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Determination of Single-Crystal Elasticity Constants in a cubic phase within a multiphase alloy - X-Ray Diffraction measurements and inverse scale transition modelling

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Synopsis

A new approach is developed in order to determine single-crystal elasticity constants of a cubic phase from the analyse of a multiphase polycrystal. This inverse method is based on the coupling of a three scale self-consistent micro-mechanical model with diffraction lattice strains measurements involving in-situ pure elastic loading.

Abstract

The scope of this work is the determination of single-crystals elastic properties from X-ray diffraction stress analysis performed on multi-phase polycrystals. An explicit three scales multi-phases inverse self-consistent model is developed in order to express the single-crystal elasticity constants of a cubic phase as a function of its X-ray elasticity constants. The model is checked on the case of single-phase materials. Finally, it is applied to a two-phases ($\alpha+\beta$) titanium based alloy (Ti-17). And, as a result, the Ti-17 β -phase single-crystal elasticity tensor is estimated.

Keywords

Single-Crystal Elasticity Constants, X-ray Elasticity Constants, multi-phases materials, titanium alloy, Self-Consistent scale transition model, inverse method.

1. Introduction

The precise identification of Single-crystal Elasticity Constants (SEC) is important in the field of engineering applications of materials. Actually, these constants are necessary to predict the micro-mechanical behaviour of polycrystalline materials. For example, SEC knowledge is required to estimate the X-Ray Elasticity Constants (XEC) involved in X-ray diffraction stresses analysis methods widely used in both fundamental research and industry (the interested reader can refer to the proceedings of both the International and European Conference on Residual Stresses: ICRS and ECRS).

SEC characterization usually requires a sufficiently large single-crystal (several millimetres). This type of sample enables the use of classical methods such as analyzing the ultrasonic

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waves velocities (Nye, 1957). Many single crystals have been studied by this technique. See for instance a synthesis by Chung and Buessem (1967). Tanaka and Koiwa (1996) described an alternative method, the Rectangular Parallelepiped Resonance (RPR), especially suitable for intermetallic compounds. But, RPR only applies to single-crystals (relatively smaller than for the classical ultrasonic method: 2 mm in length). Nevertheless, many new advanced materials are difficult to obtain in an appropriate single-crystal form. It is the case of numerous multi-phases alloys such as ($\alpha+\beta$) titanium alloys. In these circumstances, SEC determination methods described above are no more suitable.

Owing to X-ray diffraction techniques, single-crystals elastic properties can be evaluated from the mechanical macroscopic stiffness and X-Ray Elasticity Constants measured on polycrystals. XEC represents the proportionality constants relating XRD strains measurements to the residual stress state of the diffracting phase (Hauk, 1997). These constants express the mechanical stiffness of the diffracting volume of the probed phase. Thus, they are strongly related to the SEC of the crystallites of this particular phase. The procedure was first introduced for single-phase polycrystals by Hauk and Kockelmann (1979): experimental XEC are fitted using a micromechanical model (that can be either based on Reuss (1928), Voigt (1929) or Kröner (1958) formalisms, for example). Within this framework, the unknown SEC constitutes parameters whose numerical values are adjusted in order to optimise the accordance between experiments and simulations. The method was applied to the case of cubic crystals by Hayakawa et al. (1985). More recently, this approach was extended to hexagonal symmetry single phases (Bruno and Dunn, 1997; Matthies et al., 2001).

However, no satisfactory method has been yet proposed to deal with the question of SEC determination from measurement performed on multiphase materials. Effectively, several papers demonstrated the deviation existing between single-phase XEC and those of the same phase when it is embedded in multi-phase materials (Behnken and Hauk, 1990; Tanaka et al.,

2000; Fréour et al., 2003). This property of multiphase materials should also be properly taken into account for an accurate estimation of the SEC from the experimental study of such materials. The present paper details an inverse method based on the classical Kröner (1958) and Eshelby (1957) Self-Consistent (KESC) elastic model. The second part of this article aims at finding explicit forms for the SEC of cubic crystals using this formalism. The basic idea of this work is the improvement of the classical KESC scheme in order to take into account the effects of the presence of a second phase on the mechanical behaviour of the diffracting volume of the studied phase. In section 3, SEC calculated according to the present work from XEC measurements in single-phase polycrystal will be compared to results available in the literature, in order to evaluate the performance of this new approach. Then, in the last part, this new approach will be applied to the determination of β -phase SEC in an ($\alpha+\beta$) titanium based alloy having applications in aeronautic field: Ti-17.

2. Application of Kröner and Eshelby Elastic Self-Consistent model to the case of multi-phases materials

2.1 Introduction – the main assumptions

The inclusion theory of Eshelby (1957) is one of the most applied theoretical methods to simulate the multi-scale behaviour of multi-phases micro-heterogeneous materials. It is often implemented into the polycrystal self-consistent scheme proposed by Kröner (1958). The resulting model is known as Kröner and Eshelby Self-Consistent model (KESC). As part of the model, ellipsoidal inclusions (or crystallites, assumed to behave like single-crystals), are one after another embedded in an infinite Homogeneous Equivalent Medium (HEM) which behaviour is that of the polycrystal.

This approach can be used in order to predict both the elastical anisotropic mechanical states at grain scale (the so-called mesoscopic scale) and the effective properties and mechanical states of the HEM (macroscopic scale) (Kocks et al., 1998). Let us consider the case of multiphases materials. The initial two-scales scheme is insufficient to satisfactorily describe the effective elastic behaviour of a given phase. In consequence, a third, intermediate, scale was introduced by Sprael so as to investigate the localization of strains and stresses in each phase of the polycrystal. This additional level is defined as the pseudo-macroscopic scale (Sprael, 1996). Its effective properties (for example, the pseudo-macroscopic elastic stiffness) and mechanical states can be calculated from homogenization over all the crystallites of the studied phase. Examples have been recently published in the literature (Fréour et al., 2002 ; Fréour et al., 2003).

The stress and strain in a given α phase is defined as the average on the phase of the stress and strain tensors over all the crystallites constituting the phase:

$$\sigma_{\alpha} = \langle \sigma_{\Omega\alpha} \rangle_{V_{\alpha}} \quad \text{and} \quad \varepsilon_{\alpha} = \langle \varepsilon_{\Omega\alpha} \rangle_{V_{\alpha}}$$

where index Ω denotes the crystallographic orientation of the crystallite. The “pseudo-macroscopic” stiffness links these two quantities and it is different from the macroscopic stiffness, as was shown in a previous work (Fréour et al., 2003). The term “pseudo-macroscopic” is commonly used in the field of diffraction stress analysis because the volume on which it is defined is of the same magnitude than the macroscopic scale but it is not truly macroscopic.

