# Effect of irradiation on the mechanical properties of commercially produced Nb<sub>3</sub>Sn tapes

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The effect of neutron irradiation on the mechanical properties of commercially produced Nb<sub>3</sub>Sn tapes has been investigated using scanning electron microscopy, sound velocity measurements, tensile and three-point bend tests. The elastic moduli show an increase on irradiation which is independent of dose in the range  $4 \times 10^{21}$  to  $4 \times 10^{23}$  neutrons m<sup>-2</sup>. The majority of the Nb<sub>3</sub>Sn tapes show no evidence of significant amounts of plastic deformation prior to failure, which occurs by the intergranular fracture of the Nb<sub>3</sub>Sn layers followed immediately by ductile overload failures of the niobium core. The latter changes to a more brittle failure on irradiation and in tapes containing  $ZrO_2$  particles. The fracture stress decreases for doses up to  $\sim 10^{23}$  neutrons m<sup>-2</sup> but increases at higher doses. Irradiation reduces the critical stress intensity factor  $K_c$ , but  $K_c$  and the fracture stress are increased in tapes containing  $ZrO_2$ . These results are discussed in terms of various microstructural features and previously determined radiation damage.

## 1. Introduction

The superconducting compound  $Nb_3Sn$ , in common with other A15 compounds, is hard and brittle. The brittle nature of  $Nb_3Sn$  presents problems of fabrication and the possibility of inservice failure from the forces encountered in cryogenic service, e.g. hoop stresses, axial compression of adjacent coils, and so on. For these reasons a variety of techniques have been developed for the production of composite  $Nb_3Sn$ superconductors which exhibit better mechanical behaviour than the A15 compound in bulk form. The two most important composite superconductors are multifilamentary and tape composites.

A typical multifilamentary composite consists of continuous filaments (3 to  $15 \mu m$  diameter) with an unreacted niobium core surrounded by a layer of Nb<sub>3</sub>Sn set in a bronze matrix. The mechanical properties of multifilamentary Nb<sub>3</sub>Sn have been extensively investigated. It has been shown that compressive stresses develop in the Nb<sub>3</sub>Sn on cooling from the production temperature due to the small thermal expansion coefficient of Nb<sub>3</sub>Sn compared to that of the bronze matrix [1-3]. It follows that a substantial external tensile stress may be applied to the composite before the stress on the Nb<sub>3</sub>Sn changes from compressive to tensile and hence a multifilamentary composite can experience a considerable strain, i.e. > 0.5% [3-7], before cracking occurs in the Nb<sub>3</sub>Sn layers. Furthermore, it has been found [3, 7] that the unreacted niobium core acts as a crack stopper and the critical step in the mechanical failure of the composite is not the fracture of the Nb<sub>3</sub>Sn but the failure of the niobium cores. Thus the volume fraction of unreacted niobium is critical in determining whether the multifilamentary composite is brittle or ductile [7].

Tape composites are flexible, i.e. may be bent, and are produced either by chemical vapour deposition or by diffusion of tin into a niobium substrate. Other metals, such as copper or stainless steel, may be incorporated into the composite for quench protection. In contrast to multifilamentary Nb<sub>3</sub>Sn the mechanical properties of tape composites have received scant attention. Benz and co-workers have studied Nb<sub>3</sub>Sn tape composites which contained layers of copper, stainless steel and solder [8-10]. Because of these additional metal layers, a compressive stress developed in the Nb<sub>3</sub>Sn in a similar manner to that in the multifilamentary composites. However, failure strains were not reported and the failure stresses quoted were determined by measuring the stress to cause a sudden voltage increase in a sample carrying a current equal to 95% of the critical current in a field of 50 kG.

