

Powder Technology and Software Tools for Microstructure Control of AlCu₂ Samples

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Abstract. The powder technology of structurally inhomogeneous materials of $AICu₂$ sample formation is developed. The algorithm for image recognition of separate particles' microstructure of structurally inhomogeneous materials is formed and implemented in the Smart-eye software, which provides tools for analysis of surface and internal properties of structurally inhomogeneous materials. The developed powder technology is applied in obtaining the samples of $AICu₂$, which are further analysed by the Smart-eye software. The structural characteristics of the starting material $(AICu₂)$, in particular porosity, were predicted. The analysis of the average results of the study of the microstructure of $AICu₂$ particles is held, which shows that the developed models allow accurate control of the microstructure and properties of structurally inhomogeneous materials obtained based on powder technologies. Improvement of granulometric composition of structurally inhomogeneous materials is proved. Based on the obtained materials developed powder technology, it is possible to predict the structural characteristics of AlCu₂ raw materials at a qualitative level. Thus, it is possible to exercise to carry out practical realization of the received results on manufacture.

Keywords: Technology · Inhomogeneous materials · Metallographic analysis · Particles' · Smart-eye

1 Introduction

Modern mechanical engineering technologies are closely related to the development of modern production. The result is creating products from powder materials, which possess desired properties, e.g., strength, weight, porosity, and filtration coefficient. One of the ways to obtain such materials with qualitative indicators is the use of standard research methods. For this purpose, it is necessary to control all parameters of the manufacturing of a powder product. However, standard methods do not always allow to obtain the desired results of sample production AlCu₂. For improving the standard methods of research of powder metallurgy and study the perfect structure of materials that meet all the technical requirements of production, it is necessary to use a modern method of metallographic analysis. This method studies the destruction of the structure of the samples and predicts

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the internal properties of the source materials. Therefore, the study of the features of the internal connections of the structure of $AICu₂$ materials at the stage of their manufacture is a major problem of materials science.

2 Literature Review

40% of educators study the prediction of material properties worldwide. A relatively large number of researchers are engaged in studying the essential aspects of structurally heterogeneous materials at one time; one may refer to $[1]$. Scientific work is phenomenological [\[2\]](#page-7-1) and whose quality indicators do not coincide with the manufactured finished materials [\[3\]](#page-7-2). As described in paper [\[4\]](#page-7-3), the composition of the powders was obtained by chemical equations. Maintaining additional elements led to different porosity. Laboratory research [\[5\]](#page-7-4) modeling plays an important role, which allows for research without carrying out expensive and time-consuming experiments.

It should be noted that much work has been done by research teams [\[6\]](#page-7-5). The result is the final product with the help of laboratory equipment (magnifying glass, microscope, and millimeter ruler). That does not allow one to investigate completely inhomogeneous materials. According to [\[7\]](#page-7-6), the authors have investigated general analysis of structurally inhomogeneous materials. Different material parameters and boundary conditions were considered. The obtained interpolation dependences were obtained using the methods of boundary elements. In the study [\[8\]](#page-7-7), all necessary conditions for the production of an aluminum matrix are considered. The great advantage of scientific work is that the microstructure of the source powders is considered using a laser.

Today's conditions state that research in mechanical engineering technology should be conducted at a higher level [\[9\]](#page-7-8). For improving the quality of powder technologies [\[10,](#page-7-9) [11\]](#page-8-0), it should consider all the necessary technical requirements of modern production [\[12\]](#page-8-1). In turn, it is necessary to consider in detail AlCu₂ [\[13\]](#page-8-2) and its basic properties [\[14\]](#page-8-3). Starting with the filling of powders [\[15\]](#page-8-4) and ending with implementing the results at the enterprise [\[16\]](#page-8-5). The above materials indicate an imperfect level of research in this area, which leads to detailed methods of studying structurally inhomogeneous materials using technology.

3 Researches Methodology

The objective of the paper is to investigate study the internal connection of $AICu₂$ with each other. Calculation of individual AlCu₂ segments considering all the features of the Smart-eye program.

