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The Tensile Properties of Nickel-Coated Carbon Fibres

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The tensile properties of nickel-coated Grafil "HT" carbon fibres were studied as a function of the coating thickness, taking into account the diameter-dependence of the properties of the fibres themselves. The stress-strain curve exhibited three stages, following from an initially elastic coating which yields and then extends plastically before failure of the fibre. The behaviour could be described by a simple law of mixtures and the grain size of the coatings.

1. Introduction

In a previous paper [1] we presented the results of a study concerned with the dependence of the mechanical properties of Grafil "HT" carbon fibres on their diameter. We now discuss the influence of a thin metal coating on these properties. As a model system we have chosen nickel because it can be readily electroplated in a nominally stress-free condition. However, nickel is known [2] to degrade the fibre properties following heat-treatment at moderate temperatures and this fact limits the usefulness of the model system.

2. Experimental

The carbon fibres were electroplated in a sulphamate bath by two suppliers. The nickel coatings were analysed (using an electron microprobe analyser) to show a cobalt content in the range 8 to 11 wt %. This has the effect [3] of increasing tensile strength and reducing ductility of the nickel but ensures that it can be heat-treated without embrittlement.

The absence of an attack of the fibres by the nickel coating was confirmed (fig. 1), first by etching back the nickel coating and studying the fibre surface, and second by checking a fracture surface as done previously by Jackson and Marjoram [2].

The fibres were separated and tested in simple tension with an Instron machine using the window technique [4], all specimens being 5 cm in gauge length. Considerable difficulty was experienced in separating fibres with *thicker* coatings which consequently suffered more from handling damage, thus making the measured ultimate tensile stress (UTS) subject to error.

The diameter and coating thickness of all tested fibres was measured by mounting them in plastic, sectioning transversely and projecting the image on to the calibrated screen of a projection microscope.

3. Results

We showed previously [1] that both the UTS and Young's modulus of uncoated fibres depend upon their diameter. It was also found that a dispersion of values was obtained (fig. 3) at a given diameter. We have therefore referred the present data to the average values obtained previously at a given fibre diameter.

A stress-strain curve of an as-received coated fibre is compared in fig. 2 with the average properties of the uncoated ones of the same fibre diameter. The following stages are evident:

(I): elastic fibre and coating,
transition (I)-(II): yielding of the coating,
(II): elastic fibre and plastic coating (the slope being the so-called secondary modulus),
(III): fracture.

These are the stages normally observed in a composite material containing brittle fibres [5]. Some specimens fractured before the transition reflecting variations in the fibre UTS as mentioned above.

The interpretation of stage (II) was verified by two observations. First we noted stress relaxation before, but not after, the transition (I)-(II). Second, classical work-hardening was evidenced by the