The basic assumptions of KESC model are the following: it only applies to purely elastic loads; it supposes that the elementary inclusions are randomly distributed within the matrix, and finally, it is restricted to ellipsoidal-shaped crystallites. Moreover, it neglects direct interactions between the crystallites.

The present study aims to deduce SEC from XEC measurements performed by X-ray diffraction, using closed-form solutions compatible with KESC model. Since X-ray analyses are employed for comparison with the model, it is necessary to use an extension of the classical KESC scheme enabling the proper simulation of the diffracting volume of the material. The main equations involved in this model are extensively detailed in (Fréour et al., 2003). This formalism was especially designed for XEC calculations in the case of materials constituted of spherical grains and being isotropic at macroscopic scale.

In the following section, the explicit relations existing between SEC and XEC calculated by KESC model will be given.

2.2 Inverse closed-form solution obtained for SEC of the diffracting phase of a quasi-isotropic multi-phases material

In fact, it is possible to write explicit forms for the XEC of a cubic phase embedded in a multi-phases material, using KESC model. This model is based on the mathematical treatment of the elastic interaction between ellipsoidal inclusions and the Homogeneous Equivalent Medium. Into this aim, the theory of Eshelby (Eshelby, 1957) has to be used in order to take into account the elastic interactions related to the grain morphology. The calculation of the required Eshelby tensor generally involves numerical integrations. This numerical treatment would, however be incompatible with the idea of finding analytical solutions. The development of such close-form solutions thus implies an additional assumption on the shape of the elementary inclusion. According to the literature, fully analytical forms of Eshelby tensor have been worked out only in the cases that either spherical, disc- or fiber-shaped grains were embedded in elastically *isotropic* media at macroscopic scale (Mura, 1987; Qiu and Weng, 1991). Nevertheless, in general cases, the macroscopic elastic behaviour of a polycrystalline material constituted of disc- or fiber-shaped inclusions does not remain

isotropic (for a recent detailed analysis of this question, see Koch et al., 2004). Accordingly, only polycrystals constituted by an assembly of spherical grains can be treated through a fully analytical version of KESC model. The explicit equations obtained for XEC of a cubic phase included in a multi-phases material are too lengthy to be developed in the present paper. The complete demonstration is available in (Fréour, 2003). In the model, for the above detailed reasons, the crystallites were considered as spherical.

Analytical forms for XEC can be inverted in order to express the SEC of the diffracting cubic phase. It can be shown (Fréour, 2003) that, the three researched components of the mesoscopic elasticity tensor of any α -phase (c_{11}^α , c_{12}^α and c_{44}^α) verify:

$$c_{11}^\alpha = \frac{N_1}{D_1} \quad (1)$$

$$\text{with } N_1 = N_{11} + N_{12} + N_{13} \quad (2)$$

$$N_{11} = C_{11}^2 \left(7 + 7(C_{12}^\alpha - C_{11}^\alpha) \frac{1}{2} S_2^\alpha(\{h00\}) \right) \quad (3)$$

$$N_{12} = C_{12} \left(-C_{11}^{\alpha 2} \frac{1}{2} S_2^\alpha(\{h00\}) + C_{11}^\alpha \left((2C_{12} - C_{12}^\alpha) \frac{1}{2} S_2^\alpha(\{h00\}) - 2 \right) - 2(C_{12} - C_{12}^\alpha) \left(1 + C_{12}^\alpha \frac{1}{2} S_2^\alpha(\{h00\}) \right) \right) \quad (4)$$

$$N_{13} = C_{11} \left(4C_{11}^{\alpha 2} \frac{1}{2} S_2^\alpha(\{h00\}) + C_{11}^\alpha \left(8 + (5C_{12} + 4C_{12}^\alpha) \frac{1}{2} S_2^\alpha(\{h00\}) \right) - (5C_{12} + 8C_{12}^\alpha) \left(1 + C_{12}^\alpha \frac{1}{2} S_2^\alpha(\{h00\}) \right) \right) \quad (5)$$

$$D_1 = 3(4C_{11} - C_{12})(C_{11}^\alpha - C_{12}^\alpha) \frac{1}{2} S_2^\alpha(\{h00\}) \quad (6)$$

$$c_{12}^\alpha = \frac{1}{2} (C_{11}^\alpha + 2C_{12}^\alpha - c_{11}^\alpha) \quad (7)$$

$$c_{44}^\alpha = \frac{N_2}{D_2} \quad (8)$$

$$\text{with } N_2 = N_{11} + N_{22} + N_{23} \quad (9)$$

$$N_{22} = -2C_{12} \left(C_{11}^\alpha + C_{12} - C_{12}^\alpha + (C_{12}C_{12}^\alpha - C_{12}C_{11}^\alpha) \frac{1}{2} S_2^\alpha(\{hhh\}) \right) \quad (10)$$

$$N_{23} = C_{11} \left[-5C_{12} - 8C_{12}^{\alpha} - 5C_{12}C_{12}^{\alpha} \frac{1}{2} S_2^{\alpha}(\{hhh\}) + C_{11}^{\alpha} \left(8 + 5C_{12} \frac{1}{2} S_2(\{hhh\}) \right) \right] \quad (11)$$

$$D_2 = 4(4C_{11} - C_{12})(C_{11}^{\alpha} - C_{12}^{\alpha}) \frac{1}{2} S_2^{\alpha}(\{hhh\}) \quad (12)$$

The listed above equations requires the knowledge of:

- the macroscopic elasticity constants of the multi-phases material (C_{11} and C_{12}),
- α -phase pseudo-macroscopic elasticity constants (C_{11}^{α} and C_{12}^{α}), that are in most cases very close to the ones of the corresponding single α -phase, according to (Fréour, 2003; Fréour et al., 2003),
- and XEC of the analyzed phase for the diffracting planes $\{h00\}$ and $\{hhh\}$ ($\frac{1}{2} S_2^{\alpha}(\{hhh\})$ and $\frac{1}{2} S_2^{\alpha}(\{h00\})$).

In fact, equations (1-12) could be expressed as a function of a XEC measured for any diffracting plane $\{hkl\}$ (and thus, not only for $\{hhh\}$ and $\{h00\}$), using the linear relation demonstrated by Bollenrath, Hauk and Müller (1967) (actually, this equation was established in the case of single-phase materials only, but numerical computations shows that it is still satisfied for multi-phases polycrystals):

$$\frac{1}{2} S_2^{\alpha}(\{hkl\}) = \frac{1}{2} S_2^{\alpha}(\{h00\}) - 3\Gamma(\{hkl\})S \quad (13)$$

Where the slope S and the cubic orientation parameter Γ are respectively given by:

$$S = \frac{1}{2} S_2^{\alpha}(\{hhh\}) \left[\frac{\frac{1}{2} S_2^{\alpha}(\{h00\})}{\frac{1}{2} S_2^{\alpha}(\{hhh\})} - 1 \right] \quad (14)$$

$$\Gamma(\{hkl\}) = \frac{h^2 k^2 + h^2 l^2 + k^2 l^2}{(h^2 + k^2 + l^2)^2} \quad (15)$$

(13-15) could be introduced in (1-12) in order to obtain a generalized form for α -phase SEC.

