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Scheuerlein, C. (CERN) et al

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Comparison of electromechanical properties and lattice distortions of different cuprate high temperature superconductors

C. Scheuerlein, R. Bjoerstad, A. Grether, M.O. Rikel, J. Hudspeth, M. Sugano, A. Ballarino, L. Bottura

Abstract—The electromechanical properties of different cuprate high temperature superconductors, notably two ReBCO tapes, a reinforced and a non-reinforced Bi-2223 tape and a Bi-2212 wire have been studied. The axial tensile stress and strain, as well as the transverse compressive stress limits at which an irreversible critical current degradation occurs are compared. The experimental set-up has been integrated in a high energy synchrotron beamline and the self-field critical current and lattice parameter changes as a function of tensile stress and strain of a reinforced Bi-2223 tape have been measured simultaneously. Initially the Bi-2223 filaments exhibit nearly linear elastic behavior up to the strain at which an irreversible degradation is observed. At 77 K an axial Bi-2223 filament pre-compression of 0.09% in the composite tape and a Bi-2223 Poisson ratio $\nu = 0.21$ have been determined.

Index Terms—High temperature superconductor, critical current, XRD, lattice distortion, stress, strain

I. INTRODUCTION

H^{IGH} TEMPERATURE superconductors (HTS) can provide high critical current (I_c) densities at high magnetic fields that are needed for general purpose research magnets and NMR magnets that can reach fields beyond those achievable using Nb₃Sn technology [1,2,3]. HTS are also required for accelerator magnets that can produce magnetic fields above 16 T [4,5]. In these magnets the superconductors are subjected to large Lorentz forces and the understanding of the electromechanical properties of superconducting composite tapes and wires is essential. In solenoid magnets the predominant conductor load case is tensile axial stress, while in accelerator magnets transverse compressive stresses are prevailing.

In this article we compare the mechanical and the electromechanical properties of different industrially produced HTS-based conductors, notably two coated conductor (CC) tapes, a reinforced and a non-reinforced Bi-2223 tape and a Bi-2212 wire, under uniaxial tensile and under transverse compressive loading at 77 K in self-field.

We have characterized straight tapes and wires that are free to contract during cooling down. This test configuration also enables simultaneous X-ray diffraction (XRD) measurements in transmission geometry in a high energy synchrotron beamline for studying the lattice distortions in the composite wire [6,7]. Here we present the results of a simultaneous XRD, I_c , stress-strain study of a Bi-2223 tape, and compare the results with those reported previously for a Bi-2212 wire [8].

II. EXPERIMENTAL

A. The samples

Metallographic cross sections of the two coated conductor monofilament tapes produced by SuperPower and American Superconductor (AMSC), the two multifilamentary Bi-2223 tapes produced by Sumitomo Electric Industries (SEI) and the multifilamentary Bi-2212 round wire produced by Oxford Superconducting Technology (OST) are presented in Fig. 1.

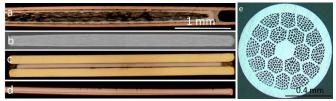


Fig. 1. Metallographic cross sections of (a) SEI Bi-2223 type HT-CA, (b) SEI Bi-2223 type G, (c) AMSC ReBCO tape, (d) SuperPower ReBCO and (e) OST Bi-2212 round wire 37x18.

The SuperPower tape SCS4050 consists of about 1 µm-thick ReBCO layer on a stack of biaxially textured buffer layers and a 50 µm-thick Hastelloy C-276 substrate [9]. The ReBCO coating and substrate are sandwiched between two ~2 µm-thick Ag layers and two 20 µm thick Cu stabiliser layers [10]. The AMSC type 8700 tape consists of a 0.8 µm thick ReBCO layer that is deposited onto an approximately 80 µm thick buffered Ni-5 at.% W substrate [11]. The tape is mechanically reinforced by two layers of brass [12]. The multifilament Bi-2223/Ag tapes produced by SEI are manufactured using the Oxide Powder-in-Tube (OPIT) method. The I_c and the mechanical properties are improved by controlled-overpressure (CT-OP) processing [13]. The type HT-CA tape is reinforced by

M. Sugano is with High Energy Accelerator Research Organization (KEK), 1-1 Oho, Tsukuba, Ibaraki 305-0801 Japan.

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C. Scheuerlein, R. Bjoerstad, A. Grether, A. Ballarino and L. Bottura are with CERN, CH-1211 Geneva 23, Switzerland, (corresponding author phone: ++41 (0)22 767 8022, e-mail: <u>Christian.Scheuerlein@cern.ch</u>).