It should be noted that the scheme for obtaining products from powders consists of the following main processes: obtaining powders and preparing the powder charge, under pressure forming and sintering of moulded products, and a method for controlling the properties of structurally inhomogeneous materials in order to improve the accuracy of obtaining the final product. For most structurally inhomogeneous materials, the main object of industrial control and analysis of structural transformations is their geometric structure (microstructure of samples), which can be represented as a set of two and threedimensional elements in three-dimensional space. It is worth mentioning that an essential

practical task of metallographic analysis of structurally inhomogeneous materials is the obtainment of an estimate, which fixes the structural state of the material obtained as a result of a set of technological operations with known parameters (temperature, time, charge composition, particle size and shape, porosity, density, and others). The mass of powders forms a completely homogeneous bronze AlCu₂, consisting of 83% of copper and 17% of aluminium. In this case, a cylindrical sample: $\varnothing = 30$ mm, h = 60 mm was used. The percentage of the components' mass of the mixture that must be taken to prepare a cylindrical sample is presented in Table [1.](#page-2-0)

Table 1. The percentage of the components' mass of the covered mixture that must be taken to prepare a cylindrical sample $\varnothing = 30$ mm, h = 60 mm.

Sample	Al. $%$	Cu. %	Carbamide, %	Calcium carbonate, %	$\%$
Sample A	30	30	30	10	100
Sample B	40	35	25		100
Sample C	30	30	20	20	100

The pressing procedure was carried out using a hydraulic press PSU-125. The pressing pressure had been changed from 50 to 80 MPa. Sintering of the formed powder products (blanks) was carried out in an electric furnace VMK – 1600. Sintering temperature varied in the range between 1550–1600 °C. The heating rate was 200°/hour, and isothermal exposure landed at -1.5 h. The procedure of microscopic analysis was carried out according to the standard technique [\[9,](#page-7-8) [10\]](#page-7-9). The sample' lines were examined using the MMR-4 microscope model at $\times 600$ and $\times 800$ magnification. The digestion process was carried out with a 4% hydrochloric acid solution. Planar porosity was determined by the micrographs of the grinds using the Smart-eye application. The porosity was equal from 20% to 30%. The Smart-eye software allows for the determination of the necessary characteristics needed for qualitative and quantitative assessment of the structure of any material, including porous ones. The algorithm is as follows: let $b_{i,j}$ is the original image, the value of $b_{i,j}$ is equal to the brightness at the point $i, j \in D$, where: $i = 1, 2, \ldots, n; j = 1, 2, 3, \ldots, m$. The image $b_{i,j}$ of the real conditions, as well as the set of images of individual objects, will be equal to:

$$
b_{i,j} = H_1(i,j) + H_2(i,j) + \ldots + H_s(i,j)
$$
 (1)

where *S* is a number of real objects; H_k (*i, j*) are images of the *k*-th object, $k = 1, 2, 3$, *… s*.

The problem of image recognition, in this case, consists of finding all objects H_k $(i,$ *j)*, which are determined from the criteria of homogeneity of the region by the formula:

$$
\frac{max}{P \in R} |f(P) - m| \times T,
$$
\n(2)

where *T* is an initial value; *P* is the value in the field *R*; *m* is an average value of pixels in the area $P; f(P)$ is a function of the brightness distribution.

4 Results

4.1 Formation of Structurally Inhomogeneous Materials

The process of forming structurally inhomogeneous powder materials is as follows. In the first stage, the particles of raw materials move in the direction of the seal pressure. Large holes «pores» are filled. The deformation process does not occur. However, a high density of backfill is formed. In the second stage, the extrusion process takes place. That is, the free movement of materials stops, and as a result, deformation occurs. The sequence of the deformation process is as follows: soft deformation-elastic deformation-plastic deformation. The phenomenon of relationships between AlCu2 contacts is observed. Thus, the granulometric composition of the powder is changing – the number of grains increases by crushing, and smaller particles arise by breaking down the grains under high pressure in the areas of contact. Compressed samples are represented in (Fig. [1\)](#page-3-0).

Fig. 1. Compressed samples AlCu_2 : a – sample A; b – sample B; c – sample C.

Sintering of carved cylindrical products was carried out using an electric furnace. It should be noted that this oven has its special characteristics. This fully satisfies the technical requirements. A general overview of the sintered samples is shown in (Fig. [2\)](#page-3-1).

Fig. 2. General overview of the sintered samples $AICu_2$: a – sample A ; b – sample B ; c – sample C.