One of the most important large-scale and longterm applications envisaged for Nb<sub>3</sub>Sn superconducting composites is in the coils of the plasma confining magnets of fusion reactors. During service, the Nb<sub>3</sub>Sn will inevitably be irradiated with thermal and fast neutrons. Neutron irradiation of Nb<sub>3</sub>Sn produces small regions of disorder [11, 12] and a low concentration of dislocation loops [12]. These irradiation-induced microstructural features, particularly the disordered regions, degrade the superconducting properties [12, 13] and there may also be a concomitant change in mechanical behaviour as mechanical properties are usually structure sensitive. To the authors' knowledge the only publication on any A15 compounds on the effect of irradiation on the mechanical properties is that by Guha et al. on the elastic properties of  $V_3$ Si [14]. These workers found that a dose of  $2.2 \times 10^{23}$  neutrons m<sup>-2</sup> increased the elastic moduli  $C_{11}, C_{44}$ , and the ratio  $(C_{11}, C_{12})/C_{44}$ .

This paper reports the results of an investigation of the mechanical properties of  $Nb_3Sn$  tapes in unirradiated and irradiated conditions. A comprehensive survey of the mechanical behaviour was carried out and included the determination of the elastic properties, the tensile fracture stress and the fracture toughness.

# 2. Experimental procedure

#### 2.1. Material and irradiation

The tapes used for the investigation were supplied by AERE Harwell, who also carried out the neutron irradiation. Full details of the tape production and the irradiation procedure are given in [13] and [15] and only a summary is given here. Most of the tapes were produced from niobium and niobium-ZrO<sub>2</sub> tape substrates approximately  $10\,\mu$ m thick and 7 mm wide which were coated with tin by passing through a molten tin bath at  $620^{\circ}$  C in about 1 min, maintained at that temperature for 8 min and cooled down in 1 min. A few different tapes, which were produced by a similar

TABLE I Tape composites used for mechanical testing

Code	Width (mm)	Thickness* (µm)	
D	7	12	
E	7	10 (detinned, pre-annealed 5 h 1150° C)	
ZrO,	7	13	
PZrO₂N	2.35	28	
PNbN	2.35	28	
PZrO <sub>2</sub>	7	14	
$CZrO_{2}$	7	14 (30 with copper)	
Niobium	7	10 (cold-worked and after 8 min at 1000° C)	

\*Includes excess tin.

production schedule, were also supplied; these were a copper-backed Nb– $ZrO_2$  substrate tape, a thicker Nb– $ZrO_2$  substrate tape and narrow (2.35 mm) Nb and Nb– $ZrO_2$  substrate tapes, designated CZrO<sub>2</sub>, PZrO<sub>2</sub>, PNbN and PZrO<sub>2</sub>N respectively.

For the irradiation treatment the tapes were canned under an argon atmosphere and irradiated to fast neutron (n) doses in the range  $3.7 \times 10^{21}$  to  $5.4 \times 10^{23}$  n m<sup>-2</sup> (defining fast neutrons as those with energies greater than 0.7 MeV). It is unlikely that the temperature exceeded 70° C for most of the irradiation treatments.

Some of the tape produced using the niobium substrate was detinned and given a pre-anneal of 5 h at  $1150^{\circ}$  C before irradiation (designated E). Also available for testing was the niobium substrate in the as-received, heavily cold-worked condition and also after 8 min heat treatment at  $1000^{\circ}$  C to simulate the heat cycle experienced during tape production. The heat treatment, irradiation history, and dimensions of the tapes are summarized in Table I.

## 2.2. Mechanical tests

The Young's modulus was determined using 2 to 3 cm lengths of the tapes in a three-point bending rig connected to a microbalance. This technique has been fully described by Bussiere *et al.* [16] and their analysis gives the following equation for the Young's modulus (E) of the tape composite:

$$F\left(x^2 + \frac{15}{8}y^2\right) = \frac{4EI\sin\theta}{1 - \nu^2} \tag{1}$$

where F is the applied force, 2x is the span, y is the vertical displacement,  $\theta$  the angle of deflection, v is Poisson's ratio and I the second moment of the cross-sectional area of the specimen. Values of y and  $\theta$  were obtained as a function of load and graphs of  $F(x^2 + 15y^2/8)$  against  $\sin \theta$  plotted. The slope of the graphs was  $(4EI)/(1 - \nu^2)$  and hence E was evaluated knowing that  $I = (wt^3)/12$ , where w and t are the width and thickness of the tape respectively and putting  $\nu = 0.35$ .

