Analysis of Internal Stress Relaxation in an Al/SiC Composite by Neutron Diffraction

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ABSTRACT

Residual thermal stresses in a SiC whisker reinforced/Al 7075 matrix composite were measured as a function of temperature by means of the neutron diffraction technique. The residual stresses in both phases decrease in magnitude with increasing temperature and at the 'equivalent temperature', measured to be about 500K, they reach the stress-free state. Increasing the temperature, these stresses increase numerically in a reverse sense for both phases. The experimental results are explained with an analytical model, developed on the basis of Eshelby's equivalent inclusion method.

1. INTRODUCTION

Modern technologies require materials with an unusual combination of properties, which cannot be achieved with conventional alloys, ceramics or polymeric materials. To enlarge the cause of these conventional properties, a variety of composite materials was developed with properties superior to each of the component phases. In the last years, a substantial amount of research has been conducted on metallic materials reinforced with ceramic phases, whiskers or particulates, driven by the need to reduce weight and increase performance properties in many structural applications. In particular, the interest in dis-

continuously reinforced Metal Matrix Composites (MMCs) has increased dramatically because they have the advantage of being formable by conventional processing and metal working processes /1-4/.

The internal stresses in short fibre metal matrix composites determine much of their mechanical behaviour. These stresses are generated not only when the composite is externally loaded, because of differences in elastic constants between the phases, but also when changes in temperature occur, because of differences in the coefficient of thermal expansion (CTE) of the two phases.

Average residual elastic strains in composites have been measured both by X-ray and neutron diffraction methods /5-16/. Diffraction techniques are in fact powerful tools in investigating residual stresses in MMCs, since it is possible to analyse both the matrix and the fibres as they give separate diffraction patterns. A significant advantage of neutron diffraction for strain analysis is that true bulk values of lattice spacings are obtained. Thus, neutrons are well suited to study the inherent volumetric microstress states found in composites.

The main objective of the present investigation is to determine by neutron diffraction the evolution of the residual stresses in an Al-SiC whisker composite, due to temperature variations. The experimental results will be compared with predictions based on an average field theory using the inclusion equivalent method developed by Eshelby.

2. PRINCIPLE OF NEUTRON DIFFRACTION TECHNIQUE FOR STRESS MEASUREMENTS

The principle of the neutron diffraction method for stress analysis is very similar to the complementary and well established X-ray technique, which involves measuring changes in the interplanar spacings of crystal planes, and each crystallite is turned into a miniature strain gauge [17].

A neutron beam impinging on a polycrystalline material can be diffracted according to Bragg's law:

$$2d_{hkl} \sin \theta_{hkl} = n\lambda \quad (1)$$

where (hkl) are the Miller indices of the diffracting planes, d_{hkl} is the spacing of the lattice planes corresponding to a Bragg reflection (hkl) observed at the scattering angle $\phi_{hkl} = 2\theta_{hkl}$, λ is the neutron wavelength, and n is the order of the reflection which will be assumed equal to unity in the following. The wave-vectors $\mathbf{K}_0$ of the incident and $\mathbf{K}$ of the diffracted beam determine the scattering vector $\mathbf{Q} = \mathbf{K} - \mathbf{K}_0$ of magnitude $|\mathbf{Q}| = 4\pi \sin \theta / \lambda$. Bragg reflection only occurs when $\mathbf{Q}$ is normal to planes giving a diffracted beam and magnitude equal to that of the reciprocal lattice vector τ_{hkl} of the planes, $|\mathbf{Q}| = \tau_{hkl}$.

Differentiating Bragg's law, the following relation can be obtained, if λ is constant:

$$\frac{\Delta d}{d} = -\cot \theta \Delta \theta. \quad (2)$$

Thus a small lattice strain $\varepsilon = \frac{d - d_0}{d_0} = \frac{\Delta d}{d_0}$ induces a change in the scattering angle $\Delta \phi = 2\Delta \theta = -2\varepsilon \tan \theta$.

