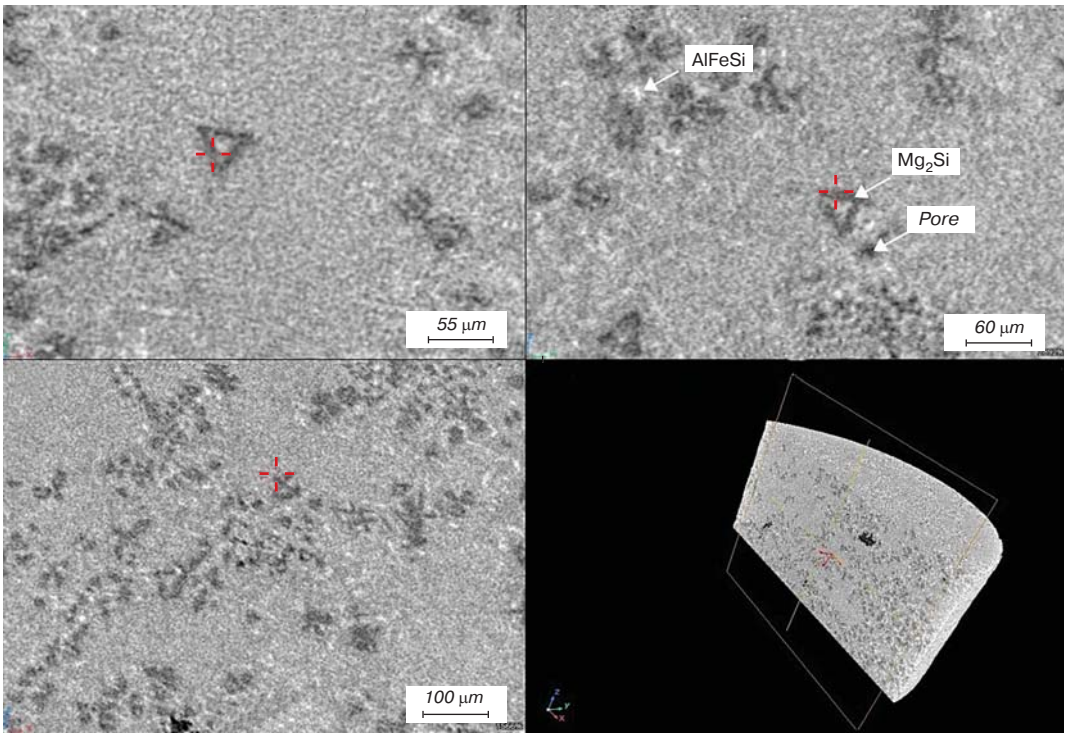

Fig. 1. Representative tomographic sections of samples of Al + 20 wt.% Mg_2Si composites in the as-cast state, obtained during optimization of tomographic scanning modes

synchrotron radiation source. The paper [22] described the application of polychromatic cone-beam phase-contrast tomography for non-destructive evaluation of materials. Using this technique, adequate detectability of the structural constituents of cast aluminum alloys of the Al – Mg – Si system was obtained for samples of small sizes (diameter 0.4 mm), and a voxel size of ~2 μm was achieved. It is important to note that in the present work, in contrast to the results reported in earlier sources, good distinguishability of structural components was achieved on macroscopic samples without any special preparation, while the achieved spatial resolution (voxel 3.2 μm) was close to the maximum reachable values for the set of conditions under consideration. This significantly expands the possibilities for the practical use of the discussed technical solutions in industrial production.

Along with the visualization of structural components, tomographic scanning made it possible to obtain preliminary data on the nature of internal defects in ingots of metal matrix composites of the Al – Mg_2Si system (**Fig. 2**). In particular, shrinkage voids of various morphologies (pores, shrinkage holes, microporosity) are revealed, which can be explained by the volumetric nature of the crystallization of composites and determines the need for further development of technological solutions to improve the quality of castings and ingots from them.

