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ON THE ORIENTATION CHARACTERISTICS IN DIRECTIONALLY CRYSTALLIZED Al-CuAl₂ EUTECTIC ALLOY

CHARAKTERYSTYKI ORIENTACJI W KIERUNKOWO KRYSTALIZUJĄCYM EUTEKTYCZNYM STOPIE Al-CuAl₂

The paper presents the orientation characteristics of (Al) and $CuAl_2$ phases in directionally crystallized Al-CuAl₂ eutectic alloy. The orientations of lamellae of the phases with respect to the crystallization direction and the orientation relationship between neighbouring lamellae were determined using the Electron Back Scattering Diffraction (EBSD) technique in a scanning electron microscope with the application of the formalism of quantitative texture analysis. The global texture was analyzed based on the orientation distribution functions (ODFs) calculated from the pole figures measured using X-ray diffraction technique.

There were examined samples obtained at various crystallization rates. The orientation measurements were carried out in selected areas in the plane perpendicular to the crystallization direction. Both the morphology and the microtexture of the alloy changed with the crystallization rate.

Keywords: directional crystallization, microtexture, orientation relationship

W pracy przedstawiono charakterystyki orientacji faz (Al) i CuAl₂ w kierunkowo krystalizującym stopie eutektycznym Al-CuAl₂. Orientacje płytek faz względem kierunku krystalizacji oraz relacje orientacji pomiędzy sąsiadującymi płytkami określono przy wykorzystaniu techniki dyfrakcji elektronów wstecznie rozproszonych (EBSD) w skaningowym mikroskopie elektronowym przy zastosowaniu formalizmów ilościowej analizy tekstur. Figury biegunowe zmierzone techniką dyfrakcji rentgenowskiej umożliwiły wyznaczenie funkcji rozkładu orientacji i charakterystykę tekstury globalnej.

Badano próbki otrzymane przy różnych prędkościach krystalizacji. Pomiary orientacji wykonano w wybranych obszarach w płaszczyźnie prostopadłej do kierunku krystalizacji. Zarówno morfologia jak i mikrotekstura stopu zmieniały się wraz z prędkością krystalizacji.

1. Introduction

Al-CuAl₂ eutectic alloy obtained in the process of directional crystallization represents an interesting case of a two-phase material. In the course of such a crystallization process the lamellae of the phases grow in the direction approximately parallel to the crystallization direction (direction of the greatest heat flow). As a result a material of a distinct crystallographic texture is formed [1, 2, 3].

In the paper there has been presented the analysis of the changes in the texture and microtexture of Al-CuAl₂ eutectic alloy obtained at various rates of directional crystallization. The orientation characteristics were determined on the basis of pole figures measured by the technique of X-ray diffraction and the orientation distribution functions (ODFs) calculated on their basis as well as sets of single orientations measured in a scanning electron microscope using EBSD technique. This allowed to present both the global as well as local texture (microtexture) in selected areas of a section perpendicular to the crystallization direction.

Microtexture of Al-CuAl₂ alloy has been described by means of the orientations of (Al) and CuAl₂ lamellae with respect to the plane perpendicular to the crystallization direction and by determination of the orientation relationship between the neighbouring lamellae of both phases. The orientation relationship was presented by means of Miller's indices of planes parallel to interface and parallel directions lying in these planes.

2. Analysis of the global texture

Using X-ray diffraction technique (Philips XPert diffractometer in IMIM PAS Kraków) there has been determined the global texture of Al-CuAl₂ alloy (the measurement area was 1.5 mm \times 1.5 mm), crystallizing

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at various rates. There were measured the pole figures of the planes $\{220\}$ and $\{311\}$ for (Al) phase and $\{213\}$, $\{411\}$ for CuAl₂ phase in the plane perpendicular to the crystallization direction.

Three-dimensional orientation distribution functions were determined by the ADC method [4, 5]. The characteristic ODF sections for the analyzed samples were given in figures 1, 2 and 3.



Fig. 1. The section of orientation distribution function of (Al) phase for $\varphi_2 = 20^\circ$ (a) and of CuAl₂ phase for $\varphi_2 = 50^\circ$ (b) (the sample obtained in crystallization process at the rate $21 \cdot 10^{-5}$ cm/s)

Max=6259.013

Min=0.000





Fig. 2. The section of orientation distribution function of (Al) phase for $\varphi_2 = 35^\circ$ (a) and of CuAl₂ phase for $\varphi_2 = 65^\circ$ (b) (the sample obtained in crystallization process at the rate $85 \cdot 10^{-5}$ cm/s)



Fig. 3. The section of orientation distribution function of (Al) phase for $\varphi_2 = 50^{\circ}$ (a) and of CuAl₂ phase for $\varphi_2 = 5^{\circ}$ (b) (the sample obtained in crystallization process at the rate 333·10⁻⁵ cm/s)

Investigations using XRD technique have shown that both phases have clearly distinct orientation characteristics for each phase. In Table 1 there are presented the orientations {hkl} planes perpendicular to the crystallization direction of the phases (Al) and CuAl₂ for samples crystallizing at the rates: $21 \cdot 10^{-5}$ cm/s, $85 \cdot 10^{-5}$ cm/s and $333 \cdot 10^{-5}$ cm/s.