Standard velocity of sound measurements for surface waves were used to determine the shear modulus,  $\mu$ , from the equation:

$$c_{\rm s} = \frac{0.87 + 1.12\nu}{1+\nu} \left(\frac{\mu}{\rho}\right) \tag{2}$$

where  $c_s$  is the surface wave velocity and  $\rho$  is the density of the specimen. The density value employed in the calculation was  $8650 \text{ kg m}^{-3}$  which is intermediate between that for Nb<sub>3</sub>Sn ( $8860 \text{ kg m}^{-3}$ ) and niobium ( $8570 \text{ kg m}^{-3}$ ).

Initially miniature tensile specimens of 4 mm gauge width and 16 mm gauge length were produced, but it was found that identical tensile data were obtained from lengths of the tape without a gauge section and hence most of the results quoted are for the latter type of specimen with 3 cm of tape between the grips. A few tests were carried out with only 2 mm of tape between the grips. This reduced specimen length enabled transverse sections as well as the normal longitudinal sections of the tapes to be tested. All tests were performed at room temperature in a JJ Instruments tensile tester at a crosshead speed of  $0.5 \,\mathrm{mm\,min^{-1}}$ . For some of the tests a small lead zirconium titanate transducer was coupled to the specimen and the output monitored with commercially available acoustic emission equipment.

The effect of a flaw in the tape on the mechanical behaviour was investigated by inserting cracks of lengths in the range 0.4 to 1.6 mm on one edge of the specimens prior to tensile testing. A few specimens were also tested with centre rather than edge cracks. Various methods for producing the cracks were tried and the most successful was found to be cutting with a sharp scalpel.

After testing of the unflawed specimens the specimen surface and the fracture surface were examined in a scanning electron microscope.

## 3. Results

The elastic moduli of niobium tape were measured in order to assess the accuracy of the techniques employed. The experimental values obtained were  $32.4 \pm 2.1$  GN m<sup>-2</sup> and  $106.8 \pm 1.9$  GN m<sup>-2</sup> for the shear modulus and Young's modulus respectively, which are in good agreement with the generally accepted values.

The effect of neutron irradiation on the elastic moduli of the tape composites D, E and  $ZrO_2$ is shown in Fig. 1. No difference was detected in the shear modulus of tapes E and ZrO<sub>2</sub> after irradiation. Only tape D was available for testing in the unirradiated state. It can be seen that for tapes E and ZrO<sub>2</sub> irradiation increased the shear modulus with respect to that of unirradiated D tape by an amount that was independent of dosage over the range  $3 \times 10^{21}$  to  $4 \times 10^{23}$  nm<sup>-2</sup>. The Young's modulus results exhibited more scatter and the values for irradiated ZrO2 were perhaps slightly lower than those for irradiated tape D. Nevertheless, it is clear that irradiation increased the Young's modulus of tape D and, as observed for the shear modulus, the increment was independent of dosage up to  $4 \times 10^{23}$  nm<sup>-2</sup>. A higher value for the Young's modulus was recorded for tape D after a dose of  $5.4 \times 10^{23}$  n m<sup>-2</sup>.

