

## Journal Publication

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# Strain induced irreversible critical current degradation in highly dense Bi-2212 round wire

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## Abstract

The strain induced critical current degradation of overpressure processed straight Bi-2212/Ag wires has been studied at 77 K in self-field. For the first time superconducting properties, lattice distortions, composite wire stress and strain have been measured simultaneously in a high energy synchrotron beamline. A permanent  $I_c$  degradation of 5% occurs when the wire strain exceeds 0.60%. At a wire strain of about 0.65% a drastic  $n$ -value and  $I_c$  reduction occur, and the composite stress and the Bi-2212 lattice parameter reach a plateau, indicating Bi-2212 filament fracturing. The XRD measurements show that Bi-2212 exhibits linear elastic behaviour up to the irreversible strain limit.

## 1 Introduction

High temperature superconductors (HTS) can be used for general purpose research magnets and NMR magnets that can reach fields beyond those achievable using Nb<sub>3</sub>Sn technology [i,ii]. HTS are also required for accelerator magnets that can produce magnetic fields above 16 T [iii] [iv]. In the frame of the EuCARD-2 Future Magnets project Bi-2212 round wires and YBCO tapes are tested as possible candidate materials for building 20 T accelerator magnets. These cuprate superconductors are strain sensitive, and their irreversible degradation under mechanical loading due to Lorentz forces is a concern for their application in future very high field magnets.

The critical current density ( $I_c$ ) of Bi-2212/Ag wires and its uniformity in long lengths can be strongly improved when the processing of the conductor is performed under a moderate process gas overpressure [v]. A pressure of 100 bar is sufficient to eliminate significantly the void space, which is present in the wires when the processing is performed at ambient pressure [vi]. In this article we present the results of a study of the electromechanical properties of an overpressure (OP) processed Bi-2212 straight wire.

Prior to the present experiment the OP processed Bi-2212 wire samples have been submitted to a special oxygenation heat treatment in order to optimise their critical current at 77 K. This has enabled the experiments in liquid nitrogen, strongly simplifying the experimental procedures. Using straight wires that can freely contract during cool down allows determining the intrinsic irreversible tensile strain limit, at which the superconductor exhibits a certain permanent degradation of its critical current, without an influence of sample holder materials properties.

Using an X-ray transparent cryostat this test configuration also enables X-ray diffraction (XRD) measurements in transmission geometry in a high energy synchrotron beamline for studying the lattice distortions in the composite wire [vii,viii], thus providing additional and complementary information about the Bi-2212 filament elastic strain.

## 2 Experimental

### The Bi-2212/Ag samples

The Bi-2212/Ag wire PMM130723-2 has been produced by Oxford Superconducting Technology (OST), using granulate precursor produced by Nexans SuperConductors GmbH. The 0.8 mm diameter powder-in-tube wire has  $37 \times 18$  Bi-2212 filaments, which are embedded in a pure Ag matrix. The Bi-2212 volume fraction stated by the manufacturer is 21.8%. After a densification heat treatment at 100 bar and 821 °C for 12 hours, a Bi-2212 volume fraction of 22.1% has been measured at the National High Magnetic Field Laboratory (NHMFL). The outer sheath of the wire is made of Ag-0.2wt.%Mg alloy, and the Mg oxide particles that are formed during the processing heat treatment [ix] lead to dispersion hardening of the outer sheath and a reinforcement of the wire.

