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DETERMINING STRESSES IN COMPOSITES

BY

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ABSTRACT

The basic diffraction techniques for examining the stress tensor are reviewed, with particular emphasis on what can be done without knowing the unstressed lattice parameter(s). Examples are given of the residual stresses in thin films, how to measure the bonding and yield stress in composites and separating the micro and macrostresses to examine load sharing between phases.

INTRODUCTION:

In this paper, I will use the term composite in a broad sense, including thin films on substrates, and steel, as well as other phase mixtures i.e. to include any arrangement where one phase has its properties affected by the presence of another.

It is in such an area that diffraction comes into its own, as a tool capable of measuring the stresses in all phases present and with almost an arbitrary geometry. There is no alternative! Of course, we do not really measure stress but strain. The interplanar spacing (d) serves as our internal strain gauge, and is obtained from Bragg's law, by measuring the diffraction angle of peaks. In particular, we can link strain, $\Delta d/d$, measured in the L_3 direction (Fig. 1) to the total (t) stress tensor in a phase α $\langle {}^t\sigma^\alpha_{ij}\rangle$ in the sample coordinate system P_1 through "diffraction elastic constants" S_1, S_2. (The carats imply an average over the diffracting volume.):

$$\langle {}^t\varepsilon^\alpha_{\phi\psi}\rangle = \frac{d^\alpha_{\phi\psi} - d^\alpha_o}{d^\alpha_o} = \frac{S_1}{2}\{\langle {}^t\sigma^\alpha_{11}\rangle\cos^2\phi + \langle {}^t\sigma^\alpha_{12}\rangle\sin 2\phi + \langle {}^t\sigma^\alpha_{22}\rangle\sin^2\phi - \langle {}^t\sigma^\alpha_{33}\rangle\}\sin^2\psi$$

$$+ \frac{S_1}{2}\langle {}^t\sigma^\alpha_{33}\rangle - S_2\{\langle {}^t\sigma^\alpha_{11}\rangle + \langle {}^t\sigma^\alpha_{22}\rangle + \langle {}^t\sigma^\alpha_{33}\rangle\}$$

$$+ \frac{S_1}{2}\{\langle {}^t\sigma^\alpha_{13}\rangle\cos\phi + \langle {}^t\sigma^\alpha_{23}\rangle\sin\phi\}\sin^2\psi. \qquad (1)$$

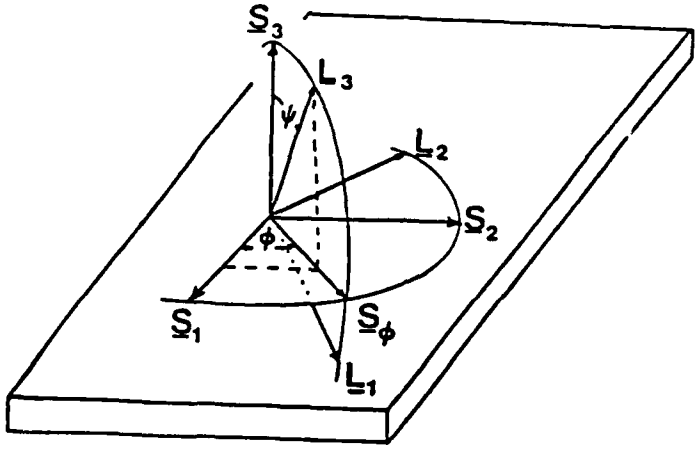


Fig. 1: Definition of the laboratory coordinate system L_i, simple coordinate system S_i, and the angles $\phi\psi$.

