

Development of Composites with Icosahedral Phase in Al–Cu–Fe Quasicrystalline Alloys Obtained by the Bridgman Method

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The composites were obtained by the Bridgman method through solidification of $\text{Al}_{61}\text{Cu}_{27}\text{Fe}_{12}$ alloy (numbers indicate at.%). The microstructure of composites with crystal matrix and quasicrystal reinforcement was studied. The crystalline β -phase was the matrix and the quasicrystalline i -phase was the reinforcement of obtained composites. The shape and spatial distribution of reinforcement fibres were specified. Some geometrical relations of the fibres arrangement were defined. Obtained composite samples were subjected to X-ray phase analysis, optical, and scanning electron microscopy observations, chemical microanalysis and the Laue diffraction.

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1. Introduction

Constantly developing automotive, aerospace, and electric power industries necessitates the use of more and more advanced construction materials. These materials must meet the still increasing requirements for strength parameters. The metal matrix composites are currently used for example as one of the main type of materials for the production of turbine engines components. Presence of the quasicrystalline phase, having properties similar to ceramics, in the crystalline matrix, results in possibility of using these type crystal-quasicrystal composites as high-temperature elements. The quasicrystals possess, *inter alia*, good stability in high temperatures and high hardness but they are fragile [1–8]. The composite phases pose specific thermal properties [9]. In order to increase the clear anisotropy of thermal conductivity, fibrous composites with continuous reinforcement with directional arrangement are produced. Potential effect of the significant anisotropy of heat abstraction make the crystal-quasicrystal composites useful as high temperature superficial materials with directional heat abstraction. The technologies of obtaining the crystal-quasicrystal composites are already developed [10–12]. However, these are mainly *ex situ* technologies. The fibrous composites of better quality may be obtained *in situ* by directional crystallization [13]. Presented composites were obtained using the vertical Bridgman method with the withdrawal rate of 0.07 mm/min. In the industrial production higher withdrawal rates are needed. Therefore the aim of this work was to obtain by the Bridgman method the composites with quasicrystal icosahedral phase with withdrawal rate of 0.5 mm/min and analysis of their microstructure, especially the type and arrangement of the reinforcement.

2. Experimental and results

The composite ingots were obtained by the vertical Bridgman method using an induction heating furnace. The raw materials of the chemical composition of $\text{Al}_{61}\text{Cu}_{27}\text{Fe}_{12}$ were melted in alumina crucibles under inert atmosphere. The withdrawal rate in solidification processes was 0.5 mm/min. Such rate causes constitutional supercooling process. Obtained ingots in the shape of crucible were 20 mm in diameter and 80 mm in length. The ingots were cut into samples with planes parallel and perpendicular to the solidification direction (Fig. 1). The samples will be referred to hereinafter respectively as parallel and perpendicular samples.

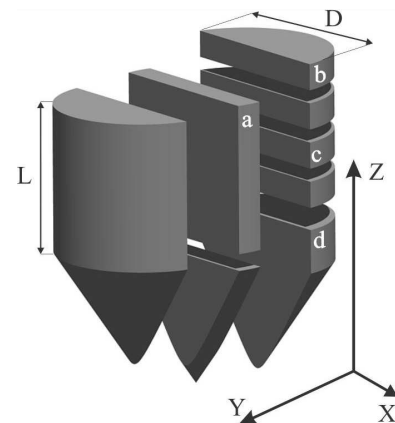


Fig. 1. Scheme of ingots cutting.

Powdered fragments of the ingots were examined by X-ray diffraction (XRD) phase analysis. A JEOL JEM-1040 transmission electron microscope, equipped with an EDX system was used to confirm the presence of the quasicrystal phase. The fragments of ingot, containing each of identified by XRD phases, were powdered and

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dispersed on a carbon grid and then the electron diffraction patterns were obtained. The microsections of perpendicular and parallel samples were subjected to optical and scanning electron microscopy observations. Chemical composition in selected areas of the samples was examined by scanning electron microscope of JEOL JSM-6480 equipped with EDXS system. The samples were subjected to the Laue diffraction method for the analysis of single-crystallinity of component phases.

Two separate phases were identified by the X-ray phase analysis: the β -phase and the i -phase. The β -phase is an Al(Cu,Fe) cubic crystalline phase and the i -phase is an Al₆Cu₂Fe icosahedral quasicrystalline phase. Typical X-ray diffraction pattern is presented in Fig. 2. Electron diffraction patterns, obtained for i -phase, show the symmetry of fivefold axis.