Overpressure processing of the about 150 mm-long wires at 100 bar total pressure and 1 bar oxygen partial pressure was performed at the NHMFL using a standard heat treatment [x]. After overpressure processing a critical current of  $I_c=329$  A at 4.2 K, 5 T has been measured at NHMFL. After the overpressure processing, the wires were annealed for 20 h at 700 °C in 32% O<sub>2</sub>/balance N<sub>2</sub> and cooled down along the  $pO_2$ - $T$  trajectory  $\log pO_2[\text{bar}] = -8.24 \times 10^{-6} T^2 + 1.847 \times 10^{-2} T - 9.39$  ( $T$  in °C) until 300° C and then in N<sub>2</sub> flow (<2 ppm O<sub>2</sub>). According to the  $\delta$ - $pO_2$ - $T$  map of Bi<sub>2</sub>Sr<sub>2</sub>CaCu<sub>2</sub>O<sub>8+ $\delta$</sub>  [xi] this heat treatment adjusts the nominal oxygen index to  $\delta=0.213 \pm 0.002$ , which optimizes self-field  $I_c$  at 77 K in round wires made of precursor powder of Bi<sub>2.17</sub>Sr<sub>1.94</sub>Ca<sub>0.89</sub>Cu<sub>2.00</sub>O<sub>8+ $\delta$</sub>  composition [xii]. After such heat treatment, the  $I_c$  at 77 K in self field and at zero strain was  $10.3 \pm 0.2$  A. The additional thermal cycle might influence somewhat the pre-stress in the wire.

During all heat treatments the wires remained in ceramic Al<sub>2</sub>O<sub>3</sub> tubes in order to keep them straight, and to avoid wire damage. The diameter of the processed wires is 0.78 mm, which is slightly larger than the diameter expected for a completely dense wire.

### **Stress-strain measurements**

Stress-strain measurements were performed using a 5 kN universal testing machine (UTM) from Hegewald & Peschke MPT GmbH, which has been equipped with a liquid nitrogen cryostat. A KAP-S load cell with a maximum load of 1 kN with an accuracy of 0.1% has been used. For strain measurements an MTS-clip on extensometer 632.27F-21 with a gauge length of 25 mm was used. The extensometer has been calibrated at RT and in liquid nitrogen (RT and 77 K calibration factors are 0,999 and 1.026, respectively).

### **$I_c$ measurements**

All  $I_c$  measurements were performed with the samples immersed in liquid nitrogen at ambient pressure. For electrical insulation from the grips four rectangular pieces of G10 have been glued on the wire extremities. The voltage taps were soldered with a spacing of 20 mm, using Kester 135 flux and 50In-50Sn solder at 150 °C.

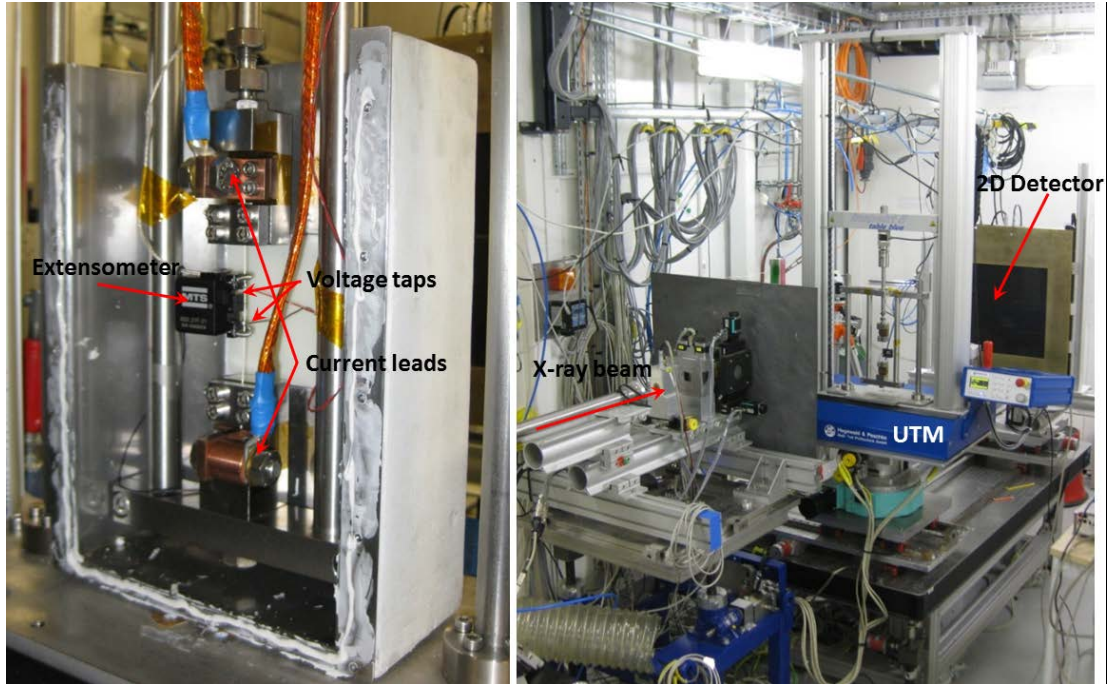
The critical current and  $n$ -values were determined using a power law and the least squares method with the electrical field criterion of 1  $\mu$ V/cm. An advantage of our set-up is that there is full control over sample stress and strain during the entire experiment. During cooling down the UTM was operated in force control to maintain a slight tensile pre-stress of about 5 MPa to keep the wire straight. For the strain dependent  $I_c$  measurements the UTM was operated in strain control. To keep the strain constant during the  $I_c$  measurements, the force did not vary more than 1 N.