When a monochromatic neutron beam is used, the lattice strain can be determined by measuring the shift of the scattering angle $\Delta \phi$. The direction of the measured deformation coincides with that of the scattering vector $\mathbf{Q}$. The experimental procedure is to scan the counter angle and to record the scattered neutron intensity as a function of the scattering angle. Usually a fitting routine, based on a least squares minimisation, is used to determine the angle and the angular width of the peak. Thus the deformations can be derived from measuring the shifts of the scattering angles ($\Delta \phi$):

$$\varepsilon = \frac{\Delta d}{d_0} = -\frac{1}{2}(\cot \theta_0) \Delta \phi \quad (3)$$

where d_0 is the lattice spacing of the reference or "stress-free" material and $2\theta_0$ the corresponding scattering angle. In a neutron diffraction experiment for strain measurements, the region of the sample to be investigated, and over which the lattice strain is averaged, is controlled by neutron adsorber collimators in the incident and diffracted beam. By adjusting the width of the slits, volumes as small as one cubic millimeter can be selected. The cross sectional area of the incident beam has to be defined both horizontally and vertically by the use of adsorber masks. Similar masks can be used to define the size of the diffracted beam.

The stresses can then be calculated from the deformations using the equations of the elasticity theory. For a general three-dimensional stress system, at least six measurements of direct strain are required. This allows the principal strains as well as the shear components to be determined. If the principal directions are known, only three orthogonal strains need to be measured and these can be used to obtain the principal stresses using the following equations:

$$\begin{aligned} \sigma_x &= \frac{E}{(1+\nu)(1-2\nu)} \left\{ (1-\nu)\varepsilon_x + \nu(\varepsilon_y + \varepsilon_z) \right\} \\ \sigma_y &= \frac{E}{(1+\nu)(1-2\nu)} \left\{ (1-\nu)\varepsilon_y + \nu(\varepsilon_x + \varepsilon_z) \right\} \\ \sigma_z &= \frac{E}{(1+\nu)(1-2\nu)} \left\{ (1-\nu)\varepsilon_z + \nu(\varepsilon_x + \varepsilon_y) \right\} \end{aligned} \quad (4)$$

In these equations E is Young's modulus and ν is Poisson's ratio for homogeneous material. For a more accurate determination of stresses and considering that most crystal systems are anisotropic, the value of the elastic properties E and ν used in the above equations should be replaced by the elastic properties for the (hkl) reflection used for diffraction measurements $E_{(hkl)}$ and $\nu_{(hkl)}$. Values of $E_{(hkl)}$ and $\nu_{(hkl)}$ can be obtained by calibration measurements [23-25] or by calculation [18-22].

3. ANALYTICAL MODEL

The model based on Eshelby's equivalent inclusion method is used to solve the problem of thermal induced residual stresses in the composite. The model consists

of aligned short fibres (W) which are transverse isotropic and embedded in an infinite matrix (D-W) /28-28/, as shown in Figure 1. All the fibres are assumed to be prolate spheroids of the same size and aligned along the x_3 axis, but otherwise distributed randomly in a three-dimensional space.

In the following, boldface symbols denote either vectors or tensors. In Eshelby's model /29/ the stress inside the inclusion could be expressed by the following equation, using the matrix representation:

$$\sigma = C(\varepsilon^C - \varepsilon^T) \quad (5)$$

where ε^T is the stress-free transformation strain, or inclusion eigenstrain, and ε^C is the inclusion total actual strain, or constrained strain, due to the constraining matrix while the inclusion transforms. Examples of eigenstrains are the coefficient of thermal expansion mismatch strain or plastic deformation. The constrained strain is related to the eigenstrain by the following equation:

$$\varepsilon^C = S \varepsilon^T \quad (6)$$

S is the so-called Eshelby's tensor, which is a function

of the geometry of the ellipsoidal inclusion and of Poisson's ratio

of the matrix. Its detailed expression for various ellipsoidal shapes have been tabulated /30-31/.