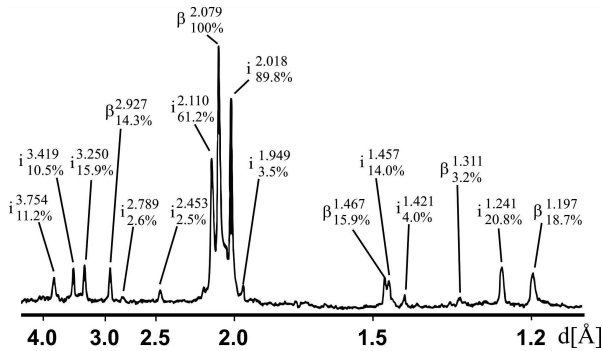


Fig. 2. Typical XRD pattern obtained for Al₆₁Cu₂₇Fe₁₂ alloy.

Main research focused on the analysis of occurring, the type and arrangement of reinforcement in the obtained composites by the SEM and optical microscopes. There are visible two kinds of areas in the obtained micrographs (Figs. 3–5). The dark area is a dominant in whole ingot (matrix of the composite) and represents the β -phase. The bright area represents the i -phase of reinforcement of the composite. It was confirmed by comparison of stoichiometric formula of previously identified phases and chemical composition (elements ratio) obtained by the chemical microanalysis at selected points of both areas.

Observation of the parallel samples microsection (Fig. 3) showed that the reinforcement occurs in the form of short irregular strand, stretched in the solidification direction. Some of them are combined into longer segments (black rim) similar to the irregular fibres. The microstructure visible on the perpendicular samples cross-sections is different depending on the area of the ingot (b, c, d — Fig. 1). Figure 4 shows the microsection of perpendicular sample of ingots area b. The bright regions represent the composite reinforcement occupy less area than the matrix. The reinforcement usually is not arranged in specific directions and has irregular shapes. There are numerous voids in the upper part of the ingots, marked in Fig. 4b by white rims. The dodecahedral structures were observed inside the voids.

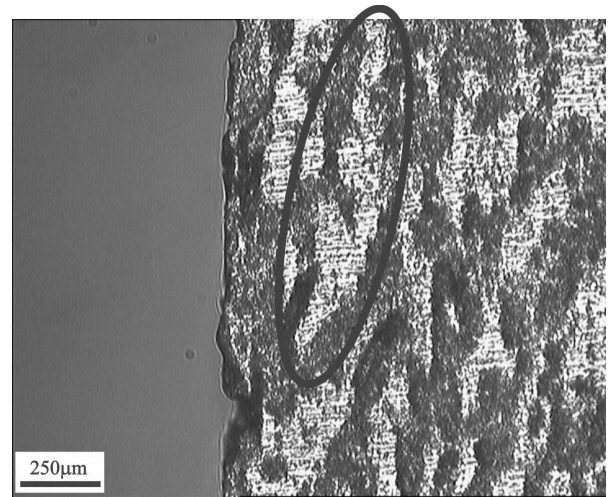


Fig. 3. Optical micrograph of parallel sample microsection of Al₆₁Cu₂₇Fe₁₂ composite.

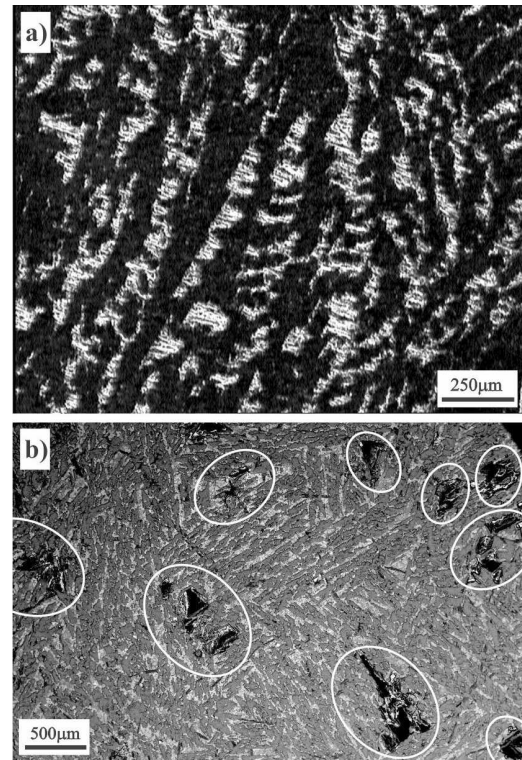


Fig. 4. (a) Optical and (b) SEM micrograph of perpendicular sample microsection of Al₆₁Cu₂₇Fe₁₂ composite.