At the same time, according to the data of quantitative analysis using the VGEasyPore module, the total volume of porosity in samples of Al + 15 wt.% Mg_2Si composites after the melt thermal-rate treatment at 900 °C does not exceed 0.05 mm^3, which corresponds to a porosity content in the sample of 0.13 vol.%. We can consider such a total value of porosity as quite a good indicator for composite castings. Thus, the melt thermal-rate treatment of metal matrix composites of the Al – Mg_2Si system, subject to all the necessary requirements for the technological process, is not accompanied by a significant increase in the porosity of cast billets.

Obviously, the potentially achievable degree of increase in the level of properties in composite materials depends on morphological structural factors, such as the volume fraction, size, shape, and spatial distribution of the reinforcing phase [23]. Along with this, structural aspects in composites (size, shape, and spatial distribution of particles) play a decisive role in their deformation behavior. *X*-ray computed tomography makes it possible to outline effective directions for optimizing the structure of cast metal matrix composites based on controlling the technological parameters of their production, focused on ensuring minimum porosity, targeted spatial distribution and volume fraction of the reinforcing phase. In general, the presented results show significant potential of tomography in the study of the structural and morphological characteristics of composite materials and internal defects in cast products made from them. Obviously, the expansion of the use of this method in the practice of scientific research will make it possible to achieve important progress in solving many fundamental and applied problems of modern materials science and engineering.

The potential economic benefits from the industrial use of *X*-ray computed tomography in non-destructive testing of cast metal matrix composites are associated, first of all, with an increase in production efficiency by minimizing the degree of uncertainty in relation to the internal spatial structure of materials and products. Specifically, tomographic methods make it possible to quickly reveal hidden defects in the cast structure of the material with obtaining precise statistical characteristics and, as a result, increase the yield of accepted products by prompt obtaining the necessary information for adjusting technological processes. Using tomography for evaluating the structure of metal matrix composites provides a drastic increase in informative value in comparison with other quality control methods and, certainly, can be considered as economically feasible in the industrial production of high-value products.

It is worth noting that, among other things, the expansion of the application of tomographic methods in the diagnostics of materials leads to the emergence of new directions in this field of science. If quasi-static 3D structures are described by spatial coordinates (x, y, z), then adding a fourth dimension (time, t) to the process of tomographic observation allows one to get a representation of the spatiotemporal evolution of the material structure. In the works of the Nobel laureate A. Zewail, such an approach to four-dimensional visualization in the coordinates (x, y, z, t) was used in the study of materials by ultrafast electron tomography with atomic scale resolution [24]. There are already some examples of effective application of *X*-ray computed tomography in the three-dimensional analysis of the evolution of various processes in materials at the microlevel in real time. In particular, significant progress in this direction has been achieved in the study of the processes of melting and crystallization, deformation and fracture, and various structural changes in composite materials under the influence of external factors [25–27]. The development of such approaches in relation to the investigation of cast metal matrix composites of the Al – Mg_2Si system will contribute to a better understanding of the real mechanisms of their structure formation, which will make it possible to determine the most promising ways to control the fractional and morphological characteristics of in-situ reinforcing phases and other structural constituents.

Conclusions

Based on the results of optimization the modes of tomographic scanning of metal matrix composites of the Al – Mg_2Si system, the identification of characteristic types of casting defects and structural constituents was carried out depending on the gray levels in their comparison with the characteristic morphology of the observed structural elements (primary Mg_2Si crystals, pseudobinary eutectic (α + Mg_2Si), iron-containing compounds). It is shown that the total volume of porosity in cast samples of Al + 15 wt.% Mg_2Si composites after