In all the practical cases the matrix and the inclusion have different elastic constants, denoted by C^M and C^I , respectively. By the same cutting, straining and welding argumentation as that used for the inclusion problem /32/, the stress inside the inhomogeneity is given by:

$$\sigma = C^I(\varepsilon^C - \varepsilon^{T*}) \quad (7)$$

where ε^{T*} is the stress-free mismatch strain between the two phases and ε^C is the constrained strain. By introducing a fictitious homogeneous inclusion, having the same shape and size as the actual inhomogeneous inclusion, the inhomogeneity problem can be reduced to the inclusion problem if the following relation is satisfied:

$$C^M(\varepsilon^C - \varepsilon^T) = C^I(\varepsilon^C - \varepsilon^{T*}) \quad (8)$$

where ε^T is the transformation strain of the fictitious homogeneous inclusion that is required to have the

Fig. 1: Schematic view of the composite tensile test sample used for neutron measurements. (a) The calculation model for an aligned short fibre composite and (b) the fibre coordinate system.

same matrix stress under the action of the constrained strain.

Equation (8) can be rewritten in the following form:

$$\mathbf{C}^M(\mathbf{S} - \mathbf{I})\boldsymbol{\varepsilon}^T = \mathbf{C}^I(\mathbf{S}\boldsymbol{\varepsilon}^T - \boldsymbol{\varepsilon}^{T*}) \quad (9)$$

where $\mathbf{I}$ is the identity tensor. Thus the equivalent homogeneous transformation strain $\boldsymbol{\varepsilon}^T$ can be expressed as a function of the stress free transformation strain of the inhomogeneous inclusion ($\boldsymbol{\varepsilon}^{T*}$):

$$\boldsymbol{\varepsilon}^T = [(\mathbf{C}^I - \mathbf{C}^M)\mathbf{S} + \mathbf{C}^M]^{-1} \mathbf{C}^I \boldsymbol{\varepsilon}^{T*} \quad (10)$$

The stress inside the homogeneity is then calculated from equation (7) /32/:

$$\boldsymbol{\sigma}_I = \mathbf{C}^M[\mathbf{S} - \mathbf{I}][(\mathbf{C}^I - \mathbf{C}^M)\mathbf{S} + \mathbf{C}^M]^{-1} \mathbf{C}^I \boldsymbol{\varepsilon}^{T*} \quad (11)$$

Eshelby's above mentioned calculation is rigorous for a single inclusion embedded in an infinite isotropic elastic matrix. However, the actual composite materials are finite and contain more than one inclusion, so the interaction among them becomes important. To take into account such an interaction, Eshelby introduced the concept of an "image stress" /32/ which can be confused with the average stress disturbance in the matrix, $\langle \boldsymbol{\sigma} \rangle_M$, that further strains the inclusion. Thus the governing equations of the preceding formulation are still valid, providing that the matrix average strain disturbance $\langle \boldsymbol{\varepsilon} \rangle_M$, is considered. In this way the general equations for the mean stress within the inhomogeneous inclusion are the following:

$$\langle \boldsymbol{\sigma} \rangle_I = \mathbf{C}^I(\boldsymbol{\varepsilon}^C - \boldsymbol{\varepsilon}^{T*} + \langle \boldsymbol{\varepsilon} \rangle_M) \quad \text{inhomogeneity} \quad (12)$$

$$= \mathbf{C}^M(\boldsymbol{\varepsilon}^C - \boldsymbol{\varepsilon}^T + \langle \boldsymbol{\varepsilon} \rangle_M) \quad \text{equivalent inclusion} \quad (13)$$

Thus from equations (12) and (13) it is possible to calculate the stress-free transformation strain of the equivalent homogeneous inclusion. For a finite number of inclusions, with a volume fraction denoted by f , the internal balance of the mean stresses can be written as following /31/:

$$(1-f)\langle \boldsymbol{\sigma} \rangle_M + f\langle \boldsymbol{\sigma} \rangle_I = 0$$

