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► **To cite this version:**

Sylvain Fréour, David Gloaguen, Manuel François, Ronald Guillén. Application of inverse models and XRD analysis to the determination of Ti-17 beta-phase Coefficients of Thermal Expansion. *Scripta Materialia*, 2006, 54 (8), pp.1475-1478. 10.1016/j.scriptamat.2005.12.051 . hal-01005257

HAL Id: hal-01005257

<https://hal.science/hal-01005257>

Submitted on 12 Jun 2014

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Application of inverse models and XRD analysis to the determination of Ti-17 β -phase coefficients of thermal expansion

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The scope of this work is the determination of the coefficients of thermal expansion of the Ti-17 β -phase. A rigorous inverse thermo-elastic self-consistent scale transition micro-mechanical model extended to multi-phase materials was used. The experimental data required for the application of the inverse method were obtained from both the available literature and especially dedicated X-ray diffraction lattice strain measurements performed on the studied ($\alpha + \beta$) two-phase titanium alloy.

Keywords: Coefficients of thermal expansion (CTE); X-ray diffraction (XRD); Multi-phases materials; Titanium alloy; Self-consistent scale transition model

1. Introduction

Ti-17 (Ti-5Al-2Sn-2Zr-4Mo-4Cr) is a “beta-rich” alpha-beta titanium alloy, due to an 8% content of β -stabilizer alloying elements such as molybdenum and chromium. This high-strength and deep hardenable alloy was primarily developed by GE Aircraft Engines [1]. Ti-17 has strength properties superior to those of Ti-6Al-4V. It also exhibits higher creep resistance at intermediate temperatures. This alloy is used for heavy-section forgings of up to 150 mm for gas turbine engine components, such as fan or compressor discs, and other elevated temperature applications demanding high tensile strength and good fracture toughness [2].

Now, it is well known that the mechanical behaviour, performance [3] and durability [4] of materials are strongly related to the presence and the amount of residual stresses.

Thermal treatments are fundamental steps in metal forming: actually, during cooling processes, residual stresses appear under specific conditions. The magnitude and the sign of these stresses are strongly related to the coefficients of thermal expansion (CTE) of the grains and phases constituting the material. In fact, in single-phase polycrystals, thermal stresses are only due to single-crystals' CTE anisotropy. In multi-phase materials, however, inter-phase thermal heterogeneities are involved in the distribution of residual stresses that occurs during heat treatments. Therefore, in order to properly select a material for a given industrial application, it is necessary to understand the nature and the source of these residual stresses. This can be investigated either using experimental techniques [5], or scale transition models [6]. This latter solution requires an accurate knowledge of the CTE of each constituent (i.e., the phases, especially) of the multi-phase structure. Thus, the precise identification of phase CTE is important in the field of engineering applications of materials. The classical experimental techniques devoted to this type of characterization are limited to the case of single-phase

materials. Their application to the case of multi-phases material obviously yields the average CTE of the whole polycrystal [7]. A recently proposed inverse method based on a coupling of both a thermo-elastic self-consistent model and experimental techniques was developed in order to ensure the direct determination of the unknown CTE of a phase embedded in a multi-phase material [8]. The method was initially successfully checked in the case of an aluminium–silicon carbide metal matrix composite. Nevertheless, it also applies to any multi-phase heterogeneous material, and in particular, to the case of $(\alpha + \beta)$ titanium alloys such as Ti-17. Since it was demonstrated in several papers that X-ray diffraction lattice strain measurement was sensitive to the strains due to thermal loading of polycrystals [3,5,8], this technique will be used in order to provide the experimental data required for the proper application of the inverse method.

2. Self-consistent inverse formalism for identifying the CTE of a phase constituting a multi-phase material

The full description of the model was given in Ref. [8]. Thus, only the most relevant results and definitions will be summarized here, for the understanding of the following sections of the present work.

A three-scales [9,10] self-consistent formalism is used. The macroscopic scale is denoted by the superscript I. It is assumed to represent the effective average behaviour of the multi-phase polycrystal. The average properties and mechanical states of the phases constituting the material are represented by the superscripts α and β for α -titanium and β -titanium, respectively. This level of the representation is classically called “pseudo-macroscopic” scale. Finally, the anisotropic mechanical states of the grains (the so-called “crystallites”) constituting each phase are denoted by the superscript II that stands for the mesoscopic scale of the material.

