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Valerie Brien, V. Khare, F. Herbst, P. Weisbecker, J-B. Ledeuil, et al.. Influence of boron content on the microstructure of sintered $\text{Al}_{62.5}\text{--}x\text{Cu}_{25.3}\text{Fe}_{12.2}\text{B}_x$ alloys ($x = 0, 3, 5$). Journal of Materials Research, 2004, 19 (10), pp.2974-2980. 10.1557/JMR.2004.0366 . hal-02882476

HAL Id: hal-02882476

<https://hal.science/hal-02882476>

Submitted on 1 Jul 2020

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Influence of boron content on the microstructure of sintered $\text{Al}_{62.5-x}\text{Cu}_{25.3}\text{Fe}_{12.2}\text{B}_x$ alloys ($x = 0, 3, 5$)

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(Received 7 May 2004; accepted 16 June 2004)

Microstructures and morphological features of a series of sintered quasicrystalline $\text{Al}_{62.5-x}\text{Cu}_{25.3}\text{Fe}_{12.2}\text{B}_x$ alloys, with x ranging from 0 to 5 at.% were studied using x-ray diffraction, scanning electron microscopy, x-ray mapping, and electron probe microanalysis. Electron backscattering diffraction (EBSD) was also used to get information about the structures of some phases and identify the crystalline relationship in-between phases. Increasing x results in the change of the nature of extra phases. These secondary phases are all less than 1% in volume of the total matter except for the β phase at 5% of boron. Whatever the percentage of boron considered, boron seems to concentrate essentially in the parasite phases confirming doubts found in literature about the solubility of boron inside the face-centered-icosahedral Al–Cu–Fe phase. No special crystallographic relationship in between the tested phases could be spotted. EBSD is thus also confirmed as an excellent technique to get quasicrystalline grains orientations.

I. INTRODUCTION

Quasicrystalline phases were discovered in 1984.¹ The particular feature of this new class of materials is its unique tendency to crystallize according to nonperiodic crystallographic structures (icosahedral, decagonal ...).^{1–3} These structures exhibit a long-range order that is not compatible with translation and impart a brand new combination of properties^{4–14} to the material. This new combination of properties is as new as it is varied, considering they are made of metallic atoms. Transport (thermal, electronic, optic ...), tribological and thermodynamic properties have been widely investigated and found to be very original. Since this is a rather critical point for possible industrial applications, their mechanical properties are also of high interest. Although these quasicrystalline intermetallic compounds can be plastically deformed at high temperature, they are very brittle at room temperature. Their hardness is high and the toughness is rather low (Refs. 15–17 give data for the Al–Cu–Fe phase).

However, some work¹⁸ mentioned that the addition of boron to Al–Cu–Fe icosahedral quasicrystal helps to modify their fragility: while it simultaneously increases hardness values, addition of boron notably improves the fracture toughness of quasicrystalline coatings formed by plasma arc spraying. Different microstructural studies were performed in an attempt to understand such behavior. Sordelet et al.¹⁹ have studied the related microstructures of samples synthesized in a previous work.¹⁸ They confirm observations mentioned by Huang et al.²⁰ Solidification behavior and resulting microstructures strongly depend not only on the addition of boron (even in small amounts) but also on the process. Huang et al.²⁰ tackled the case of melt spun ribbons (annealed or not) whereas Sordelet et al.¹⁹ dealt with chilled cast and high-pressure gas atomized samples. It appears that the appearance of secondary phases is systematic when adding boron to Al–Cu–Fe. It also turns out it is not clear whether boron is soluble in the ternary icosahedral phase or not. The study concerning the effect of a very small addition ($x = 0\%$, 0.5% , 1% , and 2%) on the solidification behavior of melt spun ribbons mentions that if boron has a solubility it should be very small.