Because of the relatively small voltage tap distance of 2 cm and the relatively low test current of 10 A used, our experiment is less sensitive to detect the onset of irreversible degradation than measurements performed for instance with Walters springs at lower temperature with higher test currents and larger voltage tap distances. Therefore, we have defined a relatively rough irreversible strain criterion as the strain at which a 5% permanent  $I_c$  degradation is obtained ( $\epsilon_{irr-5\%}$ ).

### **In situ XRD measurements**

High energy synchrotron XRD measurements in transmission geometry with an energy of 86.9 keV and a sample to detector distance of 1151.7 mm were performed at the ID15B beamline of the European Synchrotron. Diffraction patterns were acquired with a two dimensional fast read out detector Perkin Elmer XRD1621 CN3-ES.

A liquid Nitrogen cryostat with X-ray transparent Mylar windows has been built for the XRD experiments. Figure 1 (a) shows the instrumented Bi-2212 wire with the extensometer and current leads mounted in the tensile test set-up, with the front cover of the cryostat removed. In Figure 1(b) the UTM is mounted on the rotation and translation stages inside the ID15B beamline.



**Figure 1: (a) Superconducting wire instrumented for critical current measurements with voltage taps, current leads and extensometer mounted in the UTM. (b) UTM installed in the ID15 beamline of ESRF.**

Before radial integration the two dimensional diffraction patterns were dissected into 32 segments, in order to measure lattice parameters from the crystalline planes oriented both perpendicular and parallel to the applied load. In the following these are referred to as the axial and transverse directions respectively. Individual diffraction peaks were fitted with Gaussian functions in order to determine the relative peak position changes that are used to calculate the Bi-2212 elastic strain.

### 3 Results

#### Critical current as a function of wire strain

In Figure 2 the  $I_c$  and stress are presented as a function of uniaxial tensile Bi-2212 wire strain. In Figure 2 (a) the  $I_c$  return points after nearly complete unloading are shown. The load was always released such that a small tensile stress of 4 MPa remained, in order to keep the wire straight. A pre-load of 4 MPa causes an approximate wire strain in the order of 0.01 %, which has been neglected.

A permanent  $I_c$  degradation of 5% is found when the strain is  $\epsilon_{irr-5\%}=0.60\%$ . A value  $\epsilon_{irr-5\%}=0.62\%$  has been measured in a second experiment, when the load was only partly released after each  $I_c$  measurement. The corresponding wire stress at  $\epsilon_{irr-5\%}$  is  $\sigma_{irr-5\%}\approx 150$  MPa. The permanent (plastic) wire deformation as a function of the maximum applied stress recorded simultaneously with the  $I_c$  vs strain measurements is shown in Figure 2 (b). Due to the very low yield strain of the annealed pure Ag matrix a strong permanent wire deformation is already observed before strong Bi-2212 filament damage occurs [xiii]. At the strain  $\epsilon_{irr-5\%}=0.60\%$  the permanent wire deformation is about 0.34%.