It is clear from this equation and Fig. 1, that if we make enough measurements of d at various $\phi$and ψ angles, the entire stress tensor can be determined, and the appropriate set of angles to minimize errors has been examined[1]. The S_i can be measured with known applied loads. If all $\sigma_{i3} = 0$, "d" vs. $\sin^2\psi$ is linear and from the slope in Fig. 1 σ_ϕ is determined along the direction ϕ ($<{}^t\sigma^\alpha_{11}>\cos^2\phi + <{}^t\sigma^\alpha_{12}>\sin 2\phi + <{}^t\sigma^\alpha_{22}>\sin^2\phi$); the sign of the slope itself immediately reveals whether the stress is tensile (positive slope) or compressive (negative slope). If the stresses vary significantly from point to point, d vs. $\sin^2\psi$ will oscillate, but there are also ways of treating such data to obtain the stress [2,3]. Note in Eq. 1 the $\sin 2\psi$ dependence if σ_{i3} are present. This leads to curvature of d vs. $\sin^2\psi$, opposite in sense for plus or minus ψ tilts. On the other hand even in the absence of σ_{i3} strong stress gradients can also lead to curvature, but it is the same for positive or negative ψ tilts. The term σ_{33} is determined from the intercept of "d" vs $\sin^2\psi$, but the value of any σ_{i3} is strongly affected by the value for the unstressed interplanar spacing d_0 (for example, see ref. (4)), which can be difficult to determine since phase compositions are often affected by a second phase. However A. Winholtz[5] has shown that it is possible to rewrite this equation in terms of the deviatoric and hydrostatic stress components, τ_H and τ_{ij}:

$$<{}^t\sigma^\alpha_{ij}> = \begin{bmatrix} <{}^t\tau^\alpha_H> & 0 & 0 \\ 0 & <{}^t\tau^\alpha_H> & 0 \\ 0 & 0 & <{}^t\tau^\alpha_H> \end{bmatrix} + \begin{bmatrix} <{}^t\tau^\alpha_{11}> & <{}^t\tau^\alpha_{12}> & <{}^t\tau^\alpha_{13}> \\ <{}^t\tau^\alpha_{12}> & <{}^t\tau^\alpha_{22}> & <{}^t\tau^\alpha_{23}> \\ <{}^t\tau^\alpha_{13}> & <{}^t\tau^\alpha_{23}> & <{}^t\tau^\alpha_{33}> \end{bmatrix} \quad (2)$$

$$(<{}^{t}\tau^{\alpha}_{11}> + <{}^{t}\tau^{\alpha}_{22}> + <{}^{t}\tau^{\alpha}_{33}> = 0\,, \qquad (3)$$

which applies to both the stress at a point and averaged stresses, and solving for

$<d_{\phi\psi}>$ gives the equation

$$\begin{aligned}<d^{\alpha}_{\phi\psi}> = {} & <{}^{t}\tau^{\alpha}_{H}> d^{\alpha}_{0} [S^{\alpha}_{2}/2 + 3S^{\alpha}_{1}] - (<{}^{t}\tau^{\alpha}_{11}> <{}^{t}\tau^{\alpha}_{22}>)\, d_0 S^{\alpha}_{2}/2 + d_0 \\ & + <{}^{t}\tau^{\alpha}_{11}> d_0 S^{\alpha}_{2}/2\, (1 + \cos^2\phi)\sin^2\psi \\ & + <{}^{t}\tau^{\alpha}_{22}> d_0 S^{\alpha}_{2}/2\, (1 + \sin^2\phi)\sin^2\psi \\ & + <{}^{t}\tau^{\alpha}_{12}> d_0 S^{\alpha}_{2}/2\ \sin 2\phi\, \sin^2\psi \\ & + <{}^{t}\tau^{\alpha}_{13}> d_0 S^{\alpha}_{2}/2\ \cos\phi\, \sin 2\psi \\ & + <{}^{\tau}\tau^{\alpha}_{23}> d_0 S^{\alpha}_{2}/2\ \sin\phi\, \sin 2\psi \qquad (4)\end{aligned}$$

Written in this way we see that the d-spacing has five terms with different angular dependencies and three terms that are angularly independent. From a collection of $<d^{\alpha}_{\phi\psi}>$ the six deviatoric components of the total stress tensor may be determined by least squares using Eq. 4 and then using Eq. 3. By using Eq. 3 and 4 instead of Eq. 2, we eliminate the need for an accurate d_0 in the analysis (but still need this information about the hydrostatic stresses is needed).

