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Intermetallic Texture Analysis by X-Ray, Neutron and Electron Backscattered Diffraction

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Abstract. Texture characterization of intermetallic materials implies to solve the problem of overlapped pole figures of one or several phases when they are carried out by X-ray or neutron diffraction. Moreover, if the harmonic method is used to calculate the texture function, a complementary iterative method must be introduced to define the total texture function. A second iterative method must still be used to solve the problem of incomplete pole figures measured by X-ray diffraction in reflection. An other approach consists in using individual orientation measurements, which allow to estimate the local texture which can be correlated to the microstructure but also to the global one if the orientation number is sufficient.

1. INTRODUCTION

Yamaguchi and Inui [1] showed that mechanical properties of the titanium aluminide (TiAl) strongly depend of the texture. So, it becomes useful to characterize quantitatively the texture using the Orientation Distribution Function (ODF).

The global (X-ray or neutron) and local (EBSD [2]) texture analyses are compared and discussed enlightening their respective advantages and disadvantages. So, when the ODF is calculated from pole figures measured by X-ray or neutron diffraction, it is necessary to separate the (002)/(200) ... overlapped reflections of the tetragonal phase and eventually the overlapping of some TiAl and Ti_3Al (hexagonal phase) reflections in the case of γ/α_2 aluminides [3]. On an other hand, to calculate the ODF from individual orientations, it is necessary to know the minimum orientation number to obtain a statistical ODF comparable to that calculated from pole figures.

The global texture analysis assumes the homogeneity of the microstructure because only 3 orientation parameters are measured knowing that a grain in a polycrystal is defined by 6 parameters. The 3 other parameters (or 2 in the case of a two dimensional analysis) correspond to the grain location parameters in the polycrystal that can only be defined from a local technique of orientation measurements.

2. ODF ANALYSIS

To calculate the ODF $F(g)$ which corresponds to the probability to find grains with an orientation g within dg in the studied material, the harmonic method [4,5,6,7,8] is used and is described using the Roe formalism. From a global or a local analysis, the next equation must be solved :

$$F(g) = \sum_l \sum_m \sum_n f_{lmn} T_{mn}^l(g) \quad (1)$$

$F(g)$ is expressed in the Euler space; g is an orientation characterized by ψ, θ, ϕ (Roe notation) or ϕ_1, ϕ, ϕ_2 (Bunge notation); T_{mn}^l are spherical harmonic functions and f_{lmn} are the ODF coefficients.

2.1 X-ray or neutron diffraction

The equation which links the pole densities $q(\eta, \chi)$ (normalized diffracted intensities at a point defined by the η and χ spherical coordinates on the $\{hkl\}$ pole figure) to the ODF must be solved :

$$q(\eta, \chi) = \frac{1}{2\pi} \int_0^{2\pi} F(g) d\gamma \quad (2)$$

Where $d\gamma$ is the differential element of a rotation around the normal of the diffracting plane.

The two terms of this equation are thus expanded on the spherical harmonic basis. For the second term of equation (2), one finds equation (1) and for the first term of equation (2), one obtains :

$$q(\eta, \chi) = \sum_l \sum_m Q_{lm} Y_l^m(\eta, \chi) \quad (3)$$

After integration of $F(g)$ (equation (1)), the new obtained expression is identified to equation (3) allowing to define a relation between the Q_{lm} and f_{lmn} coefficients, so :

$$Q_{lm} = 2\pi \left(\frac{2}{2l+1} \right)^{1/2} \sum_n f_{lmn} Y_l^n \quad (4)$$

So, the f_{lmn} coefficients corresponding to a set of experimental pole figures (the pole figure number defines the expansion order L) can be calculated. From the f_{lmn} coefficients, the ODF can be thus calculated (equation (1)). Moreover, the pole densities corresponding to experimental pole figures or not can also be recalculated (equations (4) and (3)).

2.1.1 Incomplete pole figures

When the pole figures are measured by X-ray diffraction in reflection, the periphery of these pole figures cannot be determined because of the defocusing problem. Consequently, it becomes necessary to introduce a new mathematical approach which allows one to complete the pole figures. For that, an iterative method that takes into account the positivity condition of pole densities can be used (see [9,10] ; the method described in reference [9] is used in this study).