M.O. Rikel was with Nexans SuperConductors GmbH, Germany; present address: Deutsche Nanoschicht GmbH, Heisenbergstrasse 16, Rheinbach, Germany

J. Hudspeth is with European Synchrotron, 6 rue Jules Horowitz, F-38043 Grenoble, France.

soldering the silver matrix in between two layers of copper alloy (thickness of $2 \times 50 \ \mu\text{m}$) [14]. The silver matrix of the nonreinforced type G tape is alloyed with gold (Ag-Au5.4wt.%), in order to reduce thermal conductivity for current lead applications. The multifilamentary Bi-2212/Ag round OPIT wire has been produced by OST. The outer wire sheath is made of a Ag-0.2wt.%Mg alloy. After OP processing the wire has been submitted to an optimisation heat treatment for maximizing I_c at 77 K. For more information about the Bi-2212 wire see reference [8]. The samples have largely different cross sections and aspect ratios (see Table I).

TABLE I HTS CHARACTERISTICS

Superconductor	Cross section (mm ²)
ReBCO tape SuperPower SCS 4050	0.410±0.020
ReBCO tape AMSC 8700	1.95±0.016
B-2223 tape (multifilament) SEI HT-CA	1.64 ± 0.023
B-2223 tape (multifilament) SEI G	1.01 ± 0.005
Bi-2212 wire (multifilament) OST	0.477±0.002

B. Uniaxial tensile stress-strain measurements

Stress-strain measurements have been performed with a universal test machine (UTM) with a load capacity of 5 kN from Hegewald & Peschke MPT GmbH, and using an MTS-clip on extensometer type 632.27F-21 with a gauge length of 25 mm. For 77 K experiments the UTM has been equipped with a liquid nitrogen cryostat.

C. I_c vs uniaxial tensile strain measurements

For the self-field I_c measurements the samples were fixed with the grips of the UTM and immersed in liquid nitrogen at ambient pressure (see Fig. 2). I_c was measured with a voltage tap distance of 20 mm and an electrical field criterion of 1 μ V/cm. For electrical insulation from the grips G10 plastic pieces have been glued on the superconductor extremities.



Fig. 2. (a) UTM equipped for I_c vs stress and strain measurements in liquid nitrogen. (b) Bi-2212 wire mounted in the grips instrumented with voltage taps, clip on extensioneter and current leads.

For the strain dependent I_c measurements the UTM was operated in strain control. During cooling down the UTM was operated in force control to maintain a slight tensile pre-stress of about 5 MPa to keep the wire straight. In order to distinguish between irreversible and reversible I_c degradation during the experiment the load was periodically released and I_c was remeasured. The irreversible strain limit ($\varepsilon_{irr.5\%}$) is defined as the strain at which a permanent I_c degradation of 5% remains after unloading the sample. Correspondingly the irreversible stress limit ($\sigma_{irr.5\%}$) is defined as the stress at which a permanent I_c degradation of 5% remainent I_c degradation of 5% remainent I_c degradation of 5% remainent I_c degradation of 5% remained as the stress at which a permanent I_c degradation of 5% remained as the stress at which a permanent I_c degradation of 5% remained as the stress at which a permanent I_c degradation of 5% remained as the stress at which a permanent I_c degradation of 5% remained as the stress at which a permanent I_c degradation of 5% remained as the stress at which a permanent I_c degradation of 5% remained as the stress at which a permanent I_c degradation of 5% remained as the stress at which a permanent I_c degradation of 5% remained as the stress at which a permanent I_c degradation of 5% remained as the stress at which a permanent I_c degradation of 5% remained as the stress at which a permanent I_c degradation of 5% remained as the stress at which a permanent I_c degradation of 5% remained as the stress at which a permanent I_c degradation of 5% remained as the stress at which a permanent I_c degradation of 5% remained as the stress at which a permanent I_c degradation of 5% remained as the stress at which a permanent I_c degradation of 5% remained as the stress at which a permanent I_c degradation of 5% remained as the stress at which a permanent I_c degradation of 5% remained at the stress at which a permanent I_c degradation of 5% remained at the stress at which a permanent I_c degradation of 5% remained at the stress a

D. I_c vs transverse compressive stress measurements

Using the UTM in combination with a cryostat and a dedicated sample holder allows to perform I_c measurements of the HTS tapes as a function of transverse compressive stress, simulating the load case that is predominating in accelerator magnets.