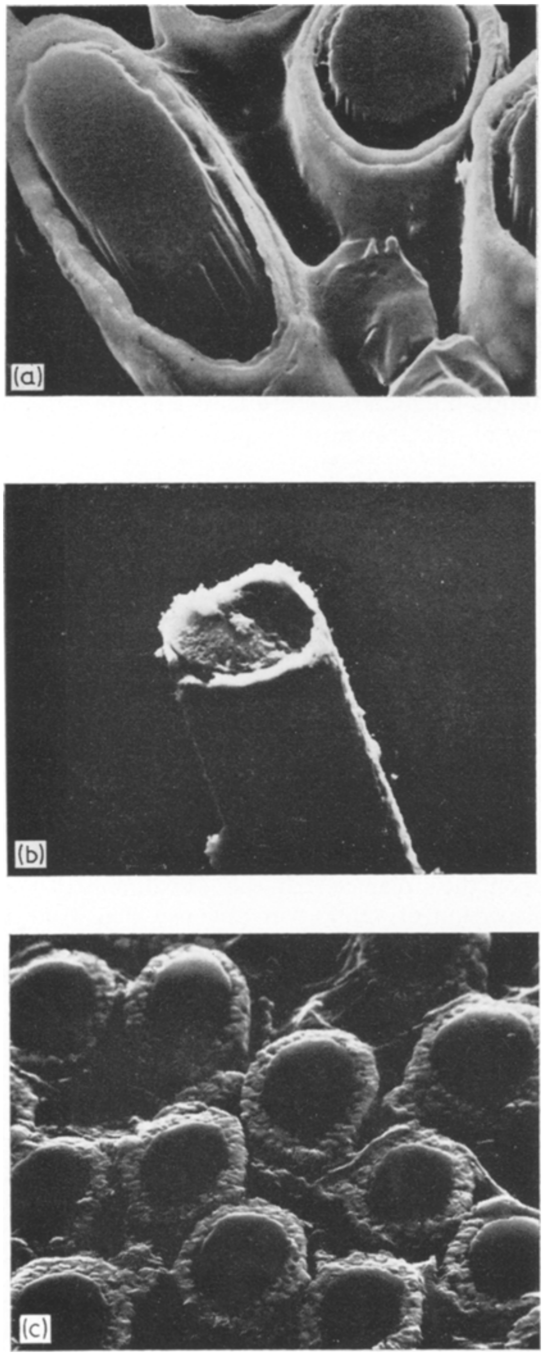


Figure 1 Scanning electron micrographs of as-received nickel-coated carbon fibres: (a) a transverse section from which the nickel has been partially etched away (× 1850), (b) a fracture surface (× 1420), showing the lack of effect of the nickel on the surface or interior structure of the fibres. Also shown, in (c), is the effect of heat-treatment at 600° C (× 1470).

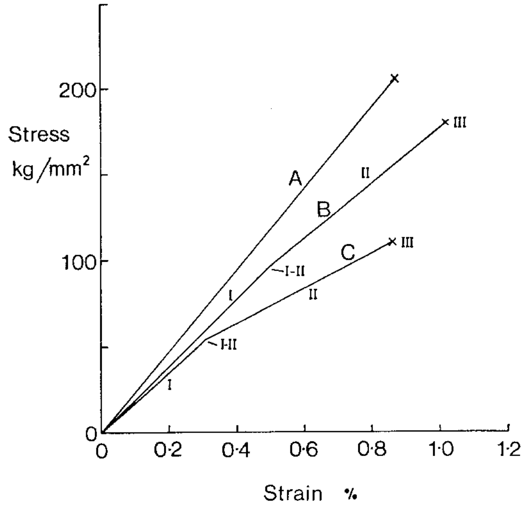


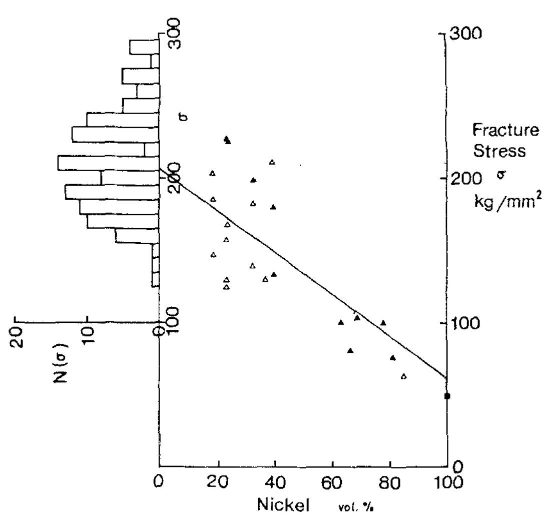
Figure 2 The effect of a volume fraction of 39.1% nickel (curve B) on the average properties of uncoated carbon fibres [1] of the same $9\frac{1}{3}$ μm diameter (curve A). Curve C shows the effect of a heat-treatment of 1 h at 600° C; volume fraction 45.9% nickel, fibre diameter 9 μm. The three stages observed in tensile experiments on coated fibres are indicated.

raised yield point following loading beyond the transition, unloading and reloading.