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DOI: 10.1557/JMR.2004.0366

More work has then been undertaken here to study the effect of sintering and a presence of boron from 0 to 5 at.% on the morphology and structural modifications. The upper limit of the range was taken as 5 at.% B because it is known that the high-dimensional cubic lattice parameter of the Al–Cu–Fe–B compound decreases linearly with increasing boron concentration in melt-spun ribbons.²¹

The first part of the work focuses on a careful identification of all the phases. The number of phases, their nature, and their morphology versus the increase of the percent of boron of the sintered samples were examined and reported here (Sec. III. A). Indeed, the ultimate goal of this investigation is to see if and how structural and morphological aspects can be linked with the mechanical properties of samples. This work is not presented here and will be the subject of later publications. Within this framework, attention has been taken to localize with precision the chemical distribution of boron (Sec. III. B) and to identify the potential crystallographic relationships between the present phases in samples (Sec. III. C).

II. EXPERIMENTS

Three different alloys having compositions $\text{Al}_{62.5}\text{Cu}_{25.3}\text{Fe}_{12.2}$ (named B0), $\text{Al}_{59.5}\text{Cu}_{25.3}\text{Fe}_{12.2}\text{B}_3$ (named B3), and $\text{Al}_{62.5}\text{Cu}_{25.3}\text{Fe}_{12.2}\text{B}_5$ (named B5) were prepared by melting mixture of the constituent elements in an induction furnace in He atmosphere. These alloy ingots were then subjected to ball milling to achieve the required particle size (25–65 μm) for sintering process. Disks (4 mm thick and 30 mm diameter) of polygrained samples were prepared by this method. Sintering has been performed under Ar in a floating carbon matrix with an external pressure of 10 MPa for 1 h. The sintering temperature was selected after performing differential thermal analysis of the ball milled powder which varies from 750 to 800 °C depending upon the composition.

Scanned electron images with and without mixtures of secondary and backscattered electrons were performed on a Philips FEG XL30G. X-ray mapping was achieved either on a SX50 CAMECA microprobe or on the electron dispersion spectroscopy (EDS) system adapted on a scanning electron microscope (SEM) Philips FEG XL30G. Phase compositions were obtained by electron probe microanalysis (EPMA) on a SX50 CAMECA microprobe. EPMA was also systematically performed to obtain the average composition of all phases found. To permit analysis of the presence of all elements (including the one of boron which is a delicate point) in phases, we used a voltage of 10 kV and an intensity of 15 nA. The SEM-FEG PHILIPS XL30G microscope was equipped with electron backscattering diffraction (EBSD).

EBSD is a technique used extensively nowadays to characterize textures and orientation relationships

between crystals. It can also be used for identifying structures. EBSD performed on a single crystal or grain produces a Kikuchi pattern. The Kikuchi pattern exhibits bands or parallel lines related to each crystallographic plane of the structure of the grain. The band sets exhibit the symmetry of the analyzed crystallographic structure.

FIG. 1. X-ray θ – 2θ diffraction patterns of sintered (a) B0, (b) B3, and (c) B5 samples recorded with $\lambda_{\text{Cu}} \text{K}\alpha$ radiation. Arrows indicate peaks related to the presence of β phase.

Normal orientation of grains can then be determined and two grains exhibiting different orientations simply produce different EBSD patterns. Naturally, “quasicrystallography” is a recent tool studying quasicrystalline structures, and as automatic structural identification software is a recent development; automatic orientation identification of such structures is not yet currently available, except at ETH-Zurich. However, qualitative and point analysis can be attempted. Visual appraisal and comparison with standard Kikuchi maps can be carried out. Recent work obtained by Cheung et al.²² showed it was possible to record EBSD pattern on quasicrystalline structure. The EBSD patterns here were obtained by subtracting from the raw pattern an average background estimated from a statistics of around 12 patterns of the same zone of the same sample.

For the sake of electron images and EBSD results, different states of surface were observed to determine the best final polishing procedure. The mirror polish-state was in most cases the one leading to optimal observa-

tions. Contrast of electron images (mixing or not secondary and back-scattered electrons) is directly linked to the electron density of the material. The lighter the density is, the darker the image is.