The essential features of powder materials are the hardness and strength of the material [\[17,](#page-8-6) [18\]](#page-8-7), which directly depends on the density of their crystal lattices in the volume of the controlled object (sample). The main foundation for this is the samples' chemical composition, oxidation state, particle size, concentration, and duration of exposure. The peculiarity of these studies is that the authors analyzed each particle of powders. The obtained results allowed us to confirm the high accuracy of calculations and to make the maximum description of the made samples. In this case, the «grains» were presented in photomicrographs as cut sections separated from each other by thick lines «boundaries». Also, «grains» can function with many areas of materials. The measurement was performed based on the total number of grains, which was carried out in millimeters. This method of monitoring the study of structurally inhomogeneous materials displays significant advantages: high sensitivity to the most dangerous defects, such as cracks and inclusions, the ability to control the density of powders, high productivity, and the ability to conduct control directly at the workplace without disrupting the process, as well as low-cost control and automation.

The Main Properties of Powders Based on the Analysis by the Smart-Eye Software Often, the practical implementation of the metallographic analysis of microimages (light, lumen, raster microscopy) considers the operations mentioned in the following paragraph. After loading the image of the object for study, pre-processed brightness and contrast adjustments were made. Then, it is necessary to carry out digital filtering. The next step is to transport the image of the micro-sections in their binary form and conduct segmentation. For interactive correction, computer visualization of the image was performed. Besides, at the final stage, the authors performed a statistical probability analysis of the distribution of particles and pores of powder materials. For detecting the image of homogeneous brightness areas in their characteristics, segmentation was performed, where different segments were likely to correspond to different structural elements. Preliminary analysis of the microstructure was performed on microsections of molding materials, which are presented in Fig. [3.](#page-5-0) The simplest and most common approach to solve the problem of image recognition is the so-called «limit discrimination method». In this case, the brightness matrix G turns into a binary matrix B, in which the non-zero elements correspond to the values of the original matrix and is biggest than the fixed threshold value g_{threshold}. Let us present the method of limiting discrimination as follows:

$$
G = \begin{pmatrix} g_{11} & \cdots & g_{1N} \\ \vdots & \ddots & \vdots \\ g_{M1} & \cdots & g_{MN} \end{pmatrix} \rightarrow B = \begin{pmatrix} b_{11} & \cdots & b_{1N} \\ \vdots & \ddots & \vdots \\ b_{1M} & \cdots & b_{MN} \end{pmatrix}
$$
(3)

where:

$$
b_{ij} = \begin{cases} 1, b_{ij} > g_{\text{threshold}} \\ 0, b_{ij} \le g_{\text{threshold}} \end{cases}
$$
 (4)

M and N – the dimensions of the micrometre matrix (computer units of length) image recognition, $g_{threshold}$ – is the threshold constant.

Figure [3,](#page-5-0) the authors presented a screenshot of the microstructure of the samples using the image recognition method.

Fig. 3. Screenshot of the microstructure with magnification $\times 600$ where: a) before etching, b) after etching

From the study results, it was found that the upper layer of microsections was obtained with heterogeneous indicators. Furthermore, the interaction of functional bonds depends on the etching of AlCu_2 materials (Fig. [3,](#page-5-0) b). The etching process, the surface of the materials, and their relief increased due to the grinding and polishing of cylindrical products. At the same time, there was observed an increase in pores. The above studies made it possible to record the porosity of structurally inhomogeneous materials.

It should be noted that all digestive processes were carried out under the same conditions in order to provide certain deviations that may have occurred during the study. It is also important to note that the experiments help determining the basic physical and mechanical properties of the original composition of the materials and obtained materials. Figure [4](#page-5-1) shows the fluctuations of the curves of the $AICu₂$ experiments. The morphological analysis of the samples shows the variation of the components that function with each other.

Fig. 4. The surface relief SNM with increasing \times 600 where: a) before etching, b) after etching.

The experiments show that the microstructure of the digestion samples in some areas differed. A larger digestive area (dark color) and a smaller digestive area (light color) were recorded. As a result, a microrelief was formed on the surface of the samples.

Table [2](#page-6-0) presents the results of experiments performed in the Smart-eye software (Fig. [3](#page-5-0) and [4\)](#page-5-1). In turn, the total area of the particles is 41 178.48 mkm2, calibration coefficient 0.83 mm \times pixel, porosity 21.48%.