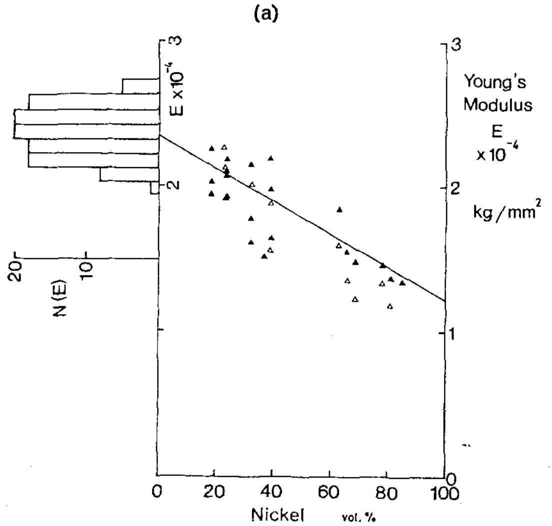
In fig. 3a-c are given the UTS, Young's and secondary modulus, and the fracture strain of as-received coated fibres of a given typical diameter as a function of the volume fraction of nickel in the coated fibre. A histogram of the data obtained previously [1] for uncoated specimens is included on the ordinate of each graph.

The microstructure of the nickel coating was examined, as shown in fig. 4a, and found to be fine-grained with a grain size comparable with that reported by Saleem *et al* [6] in nickel also deposited from a sulphamate bath. The nickel could be readily recrystallised (fig. 4b) by heat-treating for 1 to 2 h at 600° C, following Köster [7]. From the results given by Jackson and Marjoram [2], this treatment was expected to be too short for the nickel-carbon fibre interaction discussed by them; while we noted a slight drop in Young's modulus (some 10%), this interaction does not affect the yield point of the nickel which is the aspect of interest to us.

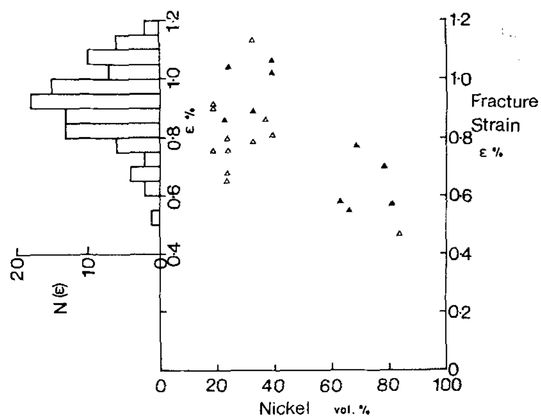
The effect of recrystallisation on the mechanical properties is included in fig. 2; graph C in this figure indicates both the lowered yield point and also the retention of its ductility by the nickel (presumably because of its cobalt content as mentioned above).



(a)



(b)



(c)

Figure 3 The mechanical properties of nickel-coated carbon fibres as typified by $9\frac{1}{3}$ μm diameter fibres tested at 10^{-2} min^{-1}. The data are: (a) fracture stress; the full and open symbols refer to samples in which the coating did and did not yield before fracture, respectively, (b) Young's and secondary modulus (full and open symbols) (c) fracture strain; full symbols refer to samples which exhibited stage II, as a function of the volume fraction of nickel. The straight lines in (a) and (b) are least-square fits calculated including the data for uncoated fibres obtained previously [1]. The square datum in (a) is the fracture stress of electrodeposited nickel according to Köster [7]. Included on each ordinate are the histograms from earlier work [1] appropriate to uncoated fibres of $9\frac{1}{3}$ μm diameter.

4. Discussion

4.1. Stage I

The Young's modulus of coated fibres for thin coatings (i.e. $C_2 \ll 1$) is given by the relation derived from the linear theory of elasticity [8]:

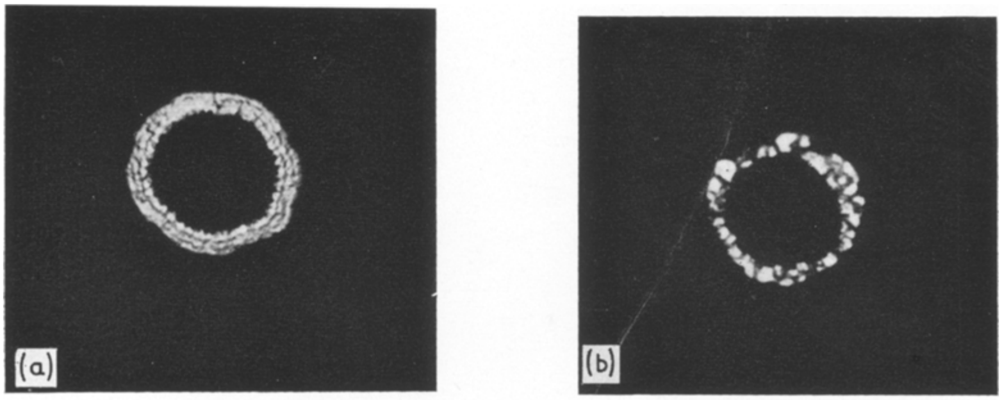
$$\frac{E}{E_{\text{law of mixtures}}} = \frac{E_1 + E_2}{E_1} \cdot \frac{(\nu_1 - \nu_2)^2}{1 - \nu_2^{\,2}} \cdot C_2 \quad (1)$$