The samples were mounted on a flat stainless steel plate that has been connected to the bottom of the reverse load frame of the UTM (see Fig. 3). We have used a 1 mm wide flat stainless steel pressing tool in order to achieve sufficiently high transverse pressures on the tapes, similarly as described in [15]. The maximum load that could be applied with the present setup is 1 kN. In combination with a 1 mm-wide pressing tool a maximum compressive stress of 250 MPa can be reached on a 4 mm-wide tape (assuming a homogenous stress distribution). The alignment of the compression tool is verified with Fuji Pressure Measurement Film MW 10-50 MPa.

The sample holder has been immersed in liquid nitrogen at ambient pressure and an electrical field criterion of 2 μ V/cm has been used to determine the relative I_c variations.



Fig. 3. (a) UTM with sample holder and pressing tool for I_c vs transverse compressive stress measurements. (b) Detailed view of the 1 mm-wide pressing tool.

E. In situ XRD measurements

High energy synchrotron XRD measurements in transmission geometry with an energy of 86.9 keV were performed at the ID15B beamline of the European Synchrotron. Before radial integration the two dimensional diffraction patterns were caked into 32 segments, in order to measure lattice parameters from the crystalline planes oriented both perpendicular and parallel to the applied load, which are in the following referred to as the axial and transverse directions, respectively. Diffraction peaks were fitted with Gaussian functions to follow the relative d-spacing changes as a function

of the wire strain measured with the extensioneter. For more information about the diffraction experiment see reference [8].

III. RESULTS

A. Mechanical properties

In Fig. 4 the 77 K stress-strain curves of the studied conductors are compared. In order to eliminate the influence of sample bending the initial parts of the stress-strain curves have been fitted, and the strain axis has been shifted such that the stress-strain curves intersect the (0, 0) point.

It can be seen that the CC tapes have highest strength because of the high strength of their substrate materials (Hastelloy C276 in the SuperPower and Ni-5 at.% W in the AMSC tape). Rupture of the non-reinforced and the reinforced Bi-2223 tapes occurred at a strain of roughly 0.2% and 0.4%, respectively. In most cases the Bi-2223 tapes ruptured close to the grips, and the results may be slightly influenced by stress concentrations due to the sample fixation. The OP processed Bi-2212 wire has the lowest ultimate strength, but ruptures at much higher strain than the Bi-2223 tapes.

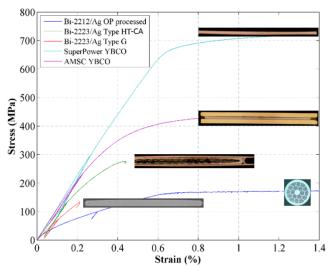


Fig. 4. Comparison of the 77 K stress-strain curves of the different HTS.

TABLE II ELASTIC MODULUS (E_a), 0.2% PROOF STRESS ($R_{p0,2}$) AND STRAIN ($\epsilon_{p0,2}$) OF THE DIFFERENT CONDUCTORS AT 77 K. *CORRESPONDS WITH STRESS AND STRAIN AT WHICH THE TAPES RUPTURED

AT WHICH THE TAFES RUFTURED.			
Sample	E _a (GPa)	$R_{p0.2}$ (MPa)	$\epsilon_{p0.2}(\%)$
ReBCO SuperPower	133±1.3	676±6.3	0.72 ± 0.02
ReBCO AMSC	109 ± 2.7	408±3.5	0.57 ± 0.01
Bi-2223 SEI HT-CA	104 ± 3.8	268±6.0*	0.41±0.02*
Bi-2223 SEI G	98.0±2.2	126±6.4*	$0.20\pm0.01*$
Bi-2212 OST 37×18	94.5±7.6	104 ± 5.3	0.31 ± 0.01

The 77 K mechanical properties are summarized in Table II. Elastic moduli (E_a) have been determined from the slopes of the linear part of the unloading stress-strain curves [16]. The unloading curves were fitted in the strain ranges 0.07-0.09% (Bi-2223 type G), 0.12-0.14% (type HT-CA), 0.11-0.21% (ReBCO AMSC), 0.15-0.25% (ReBCO SuperPower) and 0.29-0.30% (Bi-2212). All results are average values obtained from three stress-strain measurements.

