

## **Tensile properties of the individual phases in un-reacted multifilament Nb<sub>3</sub>Sn wires**

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The room temperature elastic and plastic properties under uniaxial tensile loading of the different phases of an un-reacted, internal-tin process, Nb<sub>3</sub>Sn wire have been determined by tensile tests of whole wires and of extracted Ta, Nb and Nb alloy filaments, as well as by indentation hardness measurements in metallographic wire cross sections.

### INTRODUCTION

As part of the EU-supported Next European Dipole (NED) Joint Research Activity [1] an effort has been launched in European industry to promote the development of high performance superconducting multifilament composite Nb<sub>3</sub>Sn wires and cables. To simulate cabling degradation and optimise wire design, a Finite Element (FE) model of the wire is under development [1]. FE simulations require the input of relevant materials properties. In this article, we report room temperature tensile strength and E-modulus results for the different phases in an Internal Tin (IT) Nb<sub>3</sub>Sn wire and describe how these materials properties have been measured.

### EXPERIMENTAL PROCEDURES AND RESULTS

#### The samples

The mechanical properties of the different phases in an un-reacted IT Nb<sub>3</sub>Sn wire produced by Alstom/MSA for CEA and described in detail in reference 2, have been determined. The wire with a nominal diameter of 0.825 mm consists of a Cu stabiliser, 19 Sn sub-elements made up of 198 Nb-7.5wt.% Ta filaments (nominal filament diameter: 4 µm) embedded in a Cu matrix and arranged in concentric circles around 19 pure Sn pools, and a mixed Nb and Ta diffusion barrier, surrounding the multifilament area.

The E-modulus of Ta has been measured on single filaments extracted from Ta/Cu monofilament samples produced at LNCMP for the development of resistive pulsed magnets. These monofilament wires are precursors for nanofilamentary wires, whose microstructure and mechanical properties have been extensively studied [3,4]. The Nb and Nb-7.5wt.%Ta E-moduli have been measured on filaments extracted from Nb-Ta/Cu and Nb/Cu monofilament samples provided by Alstom/MSA.

### Tensile tests

Tensile tests of Nb<sub>3</sub>Sn wires and binary composite monofilament samples were performed according to DIN EN 61788-6(2001-07) [5]. For the determination of the wire proof stress ( $R_{p0.2}$ ), the raw-data stress-strain curve is fitted by a 2<sup>nd</sup> order polynomial and displaced so as to intercept the zero stress-strain position, as described in [6]. The tensile test results are summarised in Table 1 for the whole Nb<sub>3</sub>Sn composite wire and for samples of the same wire but where the Cu stabiliser has been removed by chemical etching.  $R_m$  of the wire without stabiliser shows an unusually large variation, which is possibly due to the roughness of the sample surface (Nb barrier).

Table 1  $E_A$ ,  $R_{p0.2}$  and  $R_m$ , Nb<sub>3</sub>Sn composite wire, with and without stabiliser (an average of 5 tensile tests).

Sample	$E_A$ (GPa)	$R_{p0.2}$ (MPa)	$R_m$ (MPa)
Whole wire	108±0.9	374±14.0	523±3.6
Wire without stabiliser	93.3±0.8	516±2.8	612±99

The E-moduli obtained for the filaments extracted from the binary composite monofilament samples are:  $E_A(\text{Nb-Ta})=94.1±0.14$  GPa,  $E_A(\text{Nb})=91.7±0.99$  GPa and  $E_A(\text{Ta})=170±0.5$  GPa (an average of three tensile tests).

Tensile tests of single Nb-Ta filaments extracted from the IT Nb<sub>3</sub>Sn wire were performed with a dedicated apparatus built by BAM. The filament cross section is measured with an optical system. Since an extensometer can not be attached to the filaments, the filament E-modulus and  $R_{p0.2}$  can not be measured. The measured filament ultimate tensile strength is  $R_m=977±303$  MPa (an average of ten measurements). The tensile test result obtained for a Nb-7.5wt.%Ta monofilament extracted from the precursor binary composite is  $R_m=734±0.4$  MPa (an average of three measurements).