It is then possible to calculate both the matrix and the inclusion mean stresses. So calculating, the mean

matrix strain is given by /32/:

$$\langle \boldsymbol{\varepsilon} \rangle_M = f[\mathbf{S} - \mathbf{I}][(\mathbf{C}^M - \mathbf{C}^I)[\mathbf{S} - f(\mathbf{S} - \mathbf{I})] - \mathbf{C}^M]^{-1} \times \mathbf{C}^I \boldsymbol{\varepsilon}^{T*} \quad (14)$$

In the thermal residual stresses problem, the transformation strain, $\boldsymbol{\varepsilon}^{T*}$, is due to a mismatch between the thermal expansion coefficient of the matrix (α_M) and of the fibres (α_I). Under a temperature change ΔT , it is given by the following expression (in matrix form):

$$\begin{aligned} \varepsilon_i^{T*} &= (\alpha_F - \alpha_M) \cdot \Delta T & \text{for } i = 1, 2, 3 \\ &= 0 & \text{for } i = 4, 5, 6 \end{aligned} \quad (15)$$

The mean matrix stress can then be calculated from Hooke's law: $\langle \boldsymbol{\sigma} \rangle_M = \mathbf{C}^M \langle \boldsymbol{\varepsilon} \rangle_M$, and the mean stress in the fibre can easily be derived from the internal balance of the mean stresses.

The elastic anisotropy of the fibres, assuming transverse isotropy, is included through the stiffness matrix $\mathbf{C}^I$. For the cubic SiC phase, the compliance has the following expression /33,34/:

$$\mathbf{C}^I [\times 10^{11} \text{ Pa}] = \begin{pmatrix} 3.52 & 1.404 & 1.404 & 0 & 0 & 0 \\ 1.404 & 3.52 & 1.404 & 0 & 0 & 0 \\ 1.404 & 1.404 & 3.52 & 0 & 0 & 0 \\ 0 & 0 & 0 & 2.33 & 0 & 0 \\ 0 & 0 & 0 & 0 & 2.33 & 0 \\ 0 & 0 & 0 & 0 & 0 & 2.33 \end{pmatrix} \quad (16)$$

The anisotropy of the matrix compliances is more difficult to deal with; the model, for example, becomes non-analytic. However, it seems to be reasonable to assume that, on the average, the fibres experience a matrix with the elastic properties of polycrystalline Al, whose stiffness matrix is the following /35/:

$$\mathbf{C}^M [\times 10^{11} \text{ Pa}] = \begin{pmatrix} 1.022 & 0.613 & 0.613 & 0 & 0 & 0 \\ 0.613 & 1.022 & 0.613 & 0 & 0 & 0 \\ 0.613 & 0.613 & 1.022 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0.285 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0.285 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0.285 \end{pmatrix} \quad (17)$$

The parameters required are the fibre aspect ratio ($\alpha = l/d$) and the temperature drop $\Delta T = T_i - T_f$ with T_i as the so-called "equivalent" temperature, defined as the

temperature where the strain in the composite is zero (commonly equivalent to the fabrication temperature). Eshelby's tensor expression is given in /31/.

The specific lattice strains (e_{hkl}), measured by neutron diffraction, can be converted to an equivalent strain e_{eq} , according to the Reuss model /8,22/:

$$e_{eq} = e_{hkl} \cdot \left(\frac{E_{hkl}}{E_{bulk}} \right) \quad (18)$$

where E_{bulk} is the bulk Young's modulus of the polycrystal and E_{hkl} is the Young's modulus relative to the (hkl) reflection under investigation. The e_{eq} strain values thus obtained can then be compared with the model strains.

4. MATERIAL

The material under consideration is an aluminium matrix alloy (Al 7075), whose chemical composition is reported in Table 1, reinforced with 27 vol.% SiC whiskers (SiCw). the composite was manufactured by a squeeze casting process and it was extruded at a ratio of more than 10 to get an alignment of SiCw along the extruding direction /36/. From a complete neutron diffraction pattern of the composite (see Figure 2), the

predominant Bragg reflections of SiCw could be indexed as the cubic β -phase.

The sample, 6mm in diameter and 40mm long, was cut in such a way that the fibres were aligned perpendicular to the sample longitudinal axis.