III. EXPERIMENTAL RESULTS AND DISCUSSION

A. Nature, morphology of present phases, microstructures

The x-ray diffractograms of the sintered samples are reproduced in Fig. 1. Most of the peaks can be indexed in the face-centered icosahedral (fci) phase. Comparison of the peak positions on the 3 patterns reveals that no shift of lattice parameter can be detected with the introduction of boron. No secondary phase appears except on B5 diffractogram. This second phase can be indexed as the known β phase (CsCl type) $\text{Al}(\text{Cu}, \text{Fe})$ and its peaks are marked by arrows. Table I is a compilation of all the information obtained from the different investigation techniques and provides an overview of results.

TABLE I. Table compiling results concerning the identification of phases.

	BSE	X-ray diffraction	EBSD	Composition (EPMA)	Identification
B0					
$\text{Al}_{62.5}\text{Cu}_{25.3}\text{Fe}_{12.2}$	Matrix Bright phase	fci phase Small traces	Icosahedral pattern Pattern typical of CsCl structure No special relationship with matrix	$\text{Al}_{61.6}\text{Cu}_{25.4}\text{Fe}_{13.0}$ $\text{Al}_{55.9}\text{Cu}_{34.1}\text{Fe}_{9.9}$	fci icosahedral phase β
	Dark zones	Not detected	No difference detectable with matrix	No difference detectable with matrix	Porous zone
B3					
$\text{Al}_{59.5}\text{Cu}_{25.3}\text{Fe}_{12.2}\text{B}_3$	Matrix Needles	fci phase Not detected	Icosahedral pattern Pattern indexable with the orthorhombic Fe_2AlB_2 phase	$\text{Al}_{60.6}\text{Cu}_{26.4}\text{Fe}_{13.0}$ $\text{Al}_{21.8}\text{Fe}_{40.1}\text{B}_{38.1}$	fci icosahedral phase Orthorhombic Fe_2AlB_2 phase
	Globules	Not detected	Geometry and size of particles not adequate to perform the experiment	$\text{Al}_{7.5}\text{B}_{92.5}$	Globules B_{12}Al
	Dark zones	Not detected	No difference detectable with matrix	No difference detectable with matrix	Porous zone
B5					
$\text{Al}_{57.5}\text{Cu}_{25.3}\text{Fe}_{12.2}\text{B}_5$	Matrix Bright phase	fci phase Beta (traces)	Icosahedral pattern Pattern typical of CsCl structure No special relationship with matrix	$\text{Al}_{59.5}\text{Cu}_{28.0}\text{Fe}_{12.5}$ $\text{Al}_{49.6}\text{Cu}_{41.3}\text{Fe}_{9.1}$	fci icosahedral phase β
	Needles	Not detected	Pattern indexable with the orthorhombic Fe_2AlB_2 phase No special relationship with matrix	$\text{Al}_{22.7}\text{Fe}_{38.6}\text{B}_{38.8}$	Orthorhombic Fe_2AlB_2 phase
	Globules	Not detected	Geometry and size of particles not adequate to perform the experiment	$\text{Al}_{7.3}\text{B}_{92.7}$	Globules B_{12}Al
	Dark zones	Not detected	No difference detectable with matrix	No difference detectable with matrix	Porous zone

As it is not possible to detect secondary phases with a volume ratio less than 1% by x-ray diffraction. SEM/back scattered electron microscopy (BSEM) was used to obtain more precise information. A study was carried out as far as possible and as long as necessary by x-ray mapping, EBSD, and EPMA to obtain, respectively, distribution of elements for samples containing boron, structural identification or eventual crystallographic relationship with the surrounding matrix, and compositions of phases.

Figure 2(a) presents a typical electron image recorded on the B0 sample (do not take notice of the pyramidal shape which is the signature of hardness testing for a further study). On top of the matrix, bright zones (of 10 μm size, white dotted arrows) and very dark zones

(D zones, of a few micrometers, indicated by black dotted arrows) can be spotted on top of the matrix. B0 is then not a pure icosahedral sample. A small compositional shift or a shift from the ideal temperature treatment is known to make the β phase appear. A brighter contrast than the matrix contrast level indicates the secondary phase has a larger electronic density than the matrix. This one is compatible with the density of β as it contains more Cu and Fe than the icosahedral matrix. The analysis of the “white” grains by EBSD leads to patterns which are indexable using the CsCl structure confirming that the secondary phase is β . The EBSD experiments also revealed that the grains were monocrystalline. As further polishing does not get rid of the dark zones, shaped like diffuse clouds, they cannot be dirt or local pollution. These zones of a lighter electron density exhibit an EBSD pattern identical to the one of the matrix and no difference of composition with the matrix (EPMA measurement); we can conclude they are due to the localized presence of porosity.