## DISCUSSION

### Elastic anisotropy in the cold-drawn wire phases

Since the E-modulus is mainly determined by the binding forces between atoms, it is much less influenced by cold work and heat treatments than the tensile strength. Nevertheless, as a consequence of the asymmetric materials flow during wire processing, an important elastic anisotropy can be observed [7]. For the Cu matrix in Nb-Ti/Cu multifilament wire an E-modulus of 116 GPa [8] has been measured, which is about 10% lower than the E-modulus value of 128 GPa reported for isotropic Cu and similar to the Cu E-modulus in the Nb<sub>3</sub>Sn wire [9].

The E-modulus measured for the Nb filament extracted from the monofilament Nb/Cu sample is 92 GPa, 11% lower than the value for isotropic Nb (103 GPa [10]) and the Ta filament E-modulus is 170 GPa, 8% lower than the value reported for isotropic Ta (185 GPa [11]). These results indicate a strong elastic anisotropy in the heavily cold worked phases and it can be expected that the E-moduli perpendicular to the wire drawing direction are higher than those measured in the drawing direction.

A Sn E-modulus of 49 GPa has been derived from instrumented indentation measurements on a wire cross section. The E-modulus reported for self-annealed, fine

grain Sn is 44.3 GPa [10]. Due to the very low annealing temperature of Sn it is assumed that the Sn in the Nb<sub>3</sub>Sn wire is elastically isotropic.

#### Composite wire E-modulus calculated according to the ROM

Assuming isostrain conditions, the wire stress is the volume average of the local stresses and the composite modulus follows a Rule Of Mixtures (ROM) [12]. In Table 2, the ROM result is compared with the experimentally determined composite wire E-moduli. For the whole wire and the wire without stabiliser, the ROM result exceeds the experimentally determined composite E-modulus by 2% and 9%, respectively.

Table 2 E-modulus calculation of the Nb<sub>3</sub>Sn wire without and with stabiliser according to the ROM.

Material	E (GPa)	Wire without stabiliser		Whole wire	
		Vol.%	weighted E (GPa)	Vol.%	weighted E (GPa)
Cu	116 [Error! Bookmark not defined.]	51.1	59	80.2	93
Nb-Ta	94	21.9	21	8.9	8.4
Sn	49	13.9	6.8	5.6	2.7
Nb	92	8.7	8.0	3.5	3.2
Ta	170	4.4	7.5	1.8	3.1
<b>ROM</b>			<b>102</b>		<b>110</b>
<b>Experiment</b>			<b>93.3</b>		<b>108</b>

#### Plastic properties

For fully work-hardened and isotropic materials the plastic materials properties can be estimated from the indentation hardness according to Equation 1, or when elastic deformation can be neglected, the yield stress (YS) is about one third of the Vickers hardness (HV) expressed in identical units [13,14,15].

$$HV/YS=0.065+0.6\ln(E/YS) \quad 1$$

Table 3 summarises the HV values that have been measured for the different phases in the Nb<sub>3</sub>Sn wire cross section, together with the YS calculated as YS=HV/3 or using Equation 1 (for E/HV<44.3). The experimental details of the indentation hardness measurements are described in references 8 and 16. For the textured wire phases it is assumed that  $R_{p0.2}$  exceeds the YS values derived from the HV/YS ratio, which is valid for isotropic materials, by about 10% [8].

Table 3 HV, E-modulus and YS derived from HV for Nb, Ta, Cu and Nb-Ta in the un-reacted wire. It is estimated that due the wire texture  $R_{p0.2}$  exceeds YS by 10%.