Some plastic deformation prior to failure was observed for the niobium tape heat-treated for 8 min at 1000° C, but the only Nb<sub>3</sub>Sn tape to show any evidence of plastic deformation before fracture was the copper-backed tape composite  $(CZrO_2)$  and even then the plastic deformation was extremely limited (< 0.2%). The copperbacked tape was not monitored with the acoustic emission equipment. For those composite tapes which were monitored, no acoustic emission was detected prior to the catastrophic failure of the specimens. The as-produced tapes had a thin surface layer of excess tin [12], but tests on unirradiated D tapes with and without the tin layer gave fracture loads of  $31.7 \pm 2.5$  N and  $32.7 \pm 0.9$  N, respectively, which demonstrated that the tin did not affect the fracture load. Therefore, all fracture stresses reported in this paper were calculated on the cross-sectional area of the Nb<sub>3</sub>Sn/Nb/Nb<sub>3</sub>Sn whether the tape had been detinned or not. The values so calculated were  $452 \pm 36 \text{ MN m}^{-2}$  and  $467 \pm 13 \text{ Mn m}^{-2}$  for asproduced and detinned tapes, respectively.

The tests on the 2 mm longitudinal lengths of D tape produced fracture stresses which were only marginally less than those found for the normally used 3 cm lengths. This demonstrated that the small gauge length specimens were adequate for the investigation of the strength of the tape in the longitudinal and transverse direc-



Figure 1 Effect of neutron irradiation on the shear and Young's moduli of tapes with niobium (D, E) and Nb- $ZrO_2(ZrO_2)$  substrates. The full lines are the average values for the moduli in the unirradiated and irradiated conditions and the dotted lines ± one standard deviation. Irradiated tapes D and E,  $\circ$ . Irradiated tape  $ZrO_2$ ,  $\Box$ .

tions. The fracture stresses obtained using small specimens were  $420 \pm 28 \text{ MN m}^{-2}$  and  $411 \pm 45 \text{ MN m}^{-2}$  for the longitudinal and transverse directions, respectively, and hence it was concluded that there was no effect of orientation.

The effect of irradiation on the fracture stress of tapes D, E and  $ZrO_2$  is illustrated in

Fig. 2. Irradiation doses up to and including  $1 \times 10^{23}$  n m<sup>-2</sup> reduced the fracture stress of D and E from  $459 \pm 28$  MN m<sup>-2</sup> in the unirradiated condition to  $407 \pm 57$  MN m<sup>-2</sup>. There appeared to be an increase in the fracture stress at the highest dose of  $3.15 \times 10^{23}$  n m<sup>-2</sup>. Another niobium substrate tape was investigated in the





TABLE II Fracture stress of Nb<sub>3</sub>Sn tapes

Material	Fracture stress (MN m <sup>-2</sup> ) $452 \pm 36$
D unirradiated, as-produced	
D unirradiated, detinned	467 ± 13
D unirradiated, as-produced and detinned	459 ± 28
D unirradiated, 2 mm, longitudinal	$420 \pm 28$
D unirradiated, 2 mm, transverse	411 ± 45
D, E irradiated	419 ± 64
PNbN irradiated	283 ± 45
CZrO <sub>2</sub> irradiated	873 ± 118
ZrO, irradiated	1108 ± 133
$PZrO_2$ , $PZrO_2N$ irradiated	1139 ± 119
Nb cold-worked	698 ± 37
Nb 8 min at 1000° C	465 ± 40