where E, ν and C are Young's modulus, Poisson's ratio and volume fraction and the suffixes 1 and 2 refer to the carbon fibre and nickel coating respectively. Equation 1 predicts little deviation from the law of mixtures, independently of the value of ν_1 which has not been determined to date. The data in fig. 3b adhere quite closely to a straight line which extrapolates to 1.23×10^4 kg/mm² at 100% Ni (averaged over all fibre diameters). The line is a least squares fit calculated with the inclusion of the data which compose the histogram of uncoated fibres. If the data are analysed excluding the uncoated results, then the derived modulus for nickel is 1.46×10^4 kg/mm². These values can be compared with the range 1.5 to 2.1×10^4 kg/mm² given in the ASM handbook [9] for massive nickel; it might be remarked that D'Antonio *et al* [10] concluded that the modulus of (fine-grained) vapour-deposited thin nickel films does not differ from that of bulk nickel.

4.2. Stage II

It is apparent that the nickel coating retains the facility to be work-hardened because the observed values for the secondary modulus E_{II} extrapolate to a finite value at 100% nickel, this value being the work-hardening rate [11].* However the paucity of experimental data

*$(E_{\text{II}})_\epsilon = E_1 V_1 + \left(\frac{d\sigma}{d\epsilon}\right)_{\epsilon,2} V_{\text{Ni}}$, referring to strain value ϵ [11].



(a) (b)

Figure 4 Optical micrographs × 1500 of nickel coatings (a) as-received and (b) after 1 h at 600° C. The annular "super-structure" of the nickel coating in (a) is also apparent in fig. 1a.

renders very approximate any analysis to give a value to the work-hardening rate. Nevertheless the value is clearly quite high and corresponds to the rate immediately after yielding and at very low plastic strain. It does not correspond to the work-hardening rate of bulk nickel strained several per cent, which is of the order $E/100$.

It is interesting to contrast the present coatings with vapour deposited films which show [10] little capacity for work-hardening and, because cobalt does not affect this [12], we attribute the difference to a grain-size effect. A value of the latter was not reported by D'Antonio *et al* [10] but discussion of their later work [13] by Tarshis and Wilshaw [14] implies a value of about 1000 Å, which is smaller than the 0.3 to 1 μm in the present work.

4.3. Transition I-II

The transition I-II is attributed to the yielding of the nickel coating. The yield stress of the coating σ^* is related to the yield stress in simple tension, σ_y, through the relation derived from the linear theory of elasticity coupled with the von Mises yield criterion:

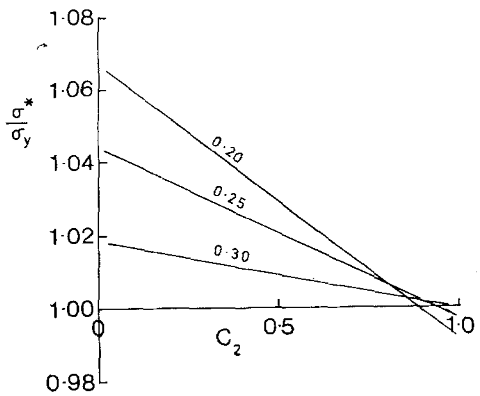


Figure 5 The ratio of the yield stress of the coating σ^* to its yield stress in simple tension σ_y as a function of coating thickness calculated from (2) for a range of values of ν_1. The values of ν_2, E_2 and E_1 were set respectively to $\frac{1}{3}$, 1.46×10^4 kg/mm² (from the present work) and 2.4×10^4 kg/mm² (as appropriate to 9 μm diameter fibres [1]). Note that if $\nu_1 = \frac{1}{3}$, the curve coincides with the abscissa.