In the above-described approach, only pure thermo-elastic sollicitation of the material is considered. In the self-consistent approach, the polycrystalline aggregate is replaced by a homogeneous equivalent matrix exhibiting the same thermo-elastic response and each grain is treated as a heterogeneity with different elastic and thermal properties. The thermal dilatation created by a temperature increment δT is considered as a stress-free strain. The temperature increment is assumed to be homogeneous at every scale of the representation (e.g., it applies equally to the polycrystal, and its constituents: phases and crystallites).

The inverse form obtained for \mathbf{M}^β , the CTE of the β -phase of a two-phases $(\alpha + \beta)$ alloy is:

$$\mathbf{M}^\beta = \frac{f^\alpha}{f^\beta} [\mathbf{C}^{\beta-1} \dots \mathbf{C}^\alpha - \mathbf{I}] \dots \left(\frac{1}{\delta T} \boldsymbol{\varepsilon}^\alpha \right) + \frac{1}{f^\beta} \mathbf{M}^I - \frac{f^\alpha}{f^\beta} \mathbf{C}^{\beta-1} \dots \mathbf{C}^\alpha \dots \mathbf{M}^\alpha \quad (1)$$

Here, \mathbf{M} stands for the coefficients of thermal expansion when \mathbf{C} defines the elastic stiffness (please note that upper-case letters are used to define the properties at both pseudo-macroscopic and macroscopic scales, whereas lower case \mathbf{m}^α and \mathbf{c}^α describe the corresponding thermo-elastic properties for a grain of α -phase); \mathbf{I} is the fourth order identity tensor. $\boldsymbol{\varepsilon}$ stands for the strains (stresses will be denoted by $\boldsymbol{\sigma}$); f^α and f^β are the volume fraction of the two-phases; $\mathbf{A} \dots \mathbf{B}$ denotes the double scalar product $\mathbf{A}_{ijkl} \mathbf{B}_{klmn}$.

Form (1) is called an inverse relation, because the classical (direct) micro-mechanical model ensures the calculation of macroscopic properties from homogenisation operations performed on the constituents of the polycrystal. Here, the macroscopic properties are assumed to be known and used in order to determine the CTE of one phase. Thus, it is an inverse method.

Actually, any state variable is excluded from the analytical form of the ratio $\frac{1}{\delta T} \boldsymbol{\varepsilon}^\alpha$:

$$\frac{1}{\delta T} \boldsymbol{\varepsilon}^\alpha = \left\langle [\mathbf{c}^\alpha(\Omega) + \mathbf{C}^I \dots \mathbf{R}^I]^{-1} \right\rangle \dots (\mathbf{C}^I + \mathbf{C}^I \dots \mathbf{R}^I) \dots \mathbf{M}^I + \left\langle [\mathbf{c}^\alpha(\Omega) + \mathbf{C}^I \dots \mathbf{R}^I]^{-1} \dots \times [\mathbf{c}^\alpha(\Omega) \dots \mathbf{m}^\alpha(\Omega) - \mathbf{C}^I \dots \mathbf{M}^I] \right\rangle_\alpha \quad (2)$$

Thus, the form (1) obtained for \mathbf{M}^β is general and applies for any loading conditions of the polycrystal.

In Eq. (2), the brackets $\langle \rangle$ denote a volume average performed here on all the elementary domains constituting the α -phase. Ω stands for the specific orientation of the considered inclusion in the specimen reference frame. Ω represents a vector in the three-dimensional crystallographic orientation (Euler) space and defines the crystallographic orientation; $\Omega = (\alpha, \beta, \phi)$, where α , β and ϕ , are three Euler angles.

The so-called reaction tensor \mathbf{R}^I , only depends on the elastic stiffness of the material on a macroscopic scale [11]:

$$\mathbf{R}^I = (\mathbf{I} - \mathbf{S}_{\text{esh}}) \dots \mathbf{S}_{\text{esh}}^{-1} = (\mathbf{C}^{I-1} - \mathbf{E}) \dots \mathbf{E}^{-1} \quad (3)$$

Hill’s tensor \mathbf{E} expresses the local interactions depending on the morphology assumed for the crystallites [12]. It is linked to the Eshelby tensor \mathbf{S}_{esh} by $\mathbf{E} = \mathbf{C}^{I-1} \dots \mathbf{S}_{\text{esh}}$. General relations for its calculation in the case of ellipsoidal inclusions are available in Ref. [11].