TABLE 1

The orientations of (A1) and $CuAl_2$ phases in samples crystallizing at different rates determined by means of X-ray diffraction technique

Crystallization rate [cm/s]	Planes perpendicular to the crystallization direction	
	(Al)	CuAl ₂
$v = 21 \cdot 10^{-5}$	{1 2 7}	{1 1 5}
$v = 85 \cdot 10^{-5}$	{1 1 2}	{2 1 3}
$v = 333 \cdot 10^{-5}$	{4 3 5}	{3 0 2}

On the basis of the ODF values the measure of texture sharpness was also estimated by determining the scatter (half widths) of peaks corresponding to local maxima of the ODF in both phases – Table 2.

TABLE 2 The peak half scattering widths corresponding to the local maxima of orientation distributions in (Al) and CuAl₂ phases

	The half scattering widths [°] estimated for the samples		
Phase	crystallizing at the rates:		
	21·10 ⁻⁵ cm/s	85·10 ⁻⁵ cm/s	333·10 ⁻⁵ cm/s
(Al)	4	5	5
CuAl ₂	4	5	7

The sharpness in the examined material for each of the three crystallization rates was very high. For this reason the orientation distributions determined in the standard network $(5^{\circ} \times 5^{\circ} \times 5^{\circ})$ on the basis of pole figures measured in the $(2^{\circ} \times 5^{\circ})$ network enabled only a rough estimation of the position of local maxima and the scatter values. The estimated values of the scatter $(4^{\circ} - 7^{\circ})$ are close to the distance of the network nodes (5°) in which the ODFs values were determined.

3. Analysis of local texture – microtexture

The EBSD technique in SEM provides information about the grain orientations, differences in the orientations of the neighbouring grains and it enables the identifications of the phases [6, 7, 8].

Measurements carried out in SEM by EBSD technique (FEI E-SEM XL30 in IMIM PAS Kraków and SEM FEG JSM 6500F in Ecole Nationale Superieure des Mines de Saint – Etienne) enabled to trace the changes in the microtexture at various places of the analyzed samples. Measured sets of orientations are presented on the orientation maps. These maps visualize the local distribution of orientations of phases.

3.1. Analysis of orientations and orientation relationships of phases in a sample obtained as a result of crystallization at the rate $21 \cdot 10^{-5}$ cm/s

The microtexture in a cross-section of a sample obtained as a result of crystallization at the rate $21 \cdot 10^{-5}$ cm/s and the corresponding maps of phase orientations are presented in Fig. 4. The orientation measurement was performed at the step 0.5 µm in the marked areas. The analyzed area contains morphological inhomogeneities occurring at the boundary of subgrains connected with the formation of an additional lamella and shifting of lamellae lying nearby.

On the basis of the measured sets of single orientations and above presented orientation maps, characterizing the microstructure of a sample obtained at the rate $21 \cdot 10^{-5}$ cm/s, there have been defined the planes perpendicular to the crystallization direction as {216} (Al) and {114} CuAl₂. Phase orientations determined above the boundary of subgrains differ by about 10° from those lying below. This means a small rotation of lamellae (at the subgrains boundary) about the direction of crystallization. Such a change of orientation, however, does not induce a change of the distinguished planes of (Al) and CuAl₂ phases.

Relation of the orientation of the phases (Al) and $CuAl_2$ occurring on both sides of interphase surface in the area presented in the figures has been defined as: {111} (Al) // {211} CuAl_2, <110> (Al) // <120> CuAl_2. There has been also distinguished another relationship:

{112} (Al) // {212} CuAl₂, <110> (Al) // <120> CuAl₂, however in this case the angle between the normal to the planes is greater.

a)



"Subgrain boundary"



Fig. 4. The microstructure of measurement area (a) and the orientation map of (Al) and $CuAl_2$ phases (b) in the sample obtained in crystallization process at the rate $21 \cdot 10^{-5}$ cm/s

Planes perpendicular to the crystallization direction:	Orientation relationship of phases (A1) and $CuAl_2$:	
(216) (Al)	(111) (Al) // (211) CuAl ₂	
$\square (11\overline{4}) \operatorname{CuAl}_2$	[110] (Al) // [120] CuAl	
	$(11\bar{2})$ (Al) // (212) CuAl ₂ $[\bar{1}10]$ (Al) // $[1\bar{2}0]$ CuAl ₂	

3.2. Analysis of orientations and orientation relationships of phases in a sample obtained as a result of crystallization at the 85.10⁻⁵ cm/s

In the case of a sample obtained as a result of crystallization at the rate $85 \cdot 10^{-5}$ cm/s the area presented in

Fig. 5 has been selected for analysis. The measurement step was 0.4 µm.