Material	HV (MPa)	E (GPa)	E/HV	HV/YS	YS (MPa)	$R_{p0.2}$ (MPa)
Nb	2420	92	38	2.9	830	910
Ta	3340	170	51	3	1110	1220

Cu	1050	115	110	3	350	385
Nb-7.5wt.%Ta	2110	94	45	3	700	770

The YS derived for the heavily cold worked Nb-7.5wt.%Ta alloy is about 16% lower than the YS of cold worked Nb. The reported tensile strength of annealed Nb-10wt.%Ta is about 10% lower than the strength of unalloyed, annealed Nb [17]. It has been found from tensile tests of monofilament samples with similar cold work as the phases in the Nb<sub>3</sub>Sn wire that the Nb, Ta and Nb-Ta elongation at fracture is smaller than 2%, and that  $R_m$  is about 100 MPa higher than  $R_{p0.2}$ . For cold-worked Cu wire,  $\epsilon=1.5\%$  and  $R_m-R_{p0.2}\approx 35$  MPa [Error! Bookmark not defined.].

## CONCLUSION

Table 4 Room temperature tensile properties under uniaxial tensile loading for the strongly cold-worked materials in the IT Nb<sub>3</sub>Sn wire.

	$E$ (GPa)	$R_{p0.2}$ (MPa)	$R_m$ (MPa)	$\epsilon$ (%)
Nb	92	910	1010	<2
Ta	170	1220	1320	<2
Nb-Ta	94	770	870	<2
Cu	116	385	420	1.5

The measured E-moduli and estimated strength for the different phases in an un-reacted IT Nb<sub>3</sub>Sn wire under uniaxial tensile loading at room temperature are summarised in Table 4. The estimated accuracy of the E-moduli and tensile strength is better than  $\pm 3\%$  and  $\pm 10\%$ , respectively. The determination of the mechanical properties perpendicular to the wire drawing direction and under compression is subject for further studies.

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## REFERENCES

- 1 A. Devred et al., *Supercond. Sci. Technol.*, (2006), **19** 67-83
- 2 M. Durante et al., *Physica C*, (2001), **354** 449-453
- 3 L. Thilly, F. Lecouturier, J. von Stebut, *Acta Materialia*, (2002), **50** 5049-5065
- 4 V. Vidal, L. Thilly, F. Lecouturier, P.-O. Renault, *Acta Materialia*, (2006), **54** 1063-1075
- 5 International standard, DIN EN 61788-6(2001-07), (2001)
- 6 K. Katagiri et al., *Physica C*, (2001), **357-360** 1302-1305
- 7 S.R. Agnew, J.R. Weertman, *Materials Science and Engineering*, (1998), **A242** 174-180
- 8 C. Scheuerlein, T. Boutboul, D. Leroy, L. Oberli, B. Rehmer, “Hardness and tensile strength of multifilamentary metal-matrix composite superconductors for the Large Hadron Collider”, Accepted for publication in *J. Mat. Sci.*
- 9 C. Scheuerlein, B. Rehmer, “CERN, AT-MAS technical note 2005-08, (2005)
- 10 ASM Handbook Volume 2, “*Properties and Selection: Nonferrous Alloys and Special-Purpose Materials*”, (1990)
- 11 M. Merkel, K.-H. Thomas, “*Taschenbuch der Werkstoffe*”, Fachbuchverlag Leipzig, 5th edition, (2000)
- 12 M.F. Ashby, Y. Bréchet, *Acta Materialia*, (2003), **51** 5801-5821

- 13 D.M. Marsh, Proc. Roy. Soc. A, (1963), 279 420-435
- 14 E.W. Collings, "*Applied Superconductivity, Metallurgy, and Physics of Titanium Alloys*", Vol.1, Plenum Press, New York, (1986)
- 15 D. Tabor, "*The hardness of solids*", Review of Physics in Technology 1, (1970), 145-179
- 16 C. Scheuerlein et al., "Vickers hardness of the individual phases in multifilament Nb<sub>3</sub>Sn superconducting wires", for submission to Materials Characterization
- 17 N. Chamdawalla et al., Metall. (1986), 40(2) 141-145