Although there is more to illustrate about these relations, we begin our discussion of the application of these equations at this point, with some new results on thin Al-Cu films, the material used for interconnects in solid state devices. This is a subject of considerable concern, as stresses in such films are believed to play an important role in producing voids and subsequent failure of devices.[6]

Al-Cu Thin Films on Si Wafers

The "d" vs $\sin^2\psi$ is linear so that the σ_{i3} $(i \neq 3) = 0$. From the slopes of this relationship the residual stresses in the film surface were obtained on films of different thicknesses, as shown in Fig. 2[7]. These results are astonishing; the residual stresses are several times larger than the yield stresses of the bulk materials, especially in the passivated films. These stresses are primarily extrinsic, that is they occur due to the difference in expansion coefficients (α) of Si and the alloy ($\frac{\Delta d}{d} = \Delta\alpha\, \Delta T$); in processing (and in use) the films are exposed to temperatures of 350 - 400°C. In fact, stresses of the order of 800 MPa would be expected due to the difference in thermal contraction so that the lower values in Fig. 2, imply that some yielding occurs on cooling. The dislocation densities determined from the peak shape are of the order of 10^{13} m^{-2} only slightly larger than for annealed materials.[8]

There has been some debate as to whether there are strong stress gradients through the film, and some of the theories for void formation require such gradients. However, using glancing angle geometry no gradients were detected

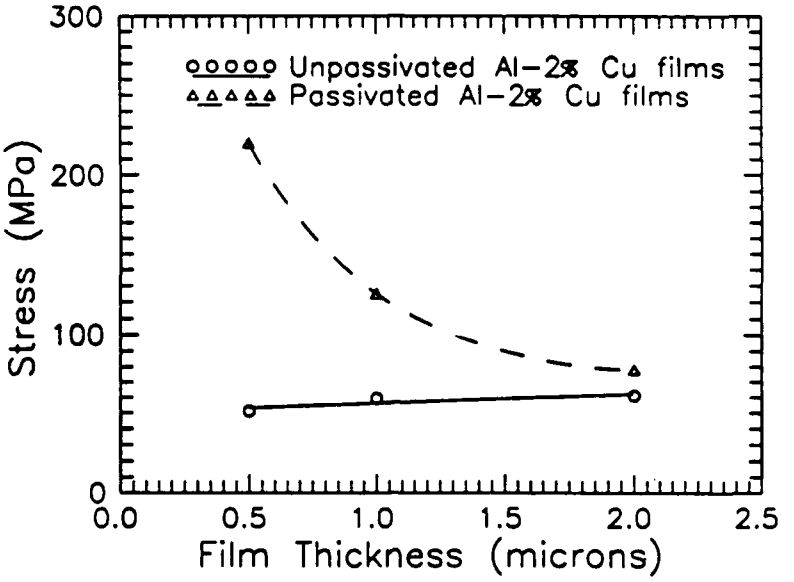


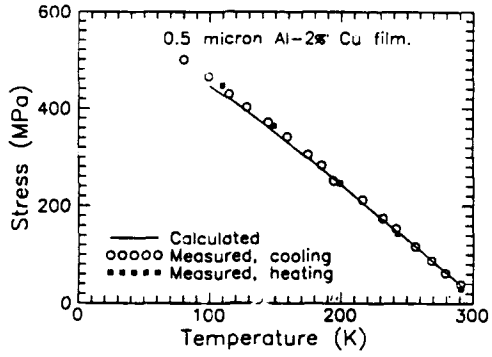
Fig. 2:[7] Total residual stress as a function of film thickness for passivated and unpassivated Al-2% Cu thin films.

[8], Fig. 3.