Figure 2(b) presents a typical electron image recorded on the B3 sample. As for the B0 sample, secondary phases can be observed. Porosity can also be spotted (dotted black arrows). The analysis of the D zones by EBSD and EPMA leads to identical results as for the B0 sample and rules out again the possibility of being boron rich zones. These porous zones often contain globules exhibiting a much darker contrast than the matrix suggesting a very light electron density (dark arrows). Most of the globules were found off their location, not polished or sectioned. Moreover, the sample exhibits scratches originating from the same locations. This proves they must be very stable and very hard. EPMA was performed taking into account the round shape of the rare but still present globules. The estimation of their composition gives AlB_{12} . Because of the round shape, no EBSD pattern could be recorded. Indeed the technique requires a planar and well-polished surface. The Inorganic Crystal Structure Database (ICSD) suggests different possibilities for the structure of such a composition: the globules might either be tetragonal (P 422) or monoclinic. From composition determination, it is clear that none of these structures can be an approximant of an Al-based quasicrystalline phase. In accordance with their composition, these globules most presumably correspond to the icosahedral form of boron, stabilized by an aluminum atom sitting in the center of the boron icosahedral clusters. A second phase is present throughout the samples: it is a slightly darker contrast than the quasicrystalline matrix and exhibits a “needle” shape (Fig. 3, white arrows). Its chemical composition was delicate to perform as it underwent a preferential polishing making the needles set back from the surface of the matrix. Moreover, the width of the needles is of the order of magnitude of the size of the EPMA probe (μm). The estimate composition

FIG. 2. Electron images (most of electrons being BSE) of sintered Al-Cu-Fe-B alloys: (a) B0, (b) B3, and (c) B5 samples. White dotted arrows indicate the residual β phase. Black ones indicate globules of phase lighter than the matrix (boron rich). Dotted black arrows indicate darker and diffuse zones of the matrix. White arrows point at the needles. Polishing is SiC paper 4000.

FIG. 3. BSE (top left) and x-ray maps recorded on (a) B3 and (b) B5 samples allowing a clear visualization of the element distribution.

then obtained was Fe_2AlB_2 . The analysis indeed reveals that copper would have no solubility in this phase. EBSD performed on needles proved that they all are monocrystalline. The indexing of the obtained patterns as presented in Fig. 3 was possible only by assuming the $Cmmm$ (65) base-centered orthorhombic Fe_2AlB_2 structure.

Figure 2(c) presents typical images recorded on the B5 sample. The x-ray diffraction pattern allows the

expectation of β presence, which can be identified, under the light of the previous remarks, as being the phase exhibiting a bright contrast [dotted white arrows, Fig. 2(c)]. This was undoubtedly confirmed by indexing the EBSD patterns of these phases using the CsCl structure. Its chemical composition measured by EPMA gave $\text{Al}_{49.6}\text{Cu}_{41.3}\text{Fe}_{9.1}$. Needles, globules and dark zones (respectively white, black and dotted black arrows) similar to the one studied in B0 or B3 samples can be seen. They all lead to similar results in terms of structure or chemistry as in the studies performed for the two other samples. Boron (5 at.%) leads then to the presence of secondary phases: monocrystalline β grains, monocrystalline Fe_2AlB_2 needles, and some porous zones where AlB_{12} globules can be found.

B. Localization of boron

To complete the previous study and, more particularly, the point chemical EPMA analysis, x-ray mapping with the 4 constitutive elements Al, Cu, Fe, and B simultaneously with and electron image (BSE) were recorded in B3 and B5 samples on representative zones (Fig. 4). These maps confirm boron is localized in globules in a high proportion and in the needles in a lower extent. These maps also confirm that the D zones are not boron-rich zones but porous zones, as no difference can be spotted with the matrix. Indeed, the B0 sample exhibits such zones and sintering is a preparation process known to be susceptible to produce porous zones. Detection of boron is very delicate. However, the very limited signals

FIG. 4. Indexed EBSD Kikuchi pattern recorded on a needle in B3 sample. Indexation could be done taking the $Cmmm$ (65) structure, base-centered orthorhombic.