irradiated condition, namely PNbN, and this tape had a fracture stress of  $283 \pm 45 \,\mathrm{MN \, m^{-2}}$ , i.e., less than that for the D and E materials. The fracture stresses of the ZrO<sub>2</sub> tape were much greater than the niobium substrate tapes at all doses and gave an average of  $1108 \pm 133$  MN m<sup>-2</sup>. This average includes the values at  $3.15 \times 10^{23}$  nm<sup>-2</sup> as, unlike for E, there was no evidence of an increase in fracture stress at the highest dose. The results from the other irradiated ZrO<sub>2</sub>-containing tapes (PZrO<sub>2</sub> and PZrO<sub>2</sub>N) were in good agreement with that for  $ZrO_2$  with an average of  $1139 \pm 119 \text{ MN m}^{-2}$ . The fracture stress of the copper-backed CZrO<sub>2</sub> tape was calculated to be  $349 \pm 39 \text{ MN m}^{-2}$  when the thickness of the copper was included in the cross-sectional area. However, basing the calculation of the fracture stress on the cross-sectional area of the Nb<sub>3</sub>Sn plus unreacted substrate gave a value of the same order as the other ZrO<sub>2</sub>-bearing tapes of  $873 \pm 118 \,\mathrm{MN \, m^{-2}}$ . Thus the copper backing has no effect on the fracture stress but, as mentioned previously, unlike the other composite tapes there was a small amount of plastic deformation prior to failure and it could just be detected in the load-displacement curves that failure occurred in two stages separated by a load drop.

Tensile tests on cold-worked niobium substrate tape and the same tape after the simulated reaction heat treatment of 8 min at  $1000^{\circ}$  C yielded fracture stresses at  $698 \pm 37$  MN m<sup>-2</sup> and  $465 \pm 40$  MN m<sup>-2</sup> respectively. The latter in particular exhibited a considerable amount of ductility. All fracture stress data are presented in Table II.

Scanning electron microscope examination of the surface of as-produced and irradiated tensile

specimens that had been unloaded prior to fracture revealed that no microcracking of the Nb<sub>3</sub>Sn layer had taken place. A similar examination after failure detected microcracking only within  $40\,\mu m$ of the fracture surface but not elsewhere along the gauge length (Fig. 3). The substrate and the Nb<sub>3</sub>Sn layers could be clearly distinguished on the fracture surfaces and for all tapes the fracture in the Nb<sub>3</sub>Sn was intergranular (Fig. 4). The unirradiated niobium substrates failed in a ductile shear mode giving a "chisel fracture" (Fig. 5), whereas the irradiated niobium substrates, and even more markedly the irradiated Nb-ZrO<sub>2</sub> substrates, exhibited more brittle fracture characteristics (Fig. 6). Fig. 7 shows failure of the coatingcomposite interface in tape CZrO<sub>2</sub>.

The stress-displacement curves for the specimens with introduced cracks were linear up to the fracture stress and then fell rapidly to zero as the crack propagated catastrophically. The only exception to this behaviour was the heat-treated niobium tape where the crack propagated slowly by ductile tearing. The failure stresses  $\sigma_f$  for specimens with edge cracks of varying lengths are given in Fig. 8. It can be seen that  $\sigma_f$  decreased with increasing crack length in accordance with the standard linear elasticity fracture mechanics equation for the stress intensity factor K of a plate specimen with an edge crack of length a, namely:

$$K = 1.1\sigma(\pi a)^{1/2}$$
(3)

where  $\sigma$  is the applied stress. Inserting the fracture stresses into this equation to obtain the critical stress intensity factor  $K_c$  gave the following mean values in  $MN m^{-3/2}$ , cold-worked niobium  $21.6 \pm 1.9$ , unirradiated D  $19.3 \pm 2.6$ , irradiated D 15.5  $\pm$  3.3, and irradiated ZrO<sub>2</sub> 25.0  $\pm$  2.3. The few specimens tested with centre cracks yielded similar, but slightly smaller, values for  $K_c$ . The full curves in Fig. 8 were the  $\sigma_f$  values computed from Equation 3 using the mean  $K_{\rm e}$  from the edge crack specimens and the dotted lines the computed  $\sigma_{f}$  values from  $K_{c} \pm$  one standard deviation. The difference in behaviour of the four tapes investigated is best shown by Fig. 9, where the full curves from Fig. 8 are combined on a single graph.