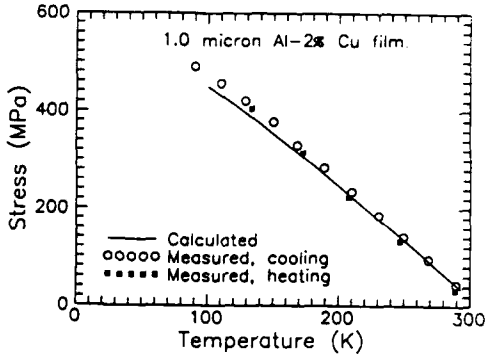
By cooling such films, additional stress can be created and the d spacing continues to change until yielding occurs; as shown in Fig. 4 [9] the yield stress can be determined quite simply in this way if the substrate doesn't yield and, without any special requirements on the film geometry, as is required for other techniques. By comparing the variation in d vs temperature with theory, the strength of the bond between film and substrate can be quantified as in, Fig. 4. (In this figure, the measured values are slightly above the theoretical curve after correcting these results for the temperature variation of the elastic constants. These points were slightly below the theoretical values in Ref. 9, without this correction.) This technique could be particularly useful with particulate or fiber composites, to examine this bonding and without requiring special mechanical pull out tests.

Steel

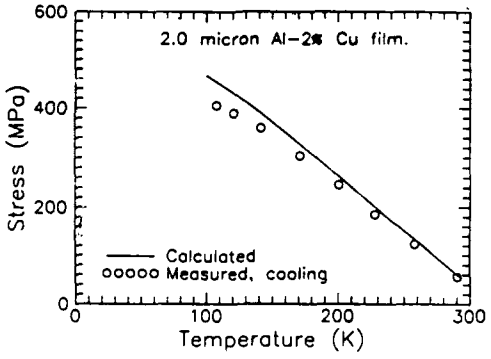
Stress measurements on steel have made for many decades, for a variety of situations, after use or after manufacturing. For the most part, these have been carried out only on the ferrite phase, because of its large volume fraction (compared to the carbide phase(s)). Yet as we will show, some elements of these stresses must balance locally and this can lead to large stresses in the brittle cementite, opposite in sign to that in the ferrite. This could result in conditions that create fracture. The macrostress must (by definition) be the same in all phases and results from differences in deformation on the macro scale, e.g. the difference, say, between the surface and bulk. It is easily shown that ${}^{M}\sigma_{i3} = 0$ for the macrostresses (M) but not for microstresses ${}^{\mu}\sigma_{i3}$, which must balance locally.[10] Thus it is possible to write the total measured stress in any phase as



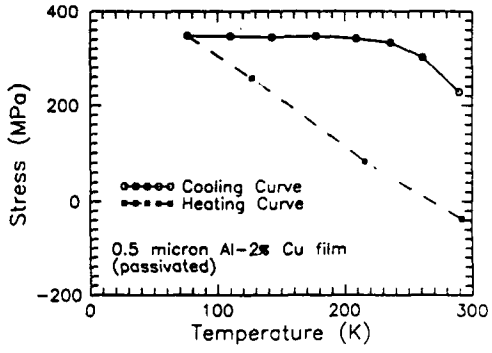
3(a)



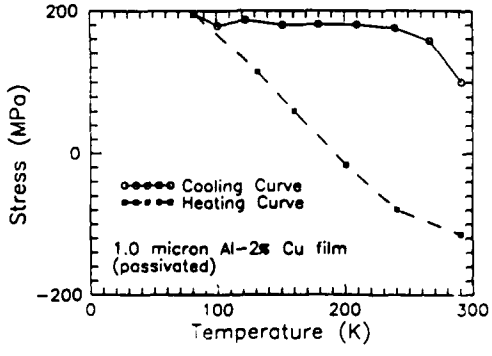
3(b)