It was demonstrated in (Fréour et al., 2003) that the pseudo-macroscopic elastic properties were not strictly the same than the ones of the corresponding single-phase. This was attributed to the existence of elastic heterogeneities between the phases and the HEM. Nevertheless, numerical simulations showed that the deviation between pseudo-macroscopic and single-phases stiffnesses components was almost always weaker than 5%. According to these results, it is often justified to introduce single-phase stiffnesses in (1-12) in order to estimate their SEC.

In the case of pure single-phase material, the method remains valid and only involves the replacement of components C_{11} and C_{12} by C_{11}^{α} and C_{12}^{α} . In these circumstances, C_{11}^{α} and C_{12}^{α} obviously define the macroscopic behaviour of the polycrystal, since the macroscopic and pseudo-macroscopic scales are by definition identical in any single-phase material.

Moreover, if the (single-phase) material is isotropic at any scale (both phase and single-crystals are considered as isotropic), then the dependency of XEC on the diffracting plane falls and $\frac{1}{2}S_2^{\alpha}(\{hkl\}) = \frac{1}{2C_{44}^{\alpha}} = \frac{1}{C_{11}^{\alpha} - C_{12}^{\alpha}}$. It is also easy to check the reliability of equations

(1-12) in this simple case:

$N_{11} = 0$, $N_{12} = -3C_{11}^{\alpha}C_{12}^{\alpha}$, $N_{13} = 12C_{11}^{\alpha 2}$ and $D_1 = 3(4C_{11}^{\alpha} - C_{12}^{\alpha})$, so that one obtains the expected relation $c_{11}^{\alpha} = C_{11}^{\alpha}$. Introducing this form in (7) yields $c_{12}^{\alpha} = C_{12}^{\alpha}$. Moreover, $N_{22} = 2C_{12}^{\alpha}(C_{12}^{\alpha} - C_{11}^{\alpha})$, $N_{23} = 8C_{11}^{\alpha}(C_{11}^{\alpha} - C_{12}^{\alpha})$ and $D_2 = 4(4C_{11}^{\alpha} - C_{12}^{\alpha})$. Thus, the last SEC is $c_{44}^{\alpha} = \frac{C_{11}^{\alpha} - C_{12}^{\alpha}}{2}$ that is the appropriate value for an elastically isotropic cubic single crystal.

3. Checking the model: application to single-phase materials

In order to check the validity of equations (1-12) and the overall inverse method, well documented values given in the literature were used. The input values of XEC were taken

from (Gnäupel-Herold et al., 1998) (see table 1). Table 2 indicates the XEC recalculated for {h00} and {hhh} lines, according to a linear regression using equation (13). The computation of the residuals of the least-square regression enabled the determination of the standard deviation associated with XEC mean values. The average elasticity constants of the phases were taken in *goodfellow database* (cf. table 3). Y and ν respectively denote Young's modulus and Poisson's ratio. The stiffness tensor components required for the numerical simulation were deduced from Y and ν using the following relations:

$$C_{11} = \frac{\nu Y - Y}{2\nu^2 + \nu - 1} \qquad C_{12} = -\frac{\nu Y}{2\nu^2 + \nu - 1} \qquad (16)$$

The standard deviation on the stiffness components was deduced from the classical method involving partial differentials. For this purpose, the relative error on the experimental values of Y and ν were assumed to be 2% (this relative error is advocated by Lemaitre and Chaboche, 1985).

The calculation led to the SEC of the materials, which are reported in table 4 and compared to the reference data from (Chung and Buessem, 1967). The errors on SEC were calculated using the relation of propagation of uncertainties (Lira, 2002). The quality of the model can be assessed by observing the deviation ratios given in table 4. The results obtained with the proposed method show a quite satisfactory agreement with experiments. This study demonstrates the reliability of the closed-form solution for the SEC and deduced from KESC model.

Now, the results and accuracy of this new method are comparable to those obtained through other methods, especially those based upon Kröner's model (see for example Gnäupel-Herold et al., 1998 where a comparative study of the SEC estimation methods described in the works of Bollenrath, Hauk and Müller (1967) or de Wit (1997) is done and show a good agreement

with the estimations given in the present work, which uses the same experimental results as input).

The main improvement of the present approach will be explained in the following section, where it will be used to solve the question of the determination of SEC of a phase being not available either as single-crystal or single-phase polycrystal.

4. Characterization of Ti-17 β -phase SEC

4.1 Introduction - the material

Ti-17 is an ($\alpha+\beta$) titanium alloy having primary applications in turbine engine due to its high specific strength (Donachie, 2000). Hexagonal α -phase is stable at low temperature. Its SEC are well known, because this phase is easy to separate from β . On the opposite, cubic β -phase is only stable at high temperatures, when no alloying element is present. A very high quantity of β -stabilizers alloying elements is necessary to obtain a fully β -structured titanium alloy at room temperature (Donachie, 2000). Consequently, the elasticity constants of β -Ti were only determined at grain (Reid et al., 1973) or phase scale on fully β -alloys (Kuroda et al., 1998; Niinomi, 1998). Nevertheless, results published by Kuroda et al. (1998) underline that β -Ti macroscopic elasticity constants depend on the nature and the concentration in β -stabilizers alloying elements (for instance, according to (Kuroda et al., 1998), Young's modulus Y is about 50 MPa in Ti-16Nb-13Ta-4Mo, when it goes up to 70 MPa in Ti-29Nb-13Ta-6Sn, in samples exhibiting similar microstructures: grain size and morphologies). The coefficient of anisotropy A_c of the single crystals determined in some β -Ti alloys is rather small (weaker than 1.8, according to table 8), thus texture related effects are not sufficient to explain such a high relative deviation between Young's modulus of pure β -Ti alloys. Macroscopic elasticity constants being strongly related to SEC, this result entails a variation of β -Ti SEC as a

function of the above-cited parameters. All this highlights the necessity to determine Ti-17 β -phase SEC directly on the two-phases polycrystal, using the formalism described in section 2, rather than to identify these constants to the values available, for example in (Reid et al., 1973. These results are reproduced in table 8).

The following paragraph deals with Ti-17 microstructural, macroscopic and pseudo-macroscopic elasticity properties. The agreement between them and the basic assumptions considered in our model will be emphasized.