#### 4. Discussion

The increase in the Young's modulus and the shear modulus of the tape composites after irradiation



Figure 3 Scanning electron micrograph of tape D showing microcracking of the Nb<sub>3</sub>Sn confined to a region  $40 \,\mu\text{m}$  from the fracture surface.

is assumed to be due to an anomalous increment in the moduli of the Nb<sub>3</sub>Sn rather than the substrate as there is no evidence for irradiation producing a significant change in the elastic behaviour of metals. The main structural features induced in Nb<sub>3</sub>Sn by irradiation are small regions of disorder and these are likely to be responsible for the observed elastic measurements. However, for most ordered solid solutions and intermetallic compounds the moduli fall with decreasing degree of long range order although there are some exceptions namely bcc Fe<sub>3</sub>Al [17, 18] and hexagonal Cd<sub>3</sub>Mg [19]. The present results suggest that A15 compounds are also an exception to the normal rule and this is confirmed by the V<sub>3</sub>Si single crystal data of Guha et al. [14]; calculation of E and  $\mu$  for a polycrystalline aggregate from their single crystal data using the Voigt-Reuss-Hill averages gives an increase of several per cent on irradiation.

The absence of acoustic emission at stresses less than the fracture stress and the direct observations in the scanning electron microscope prove that no stable microcracks form in the Nb<sub>3</sub>Sn prior to failure in a tensile test. This behaviour, which is in marked contrast to that of filamentary composites where copious microcracking can take place, may be explained as follows. The fracture stress of the unirradiated tape D was  $467 \text{ MN m}^{-2}$ and the Young's modulus was 118.8 GN m<sup>-2</sup>, hence the strain at fracture was  $4 \times 10^{-3}$ . Knowing that the Young's modulus of the niobium substrate was  $106.8 \,\mathrm{GN}\,\mathrm{m}^{-2}$  and that the tape consisted of a niobium core of  $4 \mu m$  and Nb<sub>3</sub>Sn layers of  $3 \mu m$ thick, it can be shown that just before failure approximately one-third of the load was taken by the niobium core and the rest by the Nb<sub>3</sub>Sn. The stress on the niobium core at this stage was  $378 \text{ MN m}^{-2}$ , which was below the measured fracture stress of 465 MN m<sup>-2</sup> for annealed niobium tape. When the brittle Nb<sub>3</sub>Sn failed all the load was transferred to the niobium core and for the crack to have stopped the niobium core would have had to sustain a stress of  $1168 \text{ MN m}^{-2}$ . This was not feasible and the niobium core immediately failed by overload in a ductile manner.

Irradiation would have increased the fracture stress of the niobium core and also embrittled it. Irradiated niobium tape was not tested in the present investigation, but the results of Kayano



Figure 4 Scanning electron micrograph of (a) tape D and (b) tape E showing the intergranular nature of the fracture in  $Nb_3Sn$ .

and Yajima [20] indicate that the fracture stress is about doubled by high neutron doses. Thus, a reasonable estimate for the fracture stress of the niobium core after irradiation would be  $920 \text{ MN m}^{-2}$ . Assuming that the irradiation had a negligible effect on the Young's modulus of niobium and repeating the previous calculation, it was again demonstrated that the niobium core was incapable of supporting the transferred load when the Nb<sub>3</sub>Sn cracked. Therefore, as experimentally found, no stable microcracks were formed in the irradiated tape and the niobium core, which was embrittled, failed by overload in a brittle manner.

The fracture stress of the tape was greatly increased by the addition of  $ZrO_2$ . The  $ZrO_2$  particles would have strengthened both the Nb<sub>3</sub>Sn and the niobium core, but for the moment only the strengthening of the niobium will be con-



Figure 5 Ductile fracture of the niobium substrate in tape D.