B. Reversible and irreversible I_c degradation and n-value vs uniaxial tensile strain

The relative variation of the self-field I_c at 77 K as a function of uniaxial tensile strain is compared in Fig.5 (a). The Bi-2212 wire shows a relatively larger reversible degradation than the YBCO and Bi-2223 tapes. A comparison with Bi-2212 I_c vs strain data acquired at 4.2 K [17] suggests that the relatively large reversible degradation can be related to the fact that the Bi-2212 wire is at 77 K close to its critical temperature.

The AMSC tape has the highest irreversible strain tolerance of $\varepsilon_{irr-5\%}=1\%$. The CC tape of SuperPower and the OP processed Bi-2212 wire exhibit an irreversible strain limit $\varepsilon_{irr-5\%}$ of about 0.7% and 0.6%, respectively (dashed lines in Figure 5(a)). Rupture of the non-reinforced and reinforced Bi-2223 tapes occurred at a strain of about 0.2% and 0.4%, without measurable prior irreversible degradation.

The relative n-value changes are presented in Fig. 5(b). An irreversible degradation of 5% ($\epsilon_{irr-5\%}$) and an irreversible *n-value* reduction of 10% ($\epsilon_{irr-n-value-90\%}$) are obtained at close strain values.

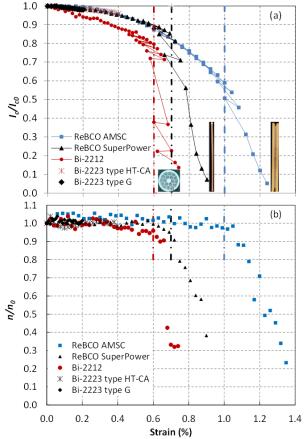


Fig 5. Comparison of (a) the I_c and (b) n-value variation of the different HTS as a function of uniaxial tensile strain.

C. I_c and n-value vs transverse compressive stress

Transverse compression experiments have been performed using the 1 mm-wide pressing tool with intermittent load release in order to distinguish between reversible and irreversible I_c degradation. The test mode (intermittent load release or monotonic loading) can influence the irreversible stress limit [18]. In some cases additional measurements were performed where the load was not released between the subsequent stress steps. The relative critical current and relative *n*-value evolution as a function of transverse compressive stress applied on the large side of the non-reinforced (type G) and reinforced (type HT-CA) Bi-2223 tapes are compared in Fig. 6 (a) and Fig. 6(b), respectively. It can be seen that the degradation is nearly completely irreversible. The irreversible damage of the superconductor is also revealed by the *n*-value reduction, which is closely correlated to the relative I_c changes.

After application of a transverse pressure of 100 MPa the critical current of the non-reinforced Bi-2223 type G tape is irreversibly reduced by about 50%. At the same stress the reinforced type HT-CA tape does not exhibit a significant I_c degradation. A 10% irreversible I_c reduction is obtained at a critical transverse stress of $\sigma_{\text{crit-trans-10\%}}\approx180$ MPa.

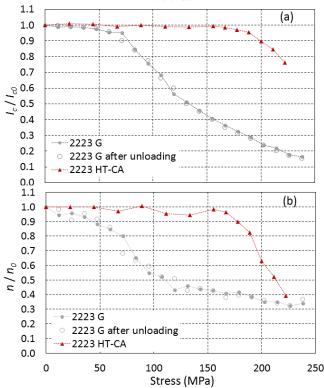


Fig. 6. Comparison of the relative (a) I_c and (b) n-value variation as a function of transverse compressive stress at 77 K in self-field. The relative variations after load release are shown by the empty symbols.

In the present study we did not observe a significant I_c degradation of the SuperPower tape with the 40 µm-thick Cu stabilizer up to $\sigma_{\text{trans}}=250$ MPa, which is the maximum stress that could be applied.

D. Bi-2223 lattice parameter, I_c and stress as a function of the extension extension that I_c and I

With the UTM integrated in the ID15 beamline of ESRF the lattice parameter changes, I_c , stress and strain of the Bi-2223 HT-CA tape were measured as described in [8]. A critical current of I_c =193 A is measured before application of an external load, which is in good agreement with the I_c =194 A value that is reported in [19]. In Fig. 7 the axial and transverse Bi-2223 (200) d-spacing changes and the simultaneously measured I_c and stress at 77 K are plotted as a function of the

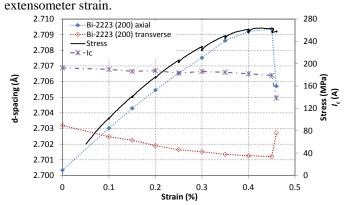


Fig.7. Axial and transverse Bi-2223 (200) d-spacing, I_c and stress of the SEI HT-CA tape as a function of the extensioneter strain at 77 K.