The microstructure in the central part of the ingots (ingots area c, Fig. 1) is more ordered. Figure 4 and 5 show typical microsection of perpendicular samples of ingots area c. There are visible different types of reinforcement arrangement. The specific directions of the reinforcement can be determined. The directions were marked by drawing lines, lying at equal distances from the opposite designated edge of the reinforcement. This

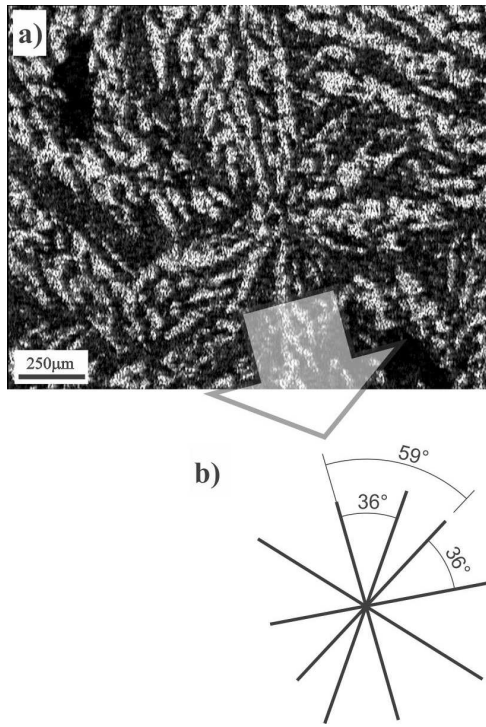


Fig. 5. (a) Optical micrograph of perpendicular sample microsection with (b) star-type fibres arrangement scheme.

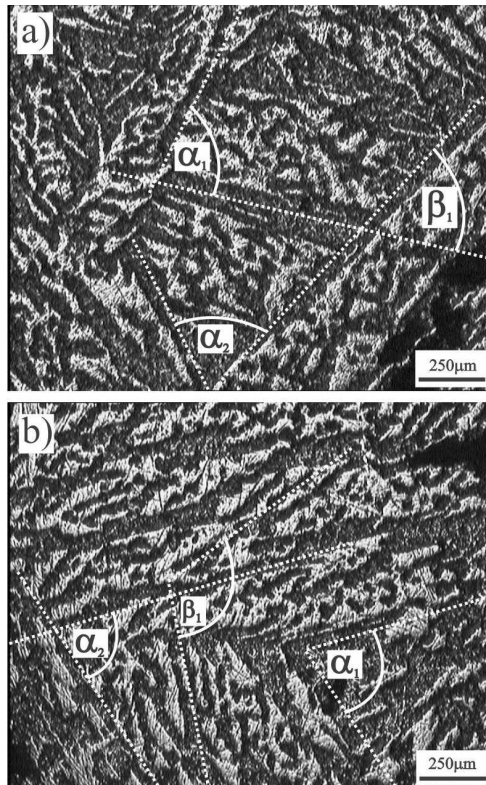


Fig. 6. Optical micrograph of perpendicular sample microsection with triangle-type fibres arrangement scheme.

edge is a straight trend line, drawn along the boundary between the reinforcement and the matrix. The geometrical relations of the fibres arrangement were determined by measuring the angles between the specific directions.

One of the types of reinforcement arrangement is presented in Fig. 5. The reinforcement propagates from the central point in specific starlike directions (Fig. 5a). This type of arrangement will be called a star-type. The angles between specific directions were measured and the results are shown in Fig. 5b. Most of the measured angles were about 36° and 60° .

Another type of reinforcement arrangement is presented in Fig. 6. There are visible bright areas of the fibers on the dark background. It can be defined specific arrangement directions. Because the fibers are parallel to ingots vertical axis Z , these directions represent the arrangement of fibers column or plane. In all cross-sections there is the same fibers arrangement. Angles between selective chains of fibers are marked with α and β symbols. The α and β symbols values are presented in Table. Measured angles represent two groups of symmetry. The α angles represent quasicrystalline symmetry (i.e. $\approx 36^\circ$, $\approx 72^\circ$, $\approx 144^\circ$) and the β angles represents crystalline symmetry (i.e. $\approx 60^\circ$, $\approx 120^\circ$). There are both angles groups shown in Fig. 6. To make the figures clear some angles have not been marked. The single underlined angle values are related with six- or threefold symmetry ($\approx 60^\circ$ or $\approx 120^\circ$; crystalline) and the double underlined angles values are related with fivefold symmetry ($\approx 72^\circ$; quasicrystalline).