4.2 Microstructural and pseudo-macroscopic properties of Ti-17

4.2.1 Morphological properties

The studied sample exhibits the classical highly β stabilized (quasi- β) microstructure, as shown on the micrographs (figure 1). The structure consists of acicular α (platelets) mixed to slightly equiaxed prior β grains. It is well known that the homogenised properties of the polycrystal remain isotropic in the case that non-spherical grains have their principal axes statistically oriented (Qiu and Weng, 1991). The effective behaviour is then comparable to the mechanical behaviour of the same material constituted of spherical grains. α -phase morphology is thus compatible with the fundamental assumptions used in the analytical version of KESC model developed in section 2.2. Accordingly, this model can be considered as suitable to apply to the studied Ti-17 alloy.

4.2.2 Determination of the volume fraction of each phase in Ti-17

Phase quantification was realised on two Ti-17 samples. The first one is a polycrystalline material and the second one a powder obtained from the same polycrystal. Quantitative phase analyses were performed using conventional X-ray powder diffraction method (Bish and Post, 1989) and the procedure is detailed in (Shang et al., 2001) for two-phases titanium alloys. The

volume fractions f^α and f^β of the phases were calculated from one plane family of each phase:

$\{200\}_\beta / \{10\bar{1}2\}_\alpha$. Texture effects were assumed to be negligible in the case of the massive polycrystal. One example of obtained diffraction patterns in the concerned range is given on figure 2.

The volume fractions deduced from these experiments are given below. Powder sample: $f^\beta = 33\%$ and $f^\alpha = 67\%$. Massive polycrystalline sample: $f^\beta = 30\%$ and $f^\alpha = 70\%$. The standard (relative) deviation on the volume fractions is about 2%. These results agree with previous studies (Gourbesville, 2000). The present analysis indirectly grants information about the crystallographic texture of each phase. The volume fractions estimated on the powder sample or on the massive polycrystal, without texture correction, are in good agreement. Thus, it indicates that the studied Ti-17 polycrystal presents a quasi-isotropic crystallographic texture. This result was confirmed a posteriori during X-Ray stress analysis: no significant variations of the diffracted intensity could be observed.

4.2.3 The question of the macroscopic and pseudo-macroscopic elasticity constants in ($\alpha+\beta$) Ti-17 alloy

In order to determine the XEC required for SEC calculation according to (1-12), pseudo-macroscopic elastic constants of Ti17- α and Ti17- β phases are necessary to proceed to the localization of the applied macroscopic stresses at pseudo-macroscopic scale. In fact, the required tensors cannot be determined independently, so that a hypothesis on α -phase elastic behaviour has to be made. Chemical elements analyses have been performed in a SEM, in order to evaluate the concentration of alloying elements in each phase. Microprobe size is around $1\ \mu\text{m}^3$, so that α and β phase can be analysed separately. Table 5 summarizes the experimental results. The mean metallurgical composition recalculated for Ti-17 samples were compared to the values given in the literature (Donachie, 2000). According to table 5,

alloying elements are often preferentially segregated in a specific phase. α -stabilizers (i.e. aluminum, and tin) are principally gathered into the dark platelets appearing on the micrographs (fig. 1). Thus, these grains correspond to the α -phase. Conversely, β -stabilizers elements (i.e.: chromium, molybdenum, vanadium and iron) are concentrated into the light zones of the micrographs, which correspond to β -grains. Alloying elements total weight fraction is weaker in α -phase (13.5%) than in β (23.7%). According to (Donachie , 2000), IMI-834 is a quasi- α titanium alloys containing more alloying elements than Ti-17 α -phase. Moreover, its Young modulus (around 114 GPa) is very close from the one of pure α -Ti deduced from KESC model based on the SEC given in (Simmons and Wang, 1971) as input parameters (114,6 GPa). Accordingly, in spite of the presence of some alloying elements in Ti-17 α -phase, its elasticity constants were assumed equal to those of commercially pure α -Ti.

This additional hypothesis enables the determination of β -Ti pseudo-macroscopic elasticity constants using an inversion of KE scale transition model, tensile tests in elastic strain domain and ultrasonic tests. The inverse micro-mechanical model, experiments and the corresponding results have been described in (Fréour et al. 2002). Table 6 presents the main results: macroscopic and pseudo-macroscopic elasticity constants of Ti-17.

The only missing information for an application of explicit forms (1-12) in order to evaluate Ti-17 β -phase SEC is the XEC of this particular phase. The required values will be determined in the following paragraph.

4.3 Experimental determination of $\{310\}_\beta$ XEC on a two-phases Ti-17 polycrystalline sample

A bending device was specifically designed in order to apply a high macroscopic stress to a Ti-17 plate with thickness $e=1$ mm. The bending device is constituted by a cylindrical

template which radius is $R=99.5$ mm. According to the macroscopic Young modulus Y given in table 6, the corresponding macroscopic longitudinal stress experienced by the sample would be $\sigma_{11}^I = Y \frac{e}{2R} = 552$ MPa. This value is about half the yield limit of the alloy, so that the material should deform purely elastically. Concentration tensors calculated according to KESC model yield the following pseudo-macroscopic biaxial stresses in α and β (refer to Freour et al., 2003 for details):

$$\sigma^\alpha = \begin{bmatrix} 559 & 0 & 0 \\ 0 & -9.5 & 0 \\ 0 & 0 & -9.5 \end{bmatrix} \text{ MPa} \quad \sigma^\beta = \begin{bmatrix} 536 & 0 & 0 \\ 0 & 22 & 0 \\ 0 & 0 & 22 \end{bmatrix} \text{ MPa} \quad (17)$$

X-Ray strain measurements were performed on a four circles XRD 3003 PTS Seifert diffractometer with a Cu-K α radiation. High Bragg angles and intensities are required for a precise experimental determination of the XEC. Since β -Ti has a body-centered cubic structure, only the $\{310\}$ planes at $2\theta = 97,5^\circ$ satisfy this requirement. α -phase has a hexagonal structure, thus, several planes families are available at high 2θ values. $\{21.3\}$ and $\{30.2\}$ lines are usually used, because they diffract at $2\theta = 139,73^\circ$ and $2\theta = 148,75^\circ$ respectively. However, $\{30.2\}$ was very broad in the studied alloy, so that the determination of the peak position could not be achieved with an acceptable accuracy. Thus, two diffracting families were studied: $\{21.3\}_\alpha$ and $\{310\}_\beta$ planes. A χ acquisition mode with a gas proportional position sensitive detector was used (χ mode is often called ψ mode in the literature, see e.g. Hauk 1997 for a description of the various acquisition modes). Diffraction patterns were obtained for 2 mechanical states of the plate: without applied stress and during bending. Diffraction patterns were recorded along the longitudinal direction for 13 tilt angles ψ varying between -55 to 60° (ψ is the inclination angle of the sample surface normal with respect to the diffraction vector). Peak positions were evaluated after background removal with the threshold centroid method. The curves giving the evolution of the lattice

strains $\varepsilon_{\phi\psi}$ vs. $\sin^2\psi$ for the two studied planes are given on figure 3 (ϕ denotes the rotation of the sample around the sample surface normal. Since the measurements are performed along the longitudinal direction, ϕ is fixed to 0°).