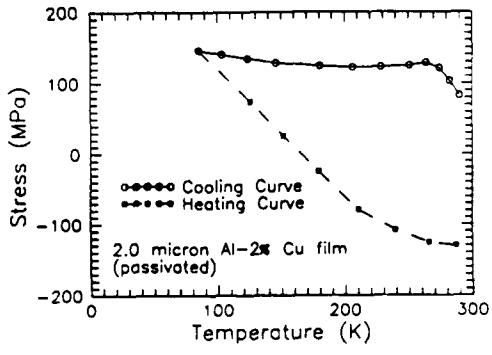
3(c)



3(d)

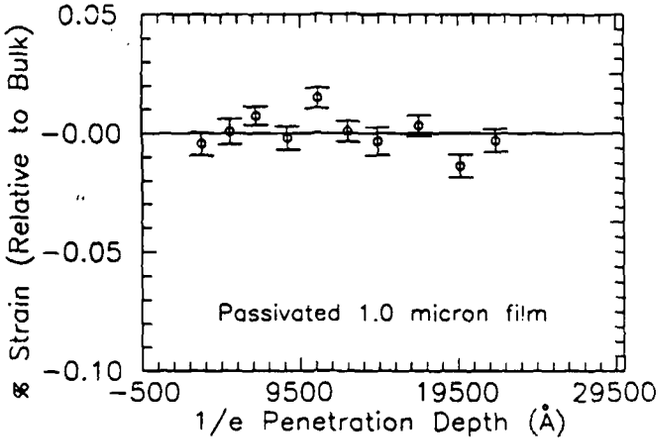


3(e)

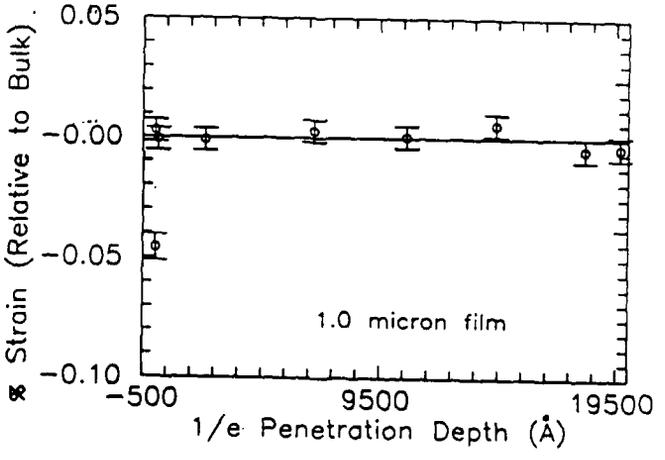


3 (f)

Fig 3: Residual stress versus temperature **a)** unpassivated 0.5μm Al-2% Cu film, **b)** unpassivated 1.0μm Al-2% Cu film, **c)** unpassivated 2.0μm Al-2% Cu film, **d)** passivated 0.5μm Al-2% Cu film, **e)** passivated 1.0μm Al-2% Cu film, **f)** passivated 2.0μm Al-2% Cu film.
(C. Shute, PhD thesis, Northwestern University, 1991.)



4(a)



4(b)

Fig. 4:[8] Relative percent strain (deviation from "bulk") versus calculated 1/e penetration for **a)** 1.0 μm passivated Al-2% Cu thin film, **b)** 1.0 μm Al-2% Cu thin film

(A. Winholtz, PhD thesis, Northwestern University, 1991.)

a sum of these two components[10]:

$$\langle {}^{t}\sigma^{\alpha}_{ij} \rangle = {}^{M}\sigma_{ij} + \langle {}^{\mu}\sigma^{\alpha}_{ij} \rangle \quad (5a)$$

$$\langle {}^{t}\sigma^{\beta}_{ij} \rangle = {}^{M}\sigma_{ij} + \langle {}^{\mu}\sigma^{\beta}_{ij} \rangle . \quad (5b)$$

These equations apply equally to the deviatoric compontents.

As an example of the use of such equations, we show in Fig. 5 the load sharing between the carbide and matrix in fatigue of 1080 steel [11]. Here the applied tensor in fully reverse d fatigue loading was added to the measured deviatroic microstresses.