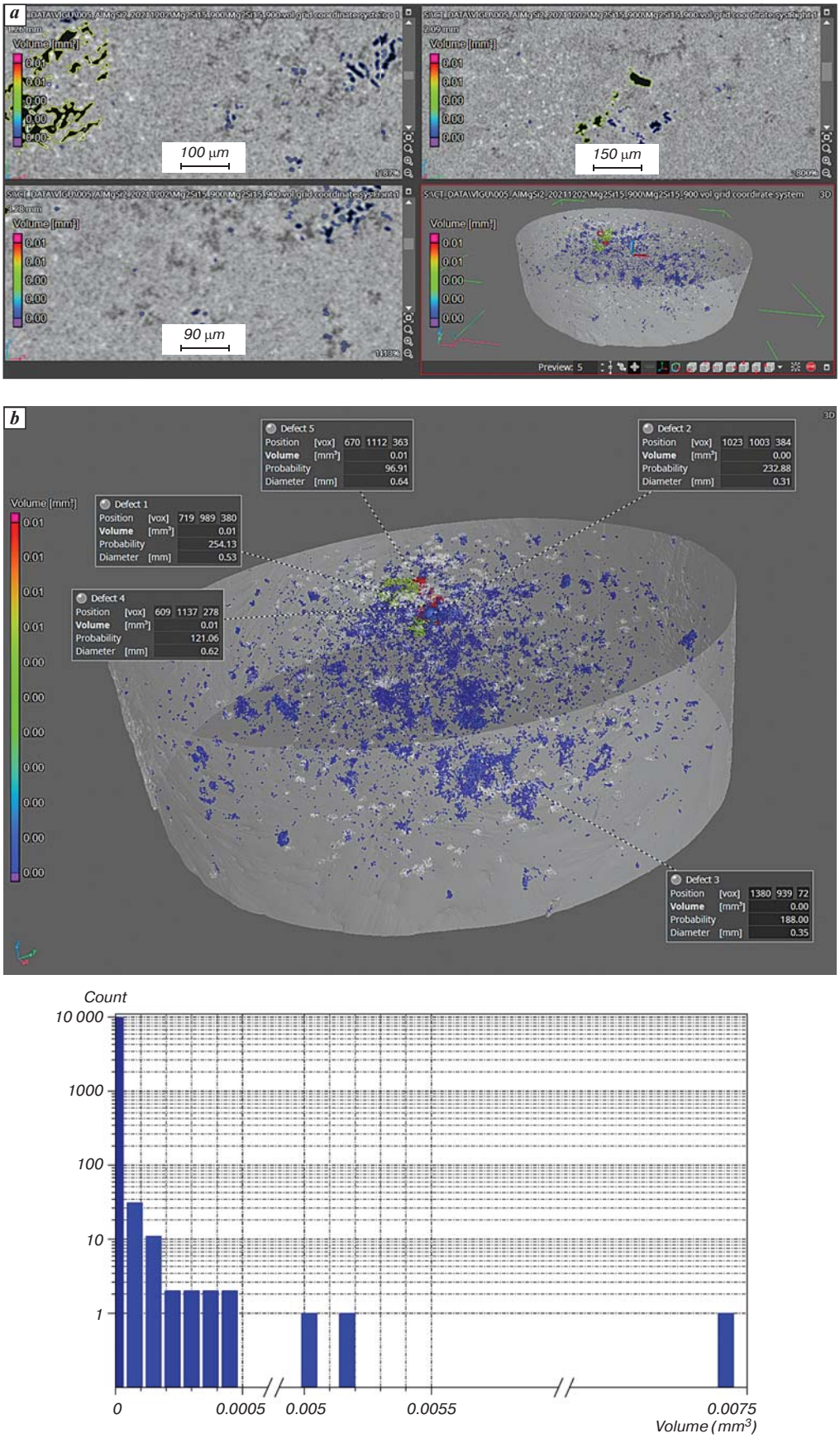

Fig. 2. Results of tomographic studies of Al + 15 wt.% Mg_2Si composites after the melt thermal-rate treatment at 900 °C, illustrating various defects in the cast structure (*a*), quantitative analysis of voids in the selected volume of the ingot (*b*) and void volume distribution (*c*)

the melt treatment-rate treatment at 900 °C does not exceed 0.05 mm^3, which corresponds to a porosity content in the sample of 0.13 vol.%. The results obtained confirm the possibility of using the melt thermal-rate treatment for the structural modification of Al – Mg_2Si composites without a significant increase in the porosity of cast billets in consequence of high-temperature overheating and prolonged isothermal exposure. In these conditions, *X*-ray computed tomography could be considered as a highly effective diagnostic tool for obtaining precision quantitative information necessary for the development of new technologies for melting and casting metal matrix composites.

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