It will be assumed that the macroscopic applied stress state is perfectly uniaxial along longitudinal axis x_1 . For an isotropic sample, the measured strain in α -phase is given by Hauk (1997):

$$\varepsilon_{\psi}^{\alpha} = \frac{1}{2} S_2^{\alpha} \left[\sigma_{11}^{\alpha,A} + \sigma_{11}^{\alpha,R} - \sigma_{33}^{\alpha,A} - \sigma_{33}^{\alpha,R} \right] \sin^2 \psi + \frac{1}{2} S_2^{\alpha} \left[\sigma_{33}^{\alpha,A} + \sigma_{33}^{\alpha,R} \right] + S_1^{\alpha} \left[\sigma_{11}^{\alpha,A} + \sigma_{11}^{\alpha,R} + \sigma_{22}^{\alpha,A} + \sigma_{22}^{\alpha,R} + \sigma_{33}^{\alpha,A} + \sigma_{33}^{\alpha,R} \right] \quad (18)$$

Where the exponents A and R respectively denote the Applied and Residual stresses in the considered α -phase (a similar equation is obtained for β -phase).

The measurement without applied stress yields the strains due to residual stresses only. The equation can be deduced from (18), introducing the condition $\sigma_{11}^{\alpha,A} = \sigma_{22}^{\alpha,A} = \sigma_{33}^{\alpha,A} = 0$.

If the applied load remains purely elastic, especially at mesoscopic and pseudo-macroscopic scales, the residual stresses do not vary during the analysis (the satisfaction of this criterion was checked through measurement of the lattice strains before loading and after unloading. The good agreement between the two sets of data shows that no plastic deformation occurred during the bending of the specimen). That condition enables XEC determination from the subtraction of the lattice strains measured under and without applied load:

$$\Delta\varepsilon_{\phi\psi} = \varepsilon_{\phi\psi}(R = 99.5) - \varepsilon_{\phi\psi}(R \rightarrow \infty) \quad (19)$$

Table 7 gives the slope and origin of the $\sin^2\psi$ curves drawn on figure 3. The numerical results obtained for $\Delta\varepsilon_{\phi\psi}$ in the case of α -phase were compared to KESC model predictions (this is possible because the properties of α -phase are already available in the literature). A satisfactory agreement was obtained for the slope, because the relative deviation between experiments and simulations is about 4% only. This represents 22 MPa, if the slope is converted to stresses through the introduction of the XEC $\frac{1}{2} S_2\{21.3\}_{\alpha} = 11.72 \cdot 10^{-6}/\text{MPa}$.

α -phase analysis was not required in order to characterize the second phase, but it is interesting, because it represents an internal gauge allowing to check the reliability of the stresses actually applied by the bending device.

Stress concentration given in (17) was introduced in (18-19) in order to evaluate the XEC of β -Ti-17 for the studied plane: $\frac{1}{2}S_2\{310\}_\beta = 14.56 \cdot 10^{-6}/\text{MPa}$.

This value will now be used in order to determine β -phase SEC.

4.4 Ti-17 β -phase calculation and discussion

The results obtained in the previous paragraph for Ti-17 β -phase $\{310\}$ planes XEC were reported in equations (1-15). The required macroscopic and pseudo-macroscopic elasticity constants are given in table 5 for Ti-17 and Ti-17 β -phase. Numerical computations yield the values given in table 8 ($c_{11}^{\text{Ti-17}\beta}=174$ (21) GPa, $c_{12}^{\text{Ti-17}\beta}=116$ (18) GPa, $c_{44}^{\text{Ti-17}\beta}=41$ (13) GPa), where Ti-17 β -phase SEC have been compared to Ti-40Nb (100% β) mesoscopic elastic properties given in (Reid et al., 1973).

The deviation observed in the SEC of the two materials is consistent with the measurements of β -Ti macroscopic elasticity constants in various (100% β) alloys (Kuroda et al., 1998; Niinomi, 1998). Contrary to α -phase elastic properties, which remain almost constant in two-phases Titanium based alloys, due to the small concentration of alloying elements in this phase, β -Ti SEC and its related pseudo-macroscopic elasticity constants vary from one alloy to the other. This is due to the very strong variation in the nature and concentration of β -stabilizers elements present in the alloys.

Conclusion

An inverse Kröner-Eshelby self-consistent scale transition model leads to explicit simplified form for the unknown single-crystal elasticity constants of a given cubic phase embedded in any multi-phase material. The application of this model requires the knowledge of one X-ray elasticity constant of the analysed phase, its pseudo-macroscopic elasticity constants and those of the multiphase polycrystal. These new analytical relations were successfully checked on pure single-phases.

This original approach was finally applied in order to characterize Ti-17 β -phase single-crystal elasticity constants. Scanning Electron Microscopy and the associated X-ray microprobe quantitative elements analysis provided a useful support for the justification of most of the assumptions taken into account at mesoscopic and pseudo-macroscopic scales in the micro-mechanical model.

$\frac{1}{2} S_2\{310\}_\beta$ X-ray elasticity constant was experimentally determined using the classical X-ray diffraction method involving an external loading on the studied two-phases sample. Ti-17 β -phase single-crystal elasticity constants were deduced from these experiments and compared to values available in the literature for a 100% β titanium alloy. The deviation between the previously cited mesoscopic elastic stiffness confirmed the dependence of Ti alloys β -phase elastic properties at grain and phase scale with the nature and concentration in β -stabilizers alloying elements present in the material.

The present approach is less precise than the classical ultrasonic single-crystal elasticity constants determination methods, but it is suitable to characterize the properties of a phase, which is not available in single-phase form. Accordingly, it could be used in order to estimate the single-crystal elasticity constants of crystalline phases embedded in either a polycrystalline or an amorphous matrix (in that last case, the contribution of the amorphous matrix to the measured diffraction pattern will have to be carefully handled).

Fréour, S., Gloaguen, D., François, M., Perronnet, A., Guillén, R. (2005). Determination of Single-Crystal Elasticity Constants in a cubic phase within a multiphase alloy - X-Ray Diffraction measurements and inverse scale transition modelling, *Journal of Applied Crystallography*, **38**, 30-37.

Finally, this study highlights the complementarities of inverse scale transition models and X-ray diffraction techniques for the characterization of mesoscopic elastic properties in a multi-phase material.