Particle	X	Y	Width	Height	R_{min}	Convexity	Perimeter	Area	Quantity
$\mathbf{1}$	0.9	0.74	0.83	34.17	4.450	0.50	60.00	9.7	0.67
\overline{c}	1.67	0.75	30.83	47.50	9.32	0.80	55.04	10.13	0.83
\mathfrak{Z}	5.83	0.76	11.67	18.33	1.86	0.61	55.82	10.51	0.61
$\overline{4}$	8.33	0.78	9.17	7.50	0.83	0.39	27.74	19.44	0.46
5	30	0.79	7.50	7.50	2.36	0.75	27.71	31.60	0.74
6	6.67	0.80	8.33	11.67	1.18	0.63	37.69	62.15	0.79
7	4.17	$0.80\,$	61.67	34.17	0.83	0.38	50.83	68.40	0.40
8	9.17	0.83	5.00	15.00	0.83	0.56	37.34	43.75	0.57
9	4.17	0.83	15.00	12.50	0.90	0.61	50.33	39.79	0.86
10	7.50	0.83	10.83	7.50	0.83	0.62	33.76	48.26	0.77
11	8.83	0.83	15.00	11.67	3.44	0.74	49.87	22.92	0.89
12	10.6	0.83	11.67	11.67	2.36	0.73	42.93	46.04	0.75
13	8.33	0.83	5.83	3.33	1.80	0.73	37.24	11.81	0.72
14	9.17	0.72	22.50	24.17	4.17	0.65	92.55	11.11	0.87
15	9.84	2.50	18.33	25.83	1.18	0.63	78.50	78.47	0.82
16	9.17	0.83	5.83	6.67	0.83	0.63	20.15	17.01	0.76
17	5.0	2.50	7.50	19.17	0.83	0.40	49.70	72.22	0.53
18	6.67	4.17	3.33	7.50	0.90	0.45	18.90	10.07	0.51
19	5.83	5.00	14.17	10.00	0.90	0.53	43.92	85.07	0.80
20	9.17	7.50	5.83	7.50	1.18	0.63	23.12	20.49	0.69
21	9.67	8.33	5.00	5.83	0.20	0.59	16.71	7.29	0.47
22	5.0	10.8	1.67	8.33	0.19	0.65	18.65	5.21	0.27
23	7.50	10.5	7.50	7.50	0.90	0.61	26.40	34.38	0.89
24	9.17	13.3	5.00	11.67	0.83	0.52	28.75	19.10	0.42
25	3.3	13.8	9.17	13.33	2.36	0.70	39.36	66.67	0.78

Table 2. Average experimental results of formation research AlCu₂.

Therefore, studies show that the behavior of particles of powder materials plays an important role, which from any point of view depends on the peculiarities of the formation of its microstructure. Based on the functional and internal relationships between the components of the studied materials and their physical and mechanical properties, it can be got a simple quality control method of the final product.

5 Conclusions

The paper develops a solid approach to design, obtainment, and control of structurally inhomogeneous materials based on powder technology. In particular, the developed models for image recognition and investigation of microstructures, both structural and inhomogeneous powder materials, allow to investigate and predict physical and mechanical properties of materials:

- linear dimensions, cross-sectional area, and perimeter of the intersection of structural components and phases;
- radius and volume of powder particles;
- distribution of particles structurally inhomogeneous materials in different forms and size, as well as the evaluation of angles of orientation;
- perimeters and cross-sectional area of ready products and the analysis of defects in the manufacture of structurally inhomogeneous materials.