According to Eqs. (1)–(3), the precise knowledge of f^α , f^β , \mathbf{c}^α , \mathbf{m}^α , \mathbf{M}^α , \mathbf{C}^α , \mathbf{C}^β , \mathbf{C}^I and \mathbf{M}^I will be necessary for an accurate determination of Ti-17 β -phase CTE using this inverse model. Consequently, the determination of the required data is the main purpose of the following section.

3. Application of the inverse scale transition method to the determination of Ti-17 β -phase CTE

3.1. Introduction

The Ti-17 alloy analyzed in the present work was previously described in Refs. [14,15]. The alloy contains about

30% (volume) of β and, obviously, 70% of α -Ti. The samples were found to be elastically quasi-isotropic in the macroscopic state (due to a quasi-isotropic crystallographic texture in both α - and β -phases). The preliminary study performed in pure elasticity in Ref. [15] reports the stiffnesses \mathbf{C}^I (measured), \mathbf{C}^α (assimilated to α -Ti single-phase macroscopic stiffness) and \mathbf{C}^β (determined through a purely elastic inverse self-consistent scale transition relation). These results are summarized in Table 1 (where Y and ν , respectively, denote Young's modulus and Poisson's ratio).

It was demonstrated in Refs. [13,8] that the pseudo-macroscopic elasticity constants and CTE respectively could, in most of the cases, be identified with the corresponding values for the pure single-phases. Thus, the CTE of Ti-17 α -phase can be taken to be equal to the CTE of pure α -titanium. The coefficients of the polynomial relating the dependence of α -titanium single crystal CTE on temperature are available for example in Ref. [16] (see Table 2).

$$m_i^\alpha(T) = m_i^{\alpha(0)} + m_i^{\alpha(1)}T + m_i^{\alpha(2)}T^2 + m_i^{\alpha(3)}T^3 \quad (4)$$

Using the classical direct self-consistent thermo-elastic model (see for example Refs. [8,11]), the macroscopic CTE of an isotropic pure α -Ti sample at room temperature (298 K) is: $M_{11}^\alpha = 13.18 \times 10^{-6} \text{ K}^{-1}$. This value will be used as input data for Ti-17 β -phase CTE identification.

Now, it appears that only the value of Ti-17 macroscopic CTE is missing in order to be able to apply the inverse method described in the second part of this work. The experiment and calculations necessary to its determination will be described in the next paragraph.

3.2. Coupling X-ray diffraction lattice strains measurement and micro-mechanical models for macroscopic CTE identification in Ti-17 samples

It was previously shown that in several works that X-ray diffraction lattice strain measurement was an effective method for the experimental determination of thermally induced local strains [3,5,8]. In order to properly interpret the experimental results, it is however necessary to properly

Table 1
Macroscopic and pseudo-macroscopic elasticity constants of Ti-17 [15]

	C_{11} (GPa)	C_{12} (GPa)	Y (GPa)	ν
Ti-17	169.4 (4.2)	87.4 (3.9)	110.0 (2.2)	0.340 (0.005)
α -phase	165.1	78.4	114.6	0.322
β -phase	183.0 (12.6)	111.0 (13.0)	99.3 (6.9)	0.377 (0.015)

Table 2
Coefficients of the polynomial relating α -Ti single-crystal CTE to the temperature, from [16]

Coefficients	$10^6 m_i^{(0)}$ (K^{-1})	$10^9 m_i^{(1)}$ (K^{-2})	$10^{12} m_i^{(2)}$ (K^{-3})	$10^{15} m_i^{(3)}$ (K^{-4})
m_1	12791	8110	-37442	30379
m_3	12088	24603	-74074	50252

separate the thermal strains from the other sources of lattice strains, and especially, the ones due to plastic deformation. In the literature (see for instance Refs. [3,5]), residual stress relaxation dedicated thermal treatments are indicated to suppress the contribution of plastic deformation on the measured lattice strains. Consequently, several thermal treatment were performed on Ti-17 samples, in the purpose to find the correct temperature-duration condition set ensuring a full relaxation of the residual stresses. After 4 h, the samples heated at a temperature higher than 475 °C were found to be free of residual stresses, after a slow cooling. X-ray diffraction scans were achieved on the thermal treated samples, on an XRD-3003 PTS Seifert diffractometer set in Ω geometry [17], with a copper tube, in the measurement direction defined by $\phi = 0^\circ$ and $\psi = 0^\circ$. The primary optics consisted of a 1 mm wide collimator slit. The diffracted intensity was measured by a position sensitive detector. Peak positions were evaluated after background removal with the threshold centroid method [17].