Acknowledgments

The authors wish to kindly acknowledge the production engineering students of the “Lycée polyvalent régional Aristide Briand de Saint-Nazaire”, who provided the bending device. Special thanks are due to J. P. Toublanc concerning this realization.

References

Behnken, H. and Hauk, V. (1990). *Z. Metallkde.* **81**, 891-985, 1990.

Bish, D. L. and Post, J. E. (1989). In: “Modern Powder Diffraction”, Reviews of mineralogy, **20**.

Bollenrath, F., Hauk, V. and Müller, E. H. (1967). *Z. Metallkde.* **58**, 76-82.

Bruno, G. and Dunn, B. D. (1997). *Meas. Sci. Technol.* **8**, 1244-1249.

Chung, D. H. and Buessem, W.R. (1967). *J. Appl. Phys.* **39**, 217-245.

de Wit, R. (1997). *J. Appl. Cryst.* **30**, 510-511.

Donachie, M. J. (2000). In: “Titanium, a technical guide, second edition”, ASM.

Eshelby, J. D. (1957). *Proc. Roy. Soc. (London)* **A 241**, 376-396.

Fréour, S., Gloaguen, D., François, M., Guillén, R., Girard E., and Bouillo, J. (2002). *Mat. Sci. For.* **404-407**, 723-728.

Fréour, S. (2003). PhD thesis, chapter 2, University of Nantes, France.

Fréour, S., Gloaguen, D., François, M. and Guillén, R. (2003). *Phys. Stat. Sol. (b)* **239**, 297-309.

Gnäupel-Herold, T., Brand, P. C., and Prask, H. J. (1998). *J. Appl. Cryst.* **31**, 929-935.

Fréour, S., Gloaguen, D., François, M., Perronnet, A., Guillén, R. (2005). Determination of Single-Crystal Elasticity Constants in a cubic phase within a multiphase alloy - X-Ray Diffraction measurements and inverse scale transition modelling, *Journal of Applied Crystallography*, **38**, 30-37.

goodfellow data base, available at www.goodfellow.com/csp/active/gfMaterialsTablesList.csp

Gourbesville, O. (2000). PhD thesis, ENSAM, Paris, France.

Hauk, V. and Kockelmann, H. (1979). *Z. Metallkde* **70**, 500-502.

Hauk, V. (1997). In: « Structural and Residual Stress Analysis by Nondestructive Methods », Elsevier Science.

Hayakawa, M., Imai, S. and Oka, M. (1985). *J. Appl. Cryst.* **18**, 513-518.

Koch, N., Welzel, U., Wern, H. and Mittemeijer, E.J. (2004), accepted for publication in *Phil. Mag.*

Kocks, U. K., Tomé, C. N., and Wenk, H. R. (1998). In: “Texture and Anisotropy: preferred orientations in polycrystals and their effects on materials properties”, Cambridge University Press.

Kröner, E. (1958). *Z. Physik.* **151**, 504-518.

Kuroda, D., Niinomi, M., Morinaga, M., Kato, Y. Yashiro, T. (1998). *Mat. Sci. Eng.* **A243**, 244-249.

Lemaitre J. and Chaboche, J.-L. (1985). In: « Mécanique des matériaux solides », Bordas, Paris.

Lira, I. (2002). In: “Evaluating the measurement uncertainty - fundamentals and practical guidance”, Institute of Physics Publishing, Bristol.

Matthies, S., Merkel, S., Wenk, H. R., Hemley, R. J. and Mao H. (2001). *Earth and Planetary Science Letters*, **194**, 210-212.

Mura, T. (1987). In: “Micromechanics of Defects in Solids”, 2nd edition, Dordrecht: Martinus-Nijhoff, chapter 2.

Niinomi, M. (1998). *Mat. Sci. Eng.* **A243**, 231-236.

Nye, J. F. (1957). In: “Physical Properties of Crystals”, Oxford.

Qiu, Y. P. and Weng, G. J. (1991). *Int. J. Solids Structures* **27**, n°12, 1537-1550.

Reid, C. N., Routbort J. L. and Maynard, R. A. (1973). *J. Appl. Phys.* **44**, n°3, 1398-1399.

Fréour, S., Gloaguen, D., François, M., Perronnet, A., Guillén, R. (2005). Determination of Single-Crystal Elasticity Constants in a cubic phase within a multiphase alloy - X-Ray Diffraction measurements and inverse scale transition modelling, *Journal of Applied Crystallography*, **38**, 30-37.

Reuss, A. (1928). In: “Lehrbuch der Kristallphysik”, Teubner, Leipzig/Berlin.

Shang, S., Hu, G. and Shen, J. (2001). *Scripta Materialia* **45**, 287-292, 2001.

Simmons, G. and Wang, H. (1971). Cambridge, MA : M. I. T. Press.

Sprauel, J. M. (1996). In: “Analyse des contraintes résiduelles par Diffraction des Rayons X et des neutrons”, chapter VIII, edited by le Commissariat à l’Energie Atomique, 185-202.

Tanaka, K. and Koiwa, M. (1996). *Intermetallics* **4**, S29-S39.

Tanaka, K., Akiniwa, Y. and Ito, T. (2000). Proc. of the ECRS 6 **2**, 1012-1019.

Voigt, W. (1929). *Z. Angew. Math. Mech* **9**, 49-58.

Figures captions

Figure 1: SEM observation of a surface prepared Ti-17 sample. Dark grains belongs to α -phase. White/grey areas are β grains.

Figure 2: diffraction patterns of both powder and bulk samples of Ti-17 in the range concerning $\{200\}$ - β and $\{10.2\}$ - α . Comparison with JCPDS database for pure α (file ICDD n° 44-1294) and β specimens (file ICDD n° 44-1288).

Figure 3: Diffracting volume pseudo-macrostrains in a Ti-17 thin plate – residual strains state and residual plus applied bending strains.

Tables captions

Table 1: XEC $\frac{1}{2}S_2$ as a function of the diffracting planes $\{hkl\}$ [$10^{-6}/\text{MPa}$], according to the measurement presented in (Gnäupel-Herold et al., 1998).

Table 2: XEC $\frac{1}{2}S_2$ $\{h00\}$ and $\frac{1}{2} S_2$ $\{hhh\}$ with their standard deviation [$10^{-6}/\text{MPa}$], deduced from a linear regression on the values given in table 1.

Table 3: Single-phase macroscopic stiffnesses according to *goodfellow data base*. The standard deviation given in bracket for C_{11} and C_{12} was determined assuming a relative error of 2% on both Y and ν .

Table 4: Comparison between calculated (present explicit forms) and experimental SEC (Chung and Buessem, 1967). The deviation stands here for the relative error between the predicted and expected values. A_c is the coefficient of anisotropy of the single-crystal.