References

- 1. Long, Z., Heng-wei, Z.: Sintering driving force of $A₁O₃$ powders at the initial stage of pulse electric current sintering under thermoelastic diffusion. Int. J. Mech. Mater. Eng. **13**(1), 2–8 (2018)
- 2. Apurba Kanti, D., Chatterjee, P.: Study of deformation microstructure of nickel samples at very short milling times: effects of addition of α-Al2O3 particles. J. Theor. Appl. Phys. **13**, 63–73 (2019)
- 3. Guo, F., Weihui, J., Jianmin, L.: A novel green nonaqueous sol-gel process for preparation of partially stabilized zirconia nanopowder. Process. Appl. Ceram. **11**(3), 220–224 (2017)
- 4. Li, W., Xu, F., Li, Y., Hu, X., Bo, D., Yu, X.: Discussion on microwave-matter interaction mechanisms by in situ observation of "core-shell" microstructure during microwave sintering. Materials **9**(3), 2–12 (2016)
- 5. Zabolotnyi, O., Pasternak, V., Andrushchak, I., Ilchuk, N., Svirzhevskyi, K.: Numerical simulation of the microstructure of structural-inhomogeneous materials. In: Ivanov, V., Trojanowska, J., Pavlenko, I., Zajac, J., Peraković, D. (eds.) DSMIE 2020. LNME, pp. 562–571. Springer, Cham (2020). https://doi.org/10.1007/978-3-030-50794-7_55
- 6. Yu, X., Xu, F., Dong, B., Li, W., Hu, X.: Discussion on the local magnetic force between reversely magnetized micro metal particles in the microwave sintering process. Metals-Open Access Metall. J. **7**(2), 2–11 (2017)
- 7. Sulym, H., Pasternak, I., Pasternak, V.: Boundary element modeling of pyroelectric solids with shell inclusions. Mech. Mech. Eng. **22**(3), 727–737 (2018)
- 8. Marchese, G., Aversa, A., Lorusso, M., Manfredi, D., Calignano, F.: Development and characterisation of aluminium matrix nanocomposites $AISi10MgMgAl_2O_4$ by laser powder bed fusion. Metals **8**(3), 1–12 (2018)
- 9. Carneiro, Í., Viana, F., Vieira, M., Fernandes, J., Simões, S.: EBSD analysis of metal matrix nanocomposite microstructure produced by powder metallurgy. Nanomaterials **9**(6), 1–12 (2019)
- 10. Magnani, G., Galvagno, S., Sico, G., Portofino, S., Freda, C., Burresi, E.: Sintering and mechanical properties of β-SiC powder obtained from waste tires. J. Adv. Ceram. **5**, 40–46 (2016)
- 11. Lytvynenko, A., et al.: Ensuring the reliability of pneumatic classification process for granular [material in a rhomb-shaped apparatus. Appl. Sci. \(Switzerland\)](https://doi.org/10.3390/app9081604) **9**(8), 1604 (2019). https:// doi.org/10.3390/app9081604
- 12. Zhang, Q., Xu, L.S., Guo, X.: Improvement of mechanical properties, microscopic structures, and antibacterial activity by Ag/ZnO nanocomposite powder for glaze-decorated ceramic. J. Adva. Ceram. **6**(3), 269–278 (2017). <https://doi.org/10.1007/s40145-017-0239-z>
- 13. Fathy, N., Ramadan, M., Hafez, K., Alghamdi, A., Halim, A.: Microstructure and induced defects of 6061 Al alloy after short times cyclic semi-solid heat treatment. MATEC Web Conf. **67**(5), 1–6 (2016)
- 14. Samar Reda, A., Hamid, A., Menam, A., Salah Elden, I., Haytham Abdelrafea, E., Hassan Abdel, S.: Laser powder cladding of Ti-6Al-4V α/β alloy. Materials **10**(11), 2–16 (2017)
- 15. Lin, Z., Tian-Shu, L., Tao-Tao, D., Tao-Tao, L., Feng, Q., Hong-Yu, Y.: Design of a new Al-Cu alloy manipulated by in-situ nanocrystals withsuperior high temperature tensile properties and its constitutive equation. Mater. Des. **181**(2), 1–12 (2019)
- 16. Vijay Ponraj, N., Azhagurajan, A., Vettivel, S.: Microstructure, consolidation and mechanical behaviour of Mg/n-TiC composite. Alex. Eng. J. **55**(2), 2077–2086 (2016)
- 17. Hu, W.J.: Effects of metal particles on cold spray deposition onto Ti-6Al-4V alloy via Abaqus/Explicit. J. Eng. Sci. **7**(2), E19–E25 (2020). [https://doi.org/10.21272/jes.2020.7\(2\).e4](https://doi.org/10.21272/jes.2020.7(2).e4)
- 18. Kubit, A., Faes, K., Jurczak,W., Bucior, M., Kluz, R.: Analysis of the properties of RFSSW lap joints of Alclad 7075–T6 aluminum alloy sheets under static and dynamic loads. Technologia i Automatyzacja Montażu **4**, 4–13 (2020)