series of Poisson's ratios. It is evident that relatively little change in the yield point is anticipated, hence this effect has been neglected in the present discussion.

$$\frac{\sigma^*}{\sigma_y} = \frac{(C_1E_1 + C_2E_2)}{E_2}(1+\nu_2)\frac{1 + (1-2\nu_2)(C_1 + QC_2)}{\sqrt{3(\nu_1-\nu_2)^2 + \{(1+\nu_2) + (1-2\nu_2)[(1+\nu_1) + Q(1+\nu_2)C_2]\}^2}} \quad (2)$$

with

$$Q = \frac{(1-2\nu_1)(1+\nu_1)}{(1-2\nu_2)(1+\nu_2)}\frac{E_2}{E_1}$$

where a difference in Poisson's ratio is necessary to affect the yield point. As remarked above, the ratio is not known for the carbon fibres, hence equation 2 has been evaluated (fig. 5) for a series of Poisson's ratios.

The yield point of the coating deduced from the present data (by taking the *mean* Young's modulus at each fibre diameter to account for the load carried by the fibres themselves) is given in fig. 6 as a function of (coating thickness)$^{-1/2}$. This form of presentation was selected to allow comparison with the Petch relation [15]

$$\sigma_y = \sigma_0 + kd^{-1/2}$$

where d is the grain diameter. No experimental investigation of the Petch relation for nickel was found in the literature, hence we proceeded in the following way. A straight line of slope k equal to 0.08×10^{-3} $mm^{1/2} \times \mu$ (μ being the shear modulus) as indicated for the f.c.c. metals copper, aluminium and silver [16] was adopted and applied to data on nickel from Macherauch and Vöringer [17]. The increase in yield point caused by cobalt was introduced by taking a line parallel to the above in accordance with data cited by Parker and Hazlett [12].

The adoption of the mean modulus of the fibres themselves at the appropriate diameters introduces a serious approximation into our analysis, which is more significant at lower coating thickness and probably accounts for the wide scatter of data. A comparison between our results and the constructed Petch curve can only be regarded as semiquantitative at best, but nevertheless the present yield point values appear to be too high. The discrepancy is attributed to handling damage coupled with the ability to work-harden the coating remarked previously.

In an attempt to confirm the latter point we heat-treated coated fibres at 600° C for 1 and 2 h in order to recrystallise the coating (fig. 3). The yield strain was thus reduced (fig. 2) and the yield stress of about 25 kg/mm² agrees reasonably with that predicted by the Petch curve in fig. 6 at a grain size in the region of 1 μm.

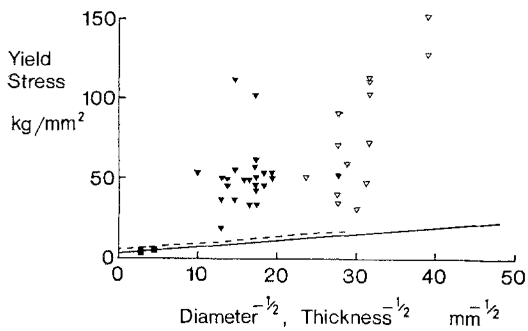


Figure 6 A superposition of the present yield point data as a function of (coating thickness)$^{-1/2}$ on a Petch plot derived as discussed in the text; the full and dashed lines are the nickel and nickel/10 wt% cobalt respectively. The square data points are taken from Macherauch and Vöringer [17]. The open and full symbols refer to specimens taken from tows plated by the two suppliers.

4.4. Stage III

The fracture strain values are shown in fig. 3c for one fibre diameter. Allowing for the handling problems experienced with the thicker coatings, the scatter of data is comparable with that of uncoated fibres (see the histogram on the ordinate of fig. 3c). Fracture is thus considered as being initiated in the fibres and is not affected by the coating. The latter retains its ductility as can be seen in fig. 1b.

5. Conclusions

The mechanical properties of nickel-coated carbon fibres in simple tension have been studied. It has been shown:

(1) The Young's modulus (i.e. before yielding of the coating) can be described by a law-of-mixtures relation.

(2) The high yield point of the nickel coating can be explained in terms of its grain-size.

(3) The fracture of the coated fibres is governed by the fibres themselves and is unaffected by the metal coating.

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