Table 5: Alloying elements weight fractions in Ti-17 and its constituents. $[\mu X]$ denotes X-ray microprobe measurements.

Table 6: macroscopic and pseudo-macroscopic elastic properties of Ti-17.

Table 7: linear regression of the type $\varepsilon_{\varphi\psi} = p \sin^2\psi + o$ obtained from experiments and KESC modeling.

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Figures

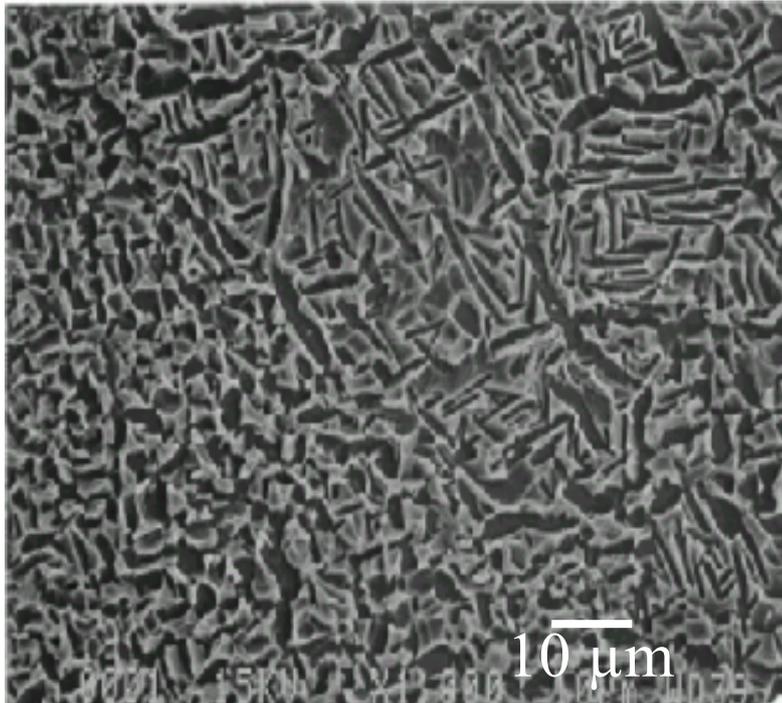


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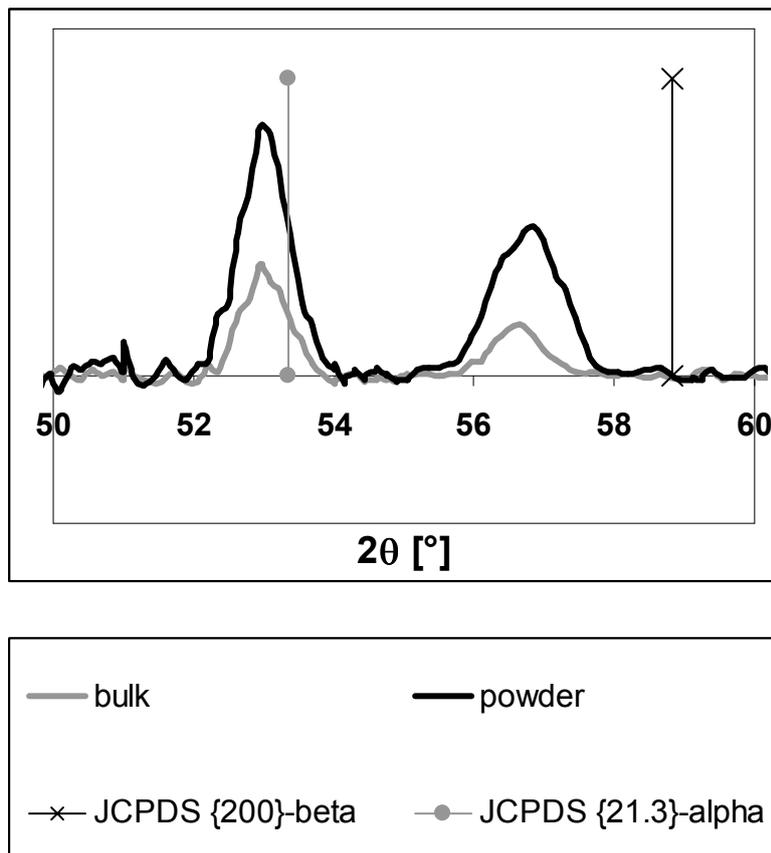


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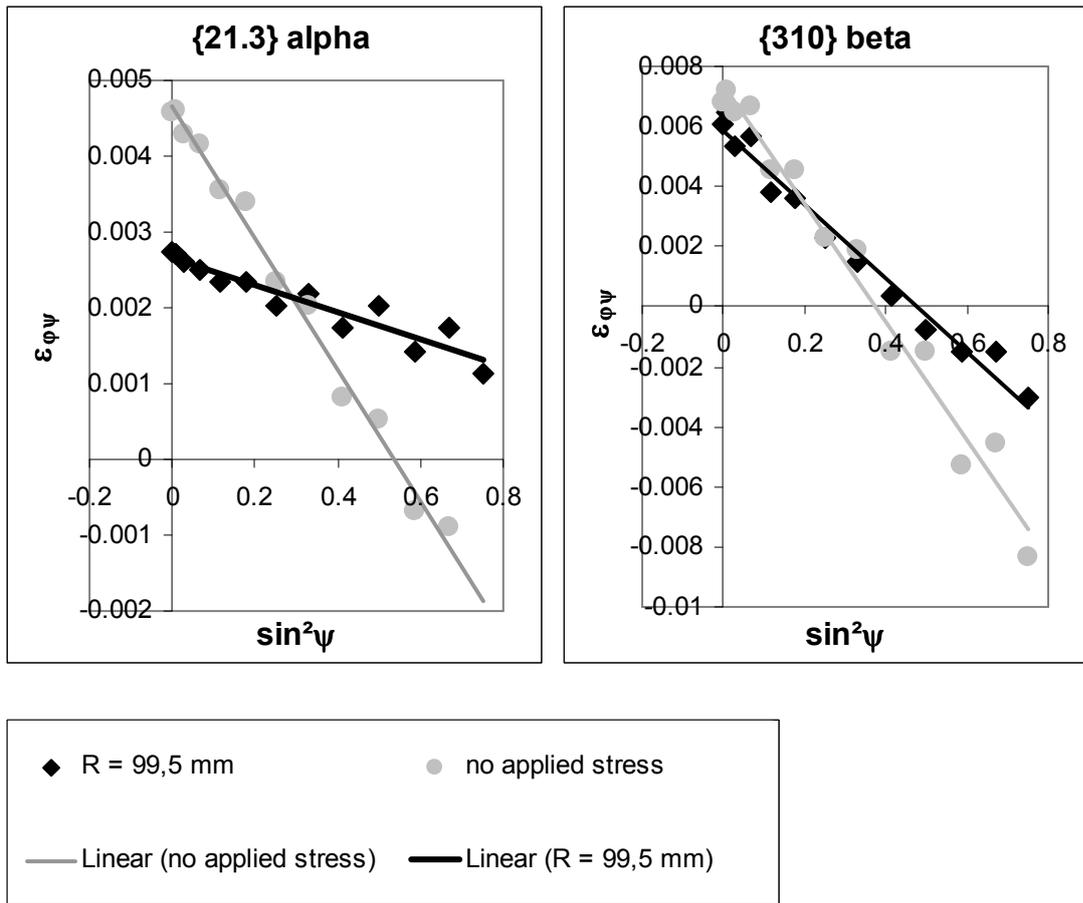