2.1.2 Overlapped pole figures [9,11]

If the j index defines a phase, for the i overlapped pole figures of the j phases, one obtains :

$$q_\theta(\eta, \chi) = \sum_{j=1}^J \sum_{i=1}^I \alpha_{ij} q_{h_{ij}}(\eta, \chi) \quad \left(\text{with } \sum_{j=1}^J \sum_{i=1}^I \alpha_{ij} = 1 \right) \quad (5)$$

Taking into account the relation between $q(\eta, \chi)$ and Q_{lm} (equation (3)), one obtains :

$$Q_{lm}(\theta) = \sum_{j=1}^J \sum_{i=1}^I \alpha_{ij} Q_{lm}(h_{ij}) \quad (6)$$

Consequently, equation (4) becomes :

$$Q_{lm}(\theta) = 2\pi \left(\frac{2}{2l+1} \right)^{1/2} \sum_{j=1}^J \sum_{n=0}^{N(l,j)} f_{lmn}(j) \sum_{i=1}^{I(j)} \alpha_{ij} Y_l^n^* (h_{ij}) \quad (7)$$

Where the f_{lmn} and the α_{ij} are the unknowns. To simplify the resolution, at the initial state (iteration N°0), the α_{ij} values are assumed to be known (so for example, if two pole figures are overlapped then $\alpha_1 = \alpha_2 = 0,5$). Thus, it is easy to solve equation (7) because it is the same that equation (4) where only the f_{lmn} coefficients are unknown. From the f_{lmn} coefficients, the Q_{lm} corresponding to the non overlapped pole figures can be recalculated and compared to the experimental overlapped ones. This comparison which uses a mean square method allows one to find new α_{ij} values to continue the iterative calculation (equation (7)) until the convergency of the α_{ij} values is obtained.

2.1.3 Total texture function

The global texture analyses do not permit to separate the $+h_i$ et $-h_i$ directions of centrosymmetrical crystals [6]. In these conditions, it appears a mathematical indetermination since only the even part $\tilde{F}(g)$ (l even) can be calculated from pole figures. To assess the odd part $\tilde{\tilde{F}}(g)$ and so the total ODF, it becomes necessary to introduce a new iterative procedure that allows one to estimate $F(g)$ from $\tilde{F}(g)$ using the positivity condition of $F(g)$ ($F(g) = \tilde{F}(g) + \tilde{\tilde{F}}(g) \geq 0$).

2.1.4 Example

To sum up, in the more defavourable case, to calculate the ODF, it is necessary to use the harmonic method plus 3 iterative methods to solve the problems of incomplete and overlapped pole figures and to calculate the total ODF. To illustrate this approach a theoretical two phases (tetragonal and hexagonal) material is defined. Each phase is characterized by one orientation that is modelled with a gaussian function (see section 2.2). This approach allows one to construct pole figures :

*Tetragonal phase : 7 pole figures ($\psi=22,67$, $\theta=56,14$, $\varphi=26,57$ with $c/a=2,0$, $\phi_0=15^\circ$ and $L=22$)

*Hexagonal phase : 6 pole figures ($\psi=\theta=\varphi=0,00$ with $c/a=1,6$, $\phi_0=25^\circ$ and $L=22$).

Then to assume X-ray measurements, the periphery (10°) of pole figures is suppressed and finally, the $\{110\}$ and $\{10\bar{1}0\}$ pole figures are numerically overlapped. Table 1 allows one to compare the given values to those calculated with the so described approach and shows the quality of this method.

α values		Tetragonal phase $\{110\}$	Hexagonal phase $\{10\bar{1}0\}$
	Given	0,7000	0,3000
Maximal values of $\tilde{F}(g)$	Calculated	0,6998	0,3002
	Local analysis	66,6	13,3
Maximal values of $F(g)$	Global analysis	66,7	13,2
	Local analysis	97,4	14,6
	Global analysis	97,0	14,4

Table 1: Comparison of theoretical and calculated values of α , $\tilde{F}(g)$ and $F(g)$

2.2 EBSD

The f_{lmn} coefficients can be calculated from individual orientations, each orientation being modelled with a gaussian function [12] :

$$f_{lmn} = \frac{1}{N} \sum_{i=1}^N K T_{mn}^{l*}(g_i) \quad (8)$$

K defines the gaussian function where the ϕ_0 parameter characterizes the width at half height of the gaussian. Obviously, the main difficulty of this approach consists to choose the right number of orientations to calculate a statistical ODF i.e. comparable to the ODF calculated from pole figures measured for example by X-ray diffraction [13,14].