It can be seen that up to an extensioneter strain of about 0.35% the Bi-2223 filaments exhibit nearly linear elastic behavior. Before application of an external strain the Bi-2223 lattice parameter in transverse direction is larger than it is in axial direction, indicating that the Bi-2223 filaments are in axial pre-compression inside the matrix.

E. Bi-2223 filament axial pre-compression

Contrary to Bi-2212 in wires [20], Bi-2223 does not have inplane texture, so that 200 and 020 diffraction rings fully overlap, and the 2(b - a)/(b + a) ~ 0.3% orthorhombicity of Bi-2223 [21] is not an obstacle for an accurate calculation of the *d*-spacing values in the transverse and longitudinal directions.

Assuming that the nearly stress free state is obtained when the axial and transverse d-spacing values are equal, an axial Bi-2223 pre-compression of 0.09% inside the composite tape can be estimated at 77 K. The axial pre-compression of the Bi-2223 filaments is caused by the mismatch of thermal expansion coefficients of the different composite constituents. In another brass laminated tape a Bi-2223 filament pre-strain value of 0.11% has been determined by neutron diffraction at room temperature [22].

F. Bi-2223 filament ultimate tensile strain limit

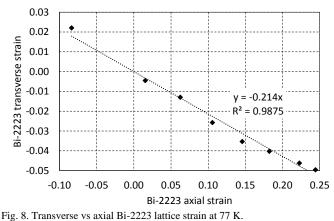
With increasing tensile strain I_c decreases initially slightly and reversibly. Unlike in A15 superconductors, no I_c maximum was observed when the external strain is similar to the axial precompression of the superconducting filaments.

Above about 0.4% extensioneter strain the composite tape stress and the elastic Bi-2223 filament strain do not increase further, indicating that the Bi-2223 filaments have reached the maximum stress that they can carry. At 0.46% a drastic reduction of I_c occurs and the n-value drops from 15 to 7. A Bi-2223 filament stress relaxation at 0.46% strain can be seen by the decrease of the axial Bi-2223 (200) d-spacing, which is a clear indication that at this strain many of the filaments fractured. Rupture of the entire tape occurred at slightly higher strain.

G. Bi-2223 Poisson ratio

In Fig. 8 the Bi-2223 elastic strain in transverse direction is plotted as a function of the Bi-2223 axial strain. The strain has

been calculated from the relative Bi-2223 (200) d-spacing changes, assuming that the stress free Bi-2223(200) d-spacing is 2.7026 Å. From this plot the Bi-2223 Poisson ratio at 77 K is determined as $\nu = 0.21$.



IV. DISCUSSION AND CONCLUSION

Using an UTM in combination with a 77 K calibrated extensioneter and an I_c measurement set-up allows to study the irreversible I_c degradation of HTS under tensile axial and transverse compressive loading. The axial tensile test configuration with a sample that is free to contract during cooling down allows to measure the intrinsic irreversible uniaxial tensile strain limit of the straight HTS tapes and wire, without an influence of sample holder substrate and solder. Such measurements can complement other commonly used experiments to characterize the electromechanical properties of superconductors using for instance Walters springs [23] or bending springs [24].

The weight and dimensions of the chosen UTM allow its integration into a high energy synchrotron beamline and enable the simultaneous measurement of I_c , n-value, lattice parameters as well as the composite wire stress and strain at 77 K in self-field. In this way a direct comparison of the lattice distortions and the superconducting properties is possible, which eliminates the influence of sample geometry, sample inhomogeneity, previous sample manipulation, thermal history, and of other experimental procedures and uncertainties that may hamper the comparison of lattice distortions and superconducting properties when these have been measured with different set-ups and samples [7].

In superconductors like Bi-2212, Bi-2223, Nb₃Sn [6] and Nb₃Al [25] several strong diffraction peaks can be acquired by *in situ* XRD measurements in transmission geometry, which can be fitted in order to determine accurate lattice parameter changes of the superconducting materials. The single-crystal like diffraction patterns of ReBCO in CC tapes require more sophisticated analysis. The acquisition of MgB₂ diffraction peaks in PIT wires is also more challenging [26,27].