For the thermally treated samples, the $\{21.3\}_\alpha$ peak position was determined as $2\theta\{21.3\}_\alpha = 140.890$ (0.060) $^\circ$. The same measurement was performed on a powder sample. The reference position for the same peak was found as follows: $2\theta_0\{21.3\}_\alpha = 140.753$ $^\circ$.

The corresponding lattice strains are $\varepsilon_{00,\text{experimental}}^\alpha\{21.3\} = -(\theta - \theta_0)\cotan\theta_0 = -490(60)10^{-6}$. For a pure thermal load, it was demonstrated in Ref. [8] that the strain tensor in a given crystallite was:

$$\varepsilon^{\text{II}} = [\mathbf{c}^\alpha(\boldsymbol{\Omega}) + \mathbf{C}^I \cdots \mathbf{R}^I]^{-1} \cdots [\mathbf{C}^I \cdots \mathbf{R}^I \cdots \mathbf{M}^I + \mathbf{c}^\alpha(\boldsymbol{\Omega}) \cdots \mathbf{m}^\alpha(\boldsymbol{\Omega})] \delta T \quad (5)$$

The lattice strain $\varepsilon_{\phi\psi}$ corresponds to the average of the mesoscopic strains over the well-oriented grains from the point of view of the diffraction and their projection on the measurement direction defined by the two angles ϕ and ψ [10,18], that are here both taken to be equal to 0° . The procedure necessary to select the properly oriented crystallites is extensively explained in Ref. [19].

An analytical calculation of the lattice strains expected for the plane $\{21.3\}$ in the α -phase was performed with Mathematica 3.0 software. Assuming a cooling from 475 °C to the ambient temperature (25 °C), and a macroscopic CTE M_{11} constant over this temperature range, the thermal lattice strains were found to verify:

$$\varepsilon_{00,\text{calculated}}^\alpha\{21.3\} = 0.00165 - 158M_{11}.$$

A comparison between the experimental and calculated forms obtained for these strains enables the identification of the macroscopic CTE for the Ti-17 researched: $M_{11} = 13.5(0.4) \times 10^{-6} \text{ K}^{-1}$.

3.3. Ti-17 β -phase CTE identification and discussion

The required input parameters described in Sections 3.1 and 3.2 were introduced in Eqs. (1)–(3). Thus, the CTE of the β -phase was obtained: $M_{11}^\beta = 14.5(1.3) \times 10^{-6} \text{ K}^{-1}$. This result is consistent with the value measured at temperatures

higher than the β -transus in pure titanium: $M_{11}^{\beta} = 13.8 \times 10^{-6} \text{ K}^{-1}$ between 900 °C and 1050 °C, according to [20].

The present study shows that the average CTE of the β -phase of a titanium alloy remains close to the ones of the pure single-phase. Consequently, it seems that contrary to the elasticity constants, the CTE of the β -phase of titanium based alloys do not strongly depend on the nature and concentration of β -stabilizing alloying elements that are present in this phase.

Basic computations enabled the prediction of the average strains due to a slow cooling from 475°C to ambient temperature in both the phases constituting Ti-17. The corresponding diagonal elastic stresses components ($\sigma_{11}^i, \sigma_{22}^i, \sigma_{33}^i, i = \alpha, \beta$) necessary to induce the same strain level are: 100 MPa in β -Ti, while α -phase is submitted to -40 MPa. Thus, contrary to the case of pure single-phase α -Ti (cf. Ref. [21]), the strains remaining after stress relaxation thermal treatments are not negligible. In consequence, thermal treatments may have a significant effect on the following plastic deformations in Ti-17. For instance, the activation of several slip systems can be strongly affected by the presence of initial thermal strains, especially in the β -phase.

4. Conclusions

In the present work, an inverse method coupling experiments and scale-transition modeling was applied to the case of a two-phase Ti-17 alloy. For this purpose, X-ray diffraction measurements were performed in order to determine the lattice strains induced by stress relaxation thermal treatments. Using to this technique, the identification of the CTE of both the titanium alloy and its body-centered β -phase phase was achieved. The results show that thermal treatment may induce strain levels on the phase scale that

are expected to have an effect on any subsequent plastic deformation of the material. These properties will be extensively analyzed in further studies.

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