When this kind of local or global analysis is used, the microstructure is assumed to be homogeneous because only the 3 orientation parameters are taken into account. Only a local approach allows one to define the 6 (or 5 in the case of a two dimensional analysis) parameters allowing to weight each orientation by the grain area measured by image analysis or defined by an orientation number measured in the grain if the local measurements are performed on a defined grid. This last approach can be used when the EBSD is automatized (stage displacement and Kikuchi pattern indexation). This technique allowing to built up the studied microstructure is named Orientation Imaging Microscopy (OIM) [15]. The orientations are measured at each point of a XY grid ($\Delta X = \Delta Y > 0,1\mu m$). Then knowing that if two consecutive points have the same orientation they are in the same grain and on the contrary if the orientations are different a grain boundary separates the two points, it becomes easy to built up the grey level microstructure as a function of the Kikuchi pattern quality (white for beautiful patterns and black for poor patterns) or the color microstructure, each grain being colored according to the crystallographic orientation [16].

3. GLOBAL TEXTURE OF A TITANIUM ALLOY (50% α / 50% β)

The used titanium alloy was annealed to obtain a "school" sample with two cubic and hexagonal phases having approximatively the same volume fraction. The texture of the two phases is characterized by X-ray diffraction in reflection and by EBSD. In the first case, 7 incomplete pole figures (10° at the periphery are not determined) have been measured knowing that the $\{110\}$ and $\{0002\}$ and the $\{211\}$ and $\{01\bar{1}3\}$ pole figures are overlapped. The ODF of each phase is calculated with $L=34$. In the second case, a square surface has been studied ($X=Y=200\mu m$; $\Delta X=\Delta Y=1\mu m$). Figures 1a and 1b show respectively the experimental microstructure with α nodules in the β matrix and the microstructure built up from orientation measurements. In this last case, it is interesting to note that the volume fraction of cubic (grey) and hexagonal (black) phases have been found equal to 46 % and 54 % so very closed to the imposed volume fraction according to the heat treatment. One can observe that the grain boundaries are not perfectly define because the exploration step was too large. Indeed, to improve the microstructure definition (that is not the aim here) it is necessary to decrease the grid step.

With the EBSD, it becomes easy to separate the two phases and so to calculate the corresponding ODFs. Figures 2a and 2b show the $\phi_1=90^\circ$ constant section plot of the ODF calculated for the cubic phase from pole figures measured by X-ray diffraction and from about 11200 individual orientations ($\phi_0=10^\circ$, $L=34$) respectively.

The obtained results (main component near $\{130\}<001>$) are quite similar even if some differences appear firstly due to the sample location during the experiments and secondly to the choice of ϕ_0 [14]. Moreover, it should be interesting to verify if the orientation number or more exactly the grain number is sufficient to calculate an ODF, knowing that it appears some microstructural heterogeneities on figure 1b.

Figure 1: (a) experimental microstructure and (b) built up microstructure from EBSD

Figure 2: $\phi_1=90^\circ$ constant section plot of the ODF calculated (a) from X-ray diffraction and (b) from EBSD

4. LOCAL TEXTURE OF THE Ti48Al48Cr2Nb2 ALLOY

This material so called GE (General Electric) provided by ONERA is particularly studied by Dupont et al. [17]. Here, it is studied after a heat treatment (1150°C-24 hours). The texture measured on the tetragonal phase (the volume fraction of the hexagonal phase is too low (about 5%)) by neutron diffraction (because of the large grain size) shows a $\{111\}\langle uvw \rangle$ fiber texture (the fiber axis is perpendicular to the ingot axis). Figure 3a shows the microstructure ($X=Y=350\mu\text{m}$; $\Delta X=\Delta Y=1\mu\text{m}$) built up from individual orientations and figure 3b shows that the crystallographic planes of lamellae sets (1 and 2) and twinned monolithic grain (about 23% of the volume fraction of the sample) (3 and 4) are near $\{111\}$ (see [17]).

Figure 3: (a) built up microstructure and (b) $\{111\}$ pole figure corresponding to some parts of microstructure (a)

5. CONCLUSION

The present study shows the advantages and the disadvantages of the global and local texture analyses in particular for intermetallic materials and in a general case for all multiphase materials. However, as it was described, all the disadvantages can be solved, and consequently it becomes difficult to choose the best method. In fact and in a general case, except for material with large or very large grains for which the X-ray and neutron cannot be used respectively, the two approaches are complementary.

In the case of EBSD measurements, the most important problem concerns the choice of the sufficient orientation number to calculate the ODF and it depends on the experimental procedure. Indeed, in a first approach, the orientations can be randomly measured on the surface analysed by X-ray diffraction and in a second approach, only a part of the microstructure can be analysed and in this last case, it is necessary to verify that the observed domain is representative of the total surface studied by X-ray diffraction.

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