Figure 6 Scanning electron micrograph showing the brittle characteristics of the failure of the substrate in tape  $ZrO_2$ .

sidered. It has been reported [21] that  $ZrO_2$  can increase the tensile strength of niobium by about a factor of three. Therefore, a reasonable estimate for the fracture stress of the Nb–ZrO<sub>2</sub> core would be in excess (allowing for some irradiation strengthening) of  $3 \times 465$  MN m<sup>-2</sup>, say 2000 MN m<sup>-2</sup>. Calculations show that even such a high strength core would be incapable of supporting the extra load when the Nb<sub>3</sub>Sn failed and so the ZrO<sub>2</sub>-bearing tapes, like those with niobium substrates, failed without any significant prior plastic deformation.

The two-stage failure of the copper-backed tape is thought to be the result of failure of the composite tape followed by failure of the very ductile copper coating at a decreasing stress due to overload.

The critical stress intensity factors  $(K_c)$  calculated from the tensile tests on tape with edge cracks are not the normally quoted plane strain fracture toughness  $(K_{IC})$ . For a test to yield a valid  $K_{IC}$  value the plastic zone at the crack tip must be small with respect to the dimensions of

the specimen which leads to the acceptance criterion that the crack length and the smallest dimension of the specimen must be greater than 2.5  $(K_{\rm IC}/\sigma_{\rm y})^2$ , where  $\sigma_{\rm y}$  is the yield stress. Substituting the experimentally obtained  $\sigma_{\rm f}$  for  $\sigma_{\rm y}$ and  $K_{\rm c}$  for  $K_{\rm IC}$ , it can be seen that this criterion was not satisfied in the present work.  $K_{\rm c}$  is a function of both the material and specimen geometry and is greater than  $K_{\rm IC}$  which is determined solely by the material. Thus the  $K_{\rm c}$  values obtained in the present work are a satisfactory measure of crack growth in thin composite tapes but are not applicable to bulk samples.

The effective surface energy of Nb<sub>3</sub>Sn has been estimated from tests on multifilamentary composites to be in the range 0.25 to  $0.83 \text{ Jm}^{-2}$  [7]. This gives an upper limit for  $K_c$  of Nb<sub>3</sub>Sn of about  $0.5 \text{ MNm}^{-3/2}$ . Even allowing for a considerable error in this estimate, there is a large difference between the estimated  $K_c$  for Nb<sub>3</sub>Sn and the measured  $K_c$  of 19.3 MNm<sup>-3/2</sup> for the unirradiated tape composite. The fracture toughness of niobium, even in the cold-worked condition, is without



Figure 7 Scanning electron micrograph showing the failure of copper-coated tape.



Figure 8 Fracture stress as a function of crack length for tapes with edge cracks.

question much higher than that for Nb<sub>3</sub>Sn and the relatively high  $K_c$  for the tape composite is attributed to the presence of the niobium core.

The lower  $K_c$  after irradiation is consistent with  $K_c$  being determined by both the Nb<sub>3</sub>Sn and the niobium core. Irradiation is known to embrittle niobium [20] and hence decrease the toughness. The effect of irradiation on Nb<sub>3</sub>Sn is unknown but is also likely to decrease  $K_c$ . However as  $K_c$  for Nb<sub>3</sub>Sn is so low the reduction in the toughness of the niobium core is probably mainly responsible for the observed fall in  $K_c$  of the tape composite on irradiation.

The presence of  $ZrO_2$  in the Nb<sub>3</sub>Sn and the niobium core had a beneficial effect on the strength and the toughness of the tapes. The  $ZrO_2$  could

have toughened the Nb<sub>3</sub>Sn indirectly through the reduction in grain size and/or by a number of "direct" mechanisms such as crack blunting, crack deviation, crack front elongation and crack interaction with pre-existing microcracks. Of these "direct" mechanisms the latter may be ruled out as (i) it normally leads to a reduction in strength and (ii) the linear expansion coefficients of Nb<sub>3</sub>Sn and  $ZrO_2$  are similar (~ 7 × 10<sup>-6</sup> ° C<sup>-1</sup>) and, therefore, any thermal stresses would not be sufficient to form microcracks at the ZrO<sub>2</sub> particles. Without more detailed information, for example Nb<sub>3</sub>Sn-ZrO<sub>2</sub> interfacial energy, it is impossible at this stage to comment on the relative roles of the other mechanisms. As far as the  $Nb-ZrO_2$  is concerned, scanning electron microscopy has shown