TABLE

The angles values of reinforcement arrangement presented in Fig. 6. Single underline — crystalline symmetry, double underline — quasicrystalline symmetry.

Fig. no.& α_1	α_2	β_1	
6a	<u>$75^\circ \pm 3^\circ$</u>	<u>$70^\circ \pm 3^\circ$</u>	<u>$60^\circ \pm 3^\circ$</u>
6b	<u>$73^\circ \pm 3^\circ$</u>	<u>$75^\circ \pm 3^\circ$</u>	<u>$123^\circ \pm 3^\circ$</u>

The investigations by the Laue method revealed that the component phases were not single-crystalline. On all obtained Laue diffraction patterns for all analysed samples sharp diffraction spots were not observed.

3. Summary

There were two phases identified in obtained composites — the dominant β -phase of matrix and i -phase of short fibres. Created fibres have very irregular shapes in cross-sections. The planes perpendicular to the solidification direction created groups of chains arranged in the optical bands. The directions of bands formed angles with characteristic repetitive values. Two groups of the angle values, related to the crystalline and quasicrystalline symmetry, may be determined. There were measured angles of $\approx 36^\circ$, $\approx 72^\circ$, and $\approx 144^\circ$, representing quasicrystalline symmetry and angles of $\approx 60^\circ$ and

$\approx 120^\circ$, representing crystalline symmetry. The most of measured angle values relate with quasicrystalline symmetry. The i-phase may grow before other phases due to similar chemical composition of the i-phase to starting chemical composition. The symmetry of i-phase may impose the characteristic angles and such fibers arrangement.

4. Conclusions

The growth rate have an impact on the quality and morphology of the reinforcement and the quantity and type of component phases of the composite, which can be determined from a comparison of the results of research and presented in Ref. [13]. The reinforcement shape is irregular and the fibers are short in composites obtained with higher growth rate. The composite contain more areas with voids and the voids are larger. The directional solidification by the Bridgman method with such high growth rate does not allow to obtain the single-crystalline component phases.

References

- [1] K. Urban, M. Feuerbacher, M. Wollgarten, *MRS Bul.* **11**, 65 (1997).
- [2] L.M. Zhang, H.C. Zhang, Q.G. Zhou, C. Dong, *Wear* **225-229**, 784 (1999).
- [3] X. Li, L. Zhang, H. Gao, *J. Phys. D, Appl. Phys.* **37**, 753 (2004).
- [4] M. Texier, J. Bonneville, A. Proult, J. Rabier, N. Baluc, P. Guyot, *Scr. Mater.* **49**, 41 (2003).
- [5] V.I. Trfilov, Y.V. Mil'man, D.V. Lotsko, A.N. Belous, S.I. Chugunova, I.I. Timofeeva, A.I. Bykov, *Dokl. Akad. Nauk* **373**, 470 (2000).
- [6] F. Tang, I.E. Anderson, S.B. Biner, *Mater. Sci. Eng.* **363**, 20 (2003); Prashanth, V. Uhlenwinkel, M. Calin, J. Eckert, *J. Alloy. Comp.* **536**, 130 (2012).
- [7] J.-M. Dubois, in: *Quasicrystals, an Introduction to Structure, Physical Properties and Applications*, Eds. J.-B. Suck, M. Schreiber, P. Häussler, Springer, Berlin 2002, p. 507.
- [8] C. Janot, L. Loreto, R. Farinato, *Phys. Status Solidi B* **222**, 121 (2000).
- [9] M. Matsukawa, M. Yoshizawa, K. Noto, Y. Yokoyama, A. Inoue, *Physica B* **263-264**, 146 (1999).
- [10] F. Masa, I. Akihisa, K. Hisamichi, Japanese Patent, publication no. 2006274311A, appl. no. 2005092072 (2006).
- [11] T. Masumato, A. Inoure, J. Nagahora, T. Shibata, K. Kita, US Patent 5,607,526 (1997).
- [12] F. Ali, S. Scudino, G. Liu, V.C. Srivastava, N.K. Mukhopadhyay, M. Samadi Khoshkhoo, K.G. Prashanth, V. Uhlenwinkel, M. Calin, J. Eckert, *J. Alloys Comp.* **536**, S130 (2012).
- [13] J. Krawczyk, W. Bogdanowicz, T. Goryczka, *Philos. Mag.* **90**, 3987 (2010).