Figure 3: Diffracting volume pseudo-macrostrains in a Ti-17 thin plate – residual strains state and residual plus applied bending strains.

Tables

α -iron	{002}	{011}	{211}	{310}	-	-
	7.522	5.499	5.342	6.852	-	-
Aluminum	{002}	{022}	{222}	{311}	{331}	{420}
	19.765	18.793	18.318	19.069	17.808	18.306
Copper	{002}	{022}	{111}	{311}	{331}	-
	13.66	9.26	8.77	10.51	8.71	-

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	$\frac{1}{2} S_2 \{h00\}$ (mean value)	$\frac{1}{2} S_2 \{h00\}$ (standard deviation)	$\frac{1}{2} S_2 \{hhh\}$ (mean value)	$\frac{1}{2} S_2 \{hhh\}$ (standard deviation)
α -iron	7.56	0.38	4.23	0.24
Aluminum	19.60	0.01	18.03	0.02
Copper	13.35	0.10	7.91	0.05

Table 2: XEC $\frac{1}{2}S_2 \{h00\}$ and $\frac{1}{2} S_2 \{hhh\}$ with their standard deviation [$10^{-6}/\text{MPa}$], deduced from a linear regression on the values given in table 1.

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Macroscopic elasticity constants	α -iron	Aluminum	Copper
Y [GPa]	211.4	70.6	129.8
ν	0.293	0.345	0.345
C_{11} [GPa]	279.2 (7.1)	110.9 (3.9)	202.2 (7.0)
C_{12} [GPa]	115.7 (5.6)	58.4 (3.7)	105.6 (6.5)

Table 3: Single-phase macroscopic stiffnesses according to *goodfellow data base*. The standard deviation given in brackets for C_{11} and C_{12} was determined assuming a relative error of 2% on both Y and ν .

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Phase	SEC	c_{11} [GPa]	deviation	c_{12} [GPa]	deviation	c_{44} [GPa]	deviation	A_c	deviation
α -iron	Calculation	236 (12)	1 %	137 (9)	2 %	132 (6)	13 %	2.69	18 %
	Experimental value	237.4		134.7		116.4		2.27	
Aluminum	Calculation	109 (7)	1 %	60 (5)	2 %	29 (3)	2 %	1.19	2 %
	Experimental value	108.2		61.3		28.5		1.22	
Copper	Calculation	171 (10)	2 %	121 (9)	1 %	76 (7)	1 %	3.06	5 %
	Experimental value	168.4		121.4		75.5		3.21	

Table 4: Comparison between calculated (present explicit forms) and experimental SEC (Chung and Buessem, 1967). The deviation stands here for the relative error between the predicted and expected values. A_c is the coefficient of anisotropy of the single-crystal.

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	Dark plates (Ti-17 α) [μ X]	Light grains (Ti-17 β) [μ X]	Ti-17 Calculated from [μ X]	Ti-17 from (Donachie , 2000)
Ti [w%]	86.4 (0.5)	76.3 (0.5)	83.2 (2.1)	80,5-85,5
Al [w%]	6.6 (0.1)	3.3 (0.1)	5.5 (0.2)	4,5-5,5
Cr [w%]	0.4 (0.1)	7.3 (0.2)	2.6 (0.2)	3,5-4,5
Zr [w%]	3.0 (0.1)	2.0 (0.1)	2.6 (0.2)	1,5-2,5
Mo [w%]	0.9 (0.1)	9.1 (0.2)	3.6 (0.2)	3,5-4,5
Sn [w%]	2.7 (0.1)	2.0 (0.1)	2.5 (0.2)	1,5-2,5

Table 5: Alloying elements weight fractions in Ti-17 and its constituents. [μ X] denotes X-ray microprobe measurements.

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	C_{11} [GPa]	C_{12} [GPa]	Y [GPa]	ν
Ti-17 (Fréour et al., 2002)	169.4 (4.2)	87.4 (3.9)	110.0 (2.2)	0.340 (0.005)
α -phase KESC model from SEC (Simmons and Wang, 1971)	165.1	78.4	114.6	0.322
β -phase (Fréour et al., 2002)	183.0 (12.6)	111.0 (13.0)	99.3 (6.9)	0.377 (0.015)

Table 6: macroscopic and pseudo-macroscopic elastic properties of Ti-17.

		slope p [10^{-6}]		origin o [10^{-6}]	
		experiment	simulation	experiment	simulation
$\{21.3\}_\alpha$	$\varepsilon_{\varphi\psi}$ (R=99,5 mm)	-1790 (187)	-	-2652 (74)	-
	$\varepsilon_{\varphi\psi}$ (no applied stress)	-8707 (226)	-	4664 (89)	-
	$\Delta\varepsilon_{\varphi\psi}$	6918	6655	-2012	-1663
$\{310\}_\beta$	$\varepsilon_{\varphi\psi}$ (R=99,5 mm)	-12230 (572)	-	5846 (224)	-
	$\varepsilon_{\varphi\psi}$ (no applied stress)	-19698 (930)	-	7386 (364)	-
	$\Delta\varepsilon_{\varphi\psi}$	7468	-	-1546	-

Table 7: linear regression of the type $\varepsilon_{\varphi\psi} = p \sin^2\psi + o$ obtained from experiments and KESC modelling.

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	c_{11} [GPa]	c_{12} [GPa]	c_{44} [GPa]	A_c
Ti-17(β)	174 (21)	116 (18)	41 (13)	1.4 (0.2)
Ti-40Nb(β) (Reid et al., 1973)	156.5 (0.4)	111.6 (0.4)	39.6 (0.1)	1.763 (0.003)

Table 8: SEC and anisotropy elastic coefficient of Ti-17 β -phase. Comparison to Ti-40Nb (100% β) SEC. A_c is the coefficient of anisotropy of the single-crystal.