Figure 9 Comparison of the fracture stresses of niobium substrate and composite tapes with edge cracks of varying lengths.

the fracture mode to be cleavage, which is a consequence of the particles increasing the stress necessary for the plastic flow associated with ductile failure. It is a misconception that the toughness of a material which exhibits a cleavage failure is necessarily less than that of a material which fails by a ductile microvoid coalescence mode, although this is often the case. Hence, it is not possible to state whether the  $ZrO_2$  particles decrease or increase  $K_c$  of niobium and therefore the only conclusion that may be drawn is that the improved  $K_c$  of the  $ZrO_2$ -bearing tape is in part, and perhaps even mainly, due to the increase in  $K_c$  of the Nb<sub>3</sub>Sn.

#### 5. Conclusions

1. Commercially produced Nb<sub>3</sub>Sn tapes irradiated to a fast neutron dose of  $5.4 \times 10^{23}$  n m<sup>-2</sup> show an increase independent of dose in both the shear and Young's modulus of the tapes in the range  $3 \times 10^{21}$  to  $4 \times 10^{23}$  n m<sup>-2</sup>. A much higher increase is observed at higher doses. The increases in the moduli result from increases in the moduli of Nb<sub>3</sub>Sn.

2. Tensile tests have shown that, with the exception of the copper-backed tape  $(CZrO_2)$ , all the tape composites fail without any prior plastic deformation. No stable microcracks are observed in the Nb<sub>3</sub>Sn before final failure, although cracks are present after failure up to a distance of  $40 \,\mu\text{m}$  from the point of fracture. The failure of the composite occurs when the Nb<sub>3</sub>Sn fractures; the failure of the Nb<sub>3</sub>Sn layer leads to a transfer of the load to the core which then fails in overload. Examination of the fracture surfaces shows that Nb<sub>3</sub>Sn exhibits intergranular failure and that the core fails in a ductile mode in unirradiated tapes, but in a more brittle manner after irradiation and/or if ZrO<sub>2</sub> particles are present.

3. The effect of the excess tin and the copper backing on the fracture stress is negligible. The fracture stress is reduced by irradiation to doses of up to  $1.0 \times 10^{23}$  nm<sup>-2</sup>, but is increased at higher doses in the niobium substrate tape. The fracture stress is greatly increased by the presence of ZrO<sub>2</sub> particles.

4. Tests on Nb<sub>3</sub>Sn tapes containing edge cracks show that: (a) the fracture stress decreases with increasing crack length, (b)  $K_{\rm e}$ , the critical stress intensity factor, is reduced on irradiation due to a reduction in the  $K_{\rm e}$  of the niobium core and (c)  $K_{\rm e}$  is greater for the ZrO<sub>2</sub>-bearing tapes, probably as the result of an increase in the  $K_c$  for Nb<sub>3</sub>Sn.

5. The mechanical properties data presented in this paper will be essential at the design stage of a critical component such as a plasma containing magnet. In particular, the loss of strength and toughness due to irradiation damage would have to be taken into account for this application. The most encouraging result was the considerable benefit arising from the addition of  $ZrO_2$  to the substrate. For a number of years,  $ZrO_2$  has been added to some Nb<sub>3</sub>Sn superconductors in order to improve the superconducting properties by limiting the grain size but it was not known that there was concomitant major mechanical advantage.

#### Acknowledgements

The financial support of the Science and Engineering Research Council and the provision of research facilities by Professor D. W. Pashley are gratefully acknowledged. The authors are also indebted to Drs J. A. Lee and P. E. Madsen of AERE, Harwell, for the provision of materials and useful discussion.

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Received 5 October and accepted 3 November 1983