Here we present for the first time an *in situ* synchrotron study with simultaneous lattice parameter, I_c , stress and strain measurements of the reinforced SEI Bi-2223 type HT-CA tape. The diffraction results show that initially the Bi-2223 filaments exhibit nearly linear elastic behavior, and the reversible I_c degradation increases continuously with increasing strain (Fig. 7). No I_c maximum is observed when the external strain compensates the axial pre-compression. Previously we have reported a similar linear elastic behavior of the Bi-2212 filaments in the OP processed Bi-2212 wire from OST [8], where I_c decreases steadily with increasing strain as well.

The I_c degradation behaviors of the different HTS that are described in this article appear to be fundamentally different under tensile axial and transverse compressive loading. Under axial tensile loading an important reversible I_c degradation of the cuprate superconductors is observed, where the initial I_c is recovered when the load is released. The reversible I_c degradation has been partly attributed to the strain dependence of their critical temperature [28,29]. In contrast, under transverse compressive loading the I_c degradation is immediately irreversible, and I_c does not recover when the load is released.

A strong n-value decrease is a good indicator to detect the irreversible degradation of the filaments and formation of cracks [30], which is also confirmed by the XRD lattice parameter measurements. In this study we find for both ReBCO tapes and for the multifilament Bi-2212 wire a reasonably good correlation between the axial strain at which a permanent I_c degradation of 5% and a permanent n-value reduction of 10% is obtained. Since the I_c degradation under transverse compressive loading is almost entirely irreversible, the relative I_c and n-value changes are closely correlated over the entire stress range.

The mechanical behavior and the critical stress at which a strong irreversible I_c degradation of the ReBCO, Bi-2223 and Bi-2212 conductors occurs is strongly influenced by the mechanical properties of the stabilizing and reinforcing materials [31]. The critical stress at which a 5% permanent I_c degradation occurs in the Bi-2212 wire with a pure Ag matrix and a Ag-0.2wt.%Mg alloy outer sheath is $\sigma_{irr-5\%} \approx 150$ MPa, which is about 4 times lower than $\sigma_{irr-5\%}$ of the SuperPower tape with a Hastelloy substrate. The reinforced Bi-2223 HT-CA tape ruptured at a stress of about 270 MPA, which is in good agreement with the critical stress of $\sigma_{crit}=250$ MPa that has been reported elsewhere [32]. The non-reinforced Bi-2223 tape has the lowest critical stress and ruptures already at $\sigma_{crit}=130$ MPa.

The irreversible axial tensile strain limit appears to be more influenced by the mechanical properties of the superconducting phase, with $\varepsilon_{irr-5\%}=0.6\%$ of the OP processed Bi-2212 wire being only slightly lower than the $\varepsilon_{irr-5\%}$ of about 0.7% that has been measured for the SuperPower ReBCO tape. Similar irreversible strain limits of SuperPower tapes have been reported elsewhere [33,34,35]. The irreversible axial tensile strain limit of the AMSC tape is with $\varepsilon_{irr-5\%}=1\%$ the highest of all conductors studied here.

In accelerator magnets the superconductor is mainly subjected to transverse compressive loads. For instance the transverse stress to which a ReBCO Roebel cable will be subjected in the EuCARD2 demonstrator magnet has been calculated to be 110 MPa at 13 T; for a 20 T magnet, calculations predict a transverse stress of 150 MPa [36]. The transverse stress values reported here have been calculated assuming a homogeneous stress distribution on the tape surface. Since most tapes studied are not perfectly flat, the true stress in the tape can be somewhat higher than the stress calculated from the force and the projected tape area. The influence of shear strain at the pressing tool edges and the influence of the pressing tool dimension and shape remain to be studied.

The reinforcement of the Bi-2223 type HT-CA tape laminate with two 50 μ m-thick Cu alloy sheaths is efficient to strongly increase the irreversible stress limit, which is an encouraging result for the potential use of Bi based HTS in accelerator magnets. It has also been shown that Bi-2223 tape reinforcement using oxide-dispersion-strengthened Ag can increase the transverse compressive stress tolerance to more than 100 MPa [37].

For the SuperPower tape with $2\times 20 \ \mu\text{m}$ Cu stabilizer thickness no significant I_c degradation has been observed up to 250 MPa, which is the maximum stress that could be applied with the present set-up. It has been reported that the transverse compressive stress limit of this tape decreases with increasing Cu layer thickness, presumably due to the so-called dog-bone effect that is assumed to cause an increasingly irregular stress distribution with increasing Cu thickness. For the SCS4050 with a 40 µm-thick Cu stabilizer the transverse stress limit reported is about 650 MPa [15]. Similar values for single SCS4050 tape are reported in [38].

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