To impose thermal loading, a special heating system was constructed /37/. It consisted of two copper shells, which were slightly conical with a diameter ranging between 17 mm and 20 mm, covering the sample to guarantee a homogeneous temperature distribution over the sample volume under investigation. Each copper shell was equipped with a thermocouple to measure the specimen temperature. The whole system (sample and copper shells) was covered by a wire-cloth of cylindrical shape, which was connected to a high current generator, thus serving as a heating element. The specimen temperature was adjusted using a temperature PID controller unit, operating the high current generator.

5. NEUTRON DIFFRACTION MEASUREMENTS

Neutron diffraction measurements were carried out on the 3T1 powder diffractometer of the Orphée reactor, with the neutron wavelength fixed at 0.193 nm. The

Fig. 2: Diffraction pattern of the Al 7075+27% vol. SiCw composite sample. This experiment was performed using a neutron wavelength of 0.252 nm.

Table 1
Chemical composition (wt%) of the Al 7075 alloy matrix

Mn	Fe	Si	Cr	Ti	Al	Zn	Mg	Cu
0.30	0.70	0.5	0.18-0.40	0.20	bal.	5.1-6.1	2.1-2.9	1.2-2.0

strains in the composite constituents were measured separately, considering the intense β -SiCw (220) reflection, at $2\theta \approx 78^\circ$, and the Al (220) reflection, corresponding to $2\theta \approx 84.9^\circ$. All the sample section was under investigation. Measurements were performed in two different orientations, that were parallel to the sample axis (i.e. perpendicular to the fibres axis, in the fibre transverse x_2 direction) and perpendicular to the sample axis (i.e. parallel to the fibres axis, in the fibre longitudinal x_3 direction).

The Al (220) and SiCw (220) diffraction peaks were scanned at different temperatures, i.e. at room temperature, at 348K, at 423K, at 573K and at 673K. The lattice stress free d-spacings, used afterwards for strain calculation, were measured at different temperatures on a powder ground from the sample. The time necessary for each scan of the Al and SiCw reflection was about 4 hours and 2 hours, respectively. To observe eventual diffusional processes, a test composite sample (cut from the same composite rod as the sample under consideration) was heated at 548K and held at this temperature for extended periods (about 13 hours), during which the lattice spacings were recorded repeatedly. The results show no significant variation of the lattice d-spacings with time /37/.

6. RESULTS AND DISCUSSION

The equivalent measured strains, derived from equation (18), are qualitatively in accord with the predictions calculated by the Eshelby-based model illustrated above, assuming $\alpha_M = 23.7 \times 10^{-6} \text{K}^{-1}$ and $\alpha_F = 4.5 \times 10^{-6} \text{K}^{-1}$, measured from the unstressed composite powder and assumed to be isotropic. Figures 4a and 4b show the longitudinal and transverse strains for the fibres and the matrix as predicted by Eshelby's inclusion method for the specified measuring temperature, on the assumption of no stress relaxation.

The families of lines are for fibre aspect ratios ($\alpha = l/d$) of 10, 5 and 1.5 and a volume fraction of 27%.

Figures 4a-b show the comparisons of the measured equivalent strains in the longitudinal and transverse fibre directions of the SiC whiskers with the computed strains. The measured strains are between the predicted values for fibres with an aspect ratio of 1.5 and 5, and the agreement is encouraging. In the case of the Al 7075 matrix, the determined equivalent strains feel clearly the influence of the fibre with an aspect ratio of 1.5. However, the errors involved are large for the Al matrix data, because the strains are relatively smaller and the expansion coefficient larger than for SiCw. Some uncertainty thus remained in the present analysis, in particular regarding the fibre aspect ratio, which may range from 1.5 to 5.

From the measured strains the average residual thermal stresses were calculated using Hooke's law, assuming transverse isotropy (i.e. with reference to Fig. 1 the strains in the x_1 and x_2 directions are supposed to be equal). To determine the residual stresses, Young's

Fig. 3: Schematic view of the heating system designed for thermal loading during neutron diffraction measurements.