As our final example, we show in Fig. 6 results on TiC-TiB_2 composite, from Ref. 12. The various terms were separated and the microstresses are shown after fabrication and after fracture. The microstresses clearly decrease sharply during fracture (the macrostresses did not), indicating failure at the particle interfaces, rather than in the matrix. Thus, this diffraction technique might prove to be a valuable tool to sense interface decohesion in composites.

SUMMARY

For over 70 years, diffraction has proved to be a valuable tool for the nondestructive assessment of residual stresses in fabricated parts. Recent developments have extended the range of uses to composite materials and films and we have attempted to show here how gradients, yield stress and bonding in

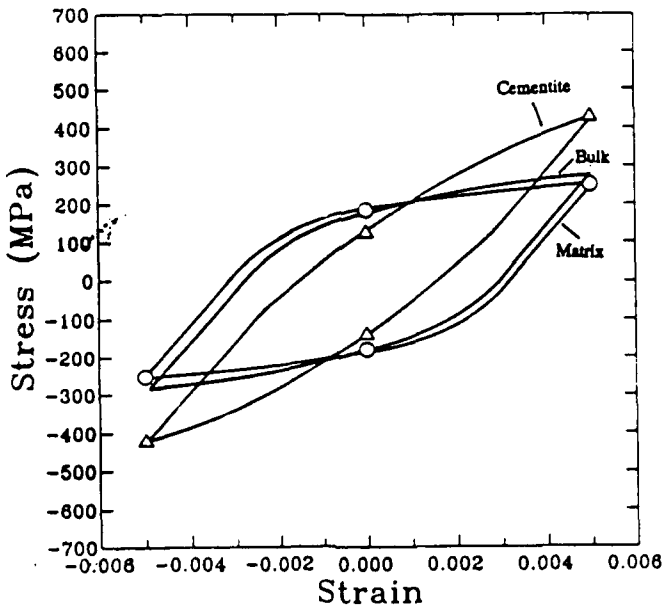


Fig. 5: Hysteresis loop for 1080 steel with components for the matrix and cementite phases.

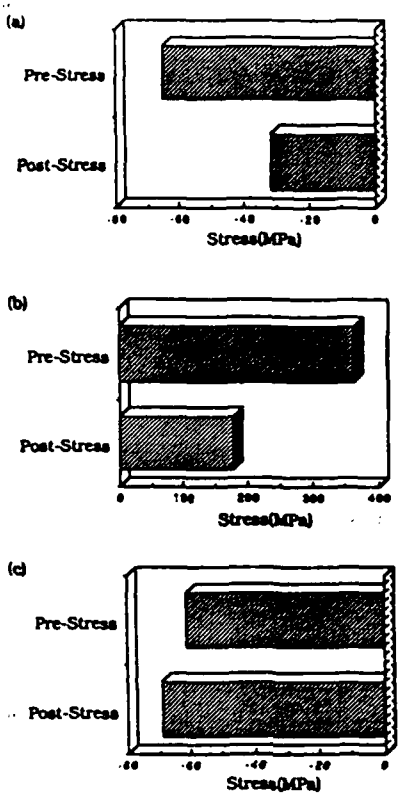


Fig. 6:[12] The "effective" hydrostatic micro $[(\sigma_{11} + \sigma_{22})/2]$ stresses before and after loading in **(a)** the SiC phase in the composite material, **(b)** the TiB_2 phase in the composite, and **(c)** the SiC standard material.

such materials can be studied - again nondestructively and with few limits. But the doors are only just opened. There is much to learn. For example, are residual stresses important for crack nucleation and growth in fatigue, or in only one or the other? Can our knowledge of stresses in films be used to control sources of failure; for example, low temperature cooling of devices may cause plastic yielding and reduce these stresses, at least during device storage after manufacture. Can these techniques be used to sense decohesion in composites? There is much exciting work ahead.

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