Fig. 5. The microstructure of measurement area (a) and the orientation map of (Al) and CuAl₂ phases (b) in the sample obtained in crystallization process at the rate 85.10⁻⁵ cm/s

Planes perpendicular to the crystallization direction:

Orientation relationship of phases (Al) and CuAl₂:

(111) (Al) // (211) CuAl₂ ♦ (213) CuAl, [110](Al)//[120]CuAl, (112) (Al) // (212) CuAl,

[110](Al)//[120]CuAl,

Orientations defined in the plane perpendicular to the crystallization direction, which occur in the analyzed area, are the following: {112} (Al) and {213} CuAl₂. The orientation relationship of the phases {111} (Al) // {211} $CuAl_2$, <110> (Al) // <120> $CuAl_2$ is identical with that found for the earlier analyzed sample $(21 \cdot 10^{-5} \text{ cm/s})$. It is also possible, similarly as in earlier case, to distinguish the relation {112} (Al) // {212} CuAl₂, <110> (Al) // <120> CuAl₂.

3.3. Analysis of orientations and orientation relationships of phases in a sample obtained as a result of crystallization at the rate $333 \cdot 10^{-5}$ cm/s

Analysis of the microstructure of a sample obtained as a result of crystallization at the rate $333 \cdot 10^{-5}$ cm/s is the most interesting on account of different morphological features of the microstructure. This microstructure is composed of eutectic cells, in which the lamellae deviate in the direction of their boundaries. The cells visible on the sample cross-section are composed of lamellae of various thickness and length. In the case of this sample the measurements of single orientations were made with the step 0.3 μ m (Fig. 6).

a)

b)





Fig. 6. The microstructure of measurement area (a) and the orientation map of (Al) and CuAl₂ phases (b) in the sample obtained in crystallization process at the rate 333.10⁻⁵ cm/s

Planes perpendicular to the crystallization direction;	Orientation relationship of phases (AI) and CuAl $_{2}$:
(334) (Al) (302) CuAl ₂	(111) (Al) // (211) CuAl ₂ [211] (Al) // [10 $\overline{2}$] CuAl ₂
	$(11\bar{1})(Al)//(\bar{1}2\bar{2})CuAl_2$
	[121](Al)//[221]CuAl ₂

Similarly as in the earlier considered samples there have been determined the phase orientations occurring in the examined area of the cross-section. These are: {334} (Al) and {302} CuAl₂. The dominating orientation relationship is, as in previous samples, {111} (Al) // {211} CuAl₂, however with the relation of planes preserved, a change of the directions has been observed, mainly <211> (Al) // <102> CuAl₂.

4. Summary

Texture investigations based on methods using the technique of X-ray diffraction (measurement of pole figures) supply the macroscale characteristics of the so-called global texture. A complete multi scale description can be obtained when analyzing also the local texture, i.e. the microtexture, which is made possible using technique of electron microscopy, e.g. EBSD.

When analyzing the texture in the examined alloy it has been found that the orientations of (Al) and CuAl₂ phases, defined in the plane perpendicular to the crystallization direction become changed with the rate of crystallization.

In the case of a sample crystallizing at $21 \cdot 10^{-5}$ cm/s the planes defined by the X-ray technique are the following: {127} (Al) and {115} CuAl₂, whereas those defined by the methods of orientation microscopy in SEM are {126} (Al) and {114} CuAl₂.

For a sample crystallizing at the rate $85 \cdot 10^{-5}$ cm/s orientation of (Al) phase, independently of the measurements method always are {112}, whereas that of CuAl₂ phase {213}.

In the case of a sample obtained as results of crystallization at $333 \cdot 10^{-5}$ cm/s, the determined orientations are: {334} (Al) (SEM EBSD) and {435} (Al) (XRD), whereas {302} CuAl₂ (SEM EBSD and XRD).

Thus orientations determined by both those methods are close to each other.

The distinguished orientation relationship of phases at the interface surface in samples crystallizing at the rates: $21 \cdot 10^{-5}$ cm/s and $85 \cdot 10^{-5}$ cm/s is {111} (Al) // {211} CuAl₂, <110> (Al) // <210> CuAl₂, whereas in a sample obtained at the rate $333 \cdot 10^{-5}$ cm/s with the relation of planes preserved, a change in directions has been observed, namely <211> (Al) // <102> CuAl₂.

Apart from the above mentioned orientation relationship, there occur also other orientation dependences, especially in the areas of irregular morphology, such as for example {112} (Al) // {212} CuAl₂, <110> (Al) // <120> CuAl₂. However in this case the angular deviations from the ideal relation are greater [9, 10]. The results obtained concerning the orientation relationship and their analysis confirms the literature data. At the same time the application of present-day measurement techniques (EBSD) and X-ray topography enabled their verification and supplementation.

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