Fig. 4: The longitudinal (o) and transverse (•) Al matrix (a) and SiC whisker (b) mean strains derived from the (220) lattice plane spacings measured at various temperatures. The continuous lines are computed by the model based on Eshelby's equivalent inclusion method, assuming different fibre aspect ratios $\alpha = l/d$.

moduli and Poisson's ratios of the (220) lattice plane of the Al matrix and of the SiCw reinforcement were calculated from the single crystal elastic constants by the Kröner model. The values of the Poisson's ratios were assumed to be independent of temperature, while the Young's moduli were introduced as temperature-dependent for the Al matrix only. Supposing that the variations with temperature of the Young's modulus of

the Al lattice plane (220) follow the same law as the macroscopic value /38/, the appropriate values of E are given in Table 2 below for the different temperatures.

The variation of the macroscopic Young's modulus with temperature is less than 2% for the cubic β -SiC whiskers over the range 20°C–400°C /39/ and, consequently, the microscopic E_{220} Young's modulus was also assumed to be independent of the temperature.

The good agreement between the experimental and the computed stress values is evident from Figures 5a-b where the experimental data for the Al matrix and the SiCw fibres have been plotted together with the calculated values. The residual stresses are tensile in the Al 7075 matrix and decrease with increasing temperature. After the "equivalent temperature", estimated at about 500K, the stresses become negative and hence the matrix is in a compressive stress state, which increases in magnitude with temperature (Fig. 5a). On the other hand, the SiCw fibres are in a compressive stress state below the equivalent temperature, while going through the equilibrium state the residual stress state becomes tensile, increasing with temperature (Fig. 5b).

Thus the thermal strains and stresses measured in the whiskers and in the Al matrix by neutron diffraction were in reasonable agreement with those computed by the model based on Eshelby's equivalent inclusion method. In the model the fibre aspect ratio ($\alpha = l/d$) of SiCw has been supposed to be variable in the range 1.5-10. The experimental strain results fit the computed values well, and in particular the Al 7075 matrix seems to feel the presence of fibres with an aspect ratio of 1.5. The present model is also strongly dependent on $\Delta T = T_i - T_f$. In the present computation the choice of the equivalent temperature T_i was made noting that at 500K the composite can be considered to be in a stress-free state [37]. Although the experimental strain evolution with temperature fits the predicted values well, some uncertainty remained in the present stress analysis because of the numerous assumptions introduced for stress evaluation from the experimental strains and significant errors in the experimental data compared with the relatively small deformation involved. In the longitudinal fibre direction the room temperature stress values are higher (in magnitude) than those predicted by the model presented here. This can probably be attributed to the presence of fabrication process residual stresses which have not been taken into account in the model. As the temperature increases, the predominant residual stresses are effectively related to the mismatch between the expansion coefficient of the matrix and of the fibres. However, there is a satisfactory agreement between the computed and measured stress values, indicating that the fibre and

matrix stresses relax in opposite directions at about 500K.

7. CONCLUSIONS

It has been demonstrated that neutron diffraction technique is a powerful tool in analysing the evolution of internal thermal stresses in the components of a metal matrix composite during temperature changes. The results showed the residual stresses to be tensile in the Al matrix, decreasing with temperature. Above the equivalent temperature, estimated at about 500K, the matrix stresses became negative, increasing in magnitude with temperature. On the other hand, the fibres were in a compressive stress state below the equivalent temperature, becoming tensile above. Eshelby's equivalent inclusion method has been used to explain the evolution of the internal stresses, measured by neutron diffraction.

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Table 2
Young's moduli E_T of the Al 7075 matrix as a function of temperature

T [K]	E_T Al (220) [GPa]
298	72
348	69
423	62
573	51
673	42

Fig. 5: The longitudinal (o) and transverse (•) Al matrix (a) and SiC whisker (b) mean stresses derived from the lattice strains measured at various temperatures. The continuous lines are computed by the model based on Eshelby's equivalent inclusion method, assuming different fibre aspect ratios $\alpha=l/d$.

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