FIG. 5. Results obtained on B3 sample. The top right image is a BSE image showing the studied needle called A (indicated by a white arrow). A is surrounded by 9 icosahedral grains AGi ($i = 1$ to 9). Other images are the EBSD patterns of the AGi quasicrystalline grains

recorded in the matrices of the two samples indicate the Al–Cu–Fe icosahedral compound would have no solubility via this technique for boron. This result is not 100% certain. Indeed, the current limit of detection on these samples was estimated to 1 at.%. On the other hand, boron was shown to be present in secondary phases. The level of composition of boron in AlB_{12} and Fe_2AlB_2 and the quantity of globules and needles observed suggests that very little or no boron is left in the matrix. It is therefore not certain whether boron enters the quasicrystalline structure. Several points establish this doubt. (i) No shift of parameter can be spotted on the x-ray diffraction patterns with increasing percent of boron. (ii) X-ray mapping analysis shows no significant boron signal could be recorded in the matrix of samples containing 3 or 5 at.% boron with the analysis conditions used so far. (iii) B3 ($\text{Al}_{59.5}\text{Cu}_{25.3}\text{Fe}_{13.2}\text{B}_3$) and B5 ($\text{Al}_{62.5}\text{Cu}_{25.3}\text{Fe}_{12.22}\text{B}_5$) samples both contain impurity phases other than the quasicrystalline one that all contain boron. Indeed B3 and B5 samples contain AlB_{12} globules. (iv) B3 and B5 contains Fe_2AlB_2 needles. All these

results support the idea that the solubility of boron in the icosahedral phase is non-existent or very small at (or close to) the thermodynamic equilibrium.

C. Orientation relationship between secondary phases and surrounding quasicrystalline grains

EBSD was attempted on the B3 SiC 4000 paper polished sample, and no pattern could be recorded coming from the needles. This was certainly due to work-hardening. Mirror polishing was then performed on all samples (carbon diamond paste, colloidal silica). Figure 5 gives the EBSD patterns of a needle and its surrounding quasicrystalline grains. As the available EBSD commercial indexing systems are programmed to index exclusively periodic crystals, no indexing of the icosahedral phases can automatically be performed yet. However, a glance at all these patterns shows they are all different and so shows the surrounding grains exhibit as many different orientations as they are in number. This leads us to conclude that no specific relationship between the needle and the icosahedral grains can be spotted. We found this result can be reproduced whatever the percent of boron is. Moreover, in the same way as for the needles, no special crystallographic relationship can be noticed between the β grains and their surrounding quasicrystalline grains whatever the percent of boron is. One recalls that no EBSD pattern could be recorded on the AlB_{12} globules because of their shape and size.

IV. CONCLUSION

A study looking at the influence of addition of boron on the microstructures of sintered Al–Cu–Fe–B samples has been carried out. Identification of the nature (composition–crystallographic structure) of constituting phases, their morphology, and their eventual relationship with the quasicrystalline matrix has been carried out. Boron (3% and 5%) leads to icosahedral material containing extra phases. The sample containing 3% boron exhibits AlB_{12} globules and Fe_2AlB_2 needles.

The sample containing 5% boron contains AlB_{12} globules, Fe_2AlB_2 needles, and β grains. The volume of these extra phases (except for the β phase at 5%) is less than 1%.

The Fe_2AlB_2 needles and the β grains do not have specific crystallographic relationship with their surrounding quasicrystalline grains.

The study also stresses the success of the EBSD technique for obtaining the grain orientations of quasicrystalline grains. Sintering does not seem to favor a significant solubility of boron in the quasicrystalline structure.

ACKNOWLEDGMENT

The authors wish to acknowledge the European Commission Growth 2000 Project No. G5RD-CT-2001-

00584 entitled Smart Quasicrystals for subsidizing the research.

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