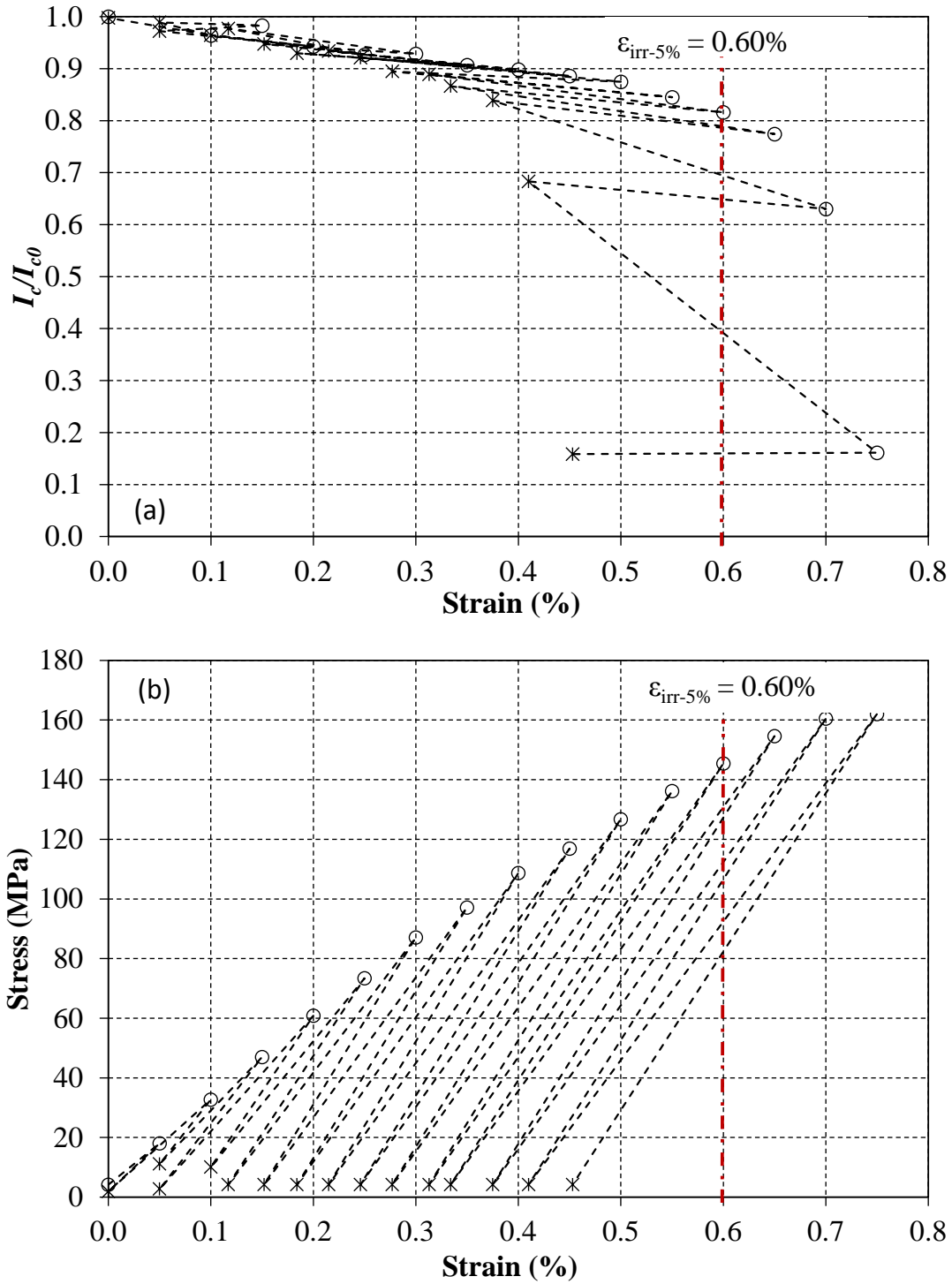


Figure 2: (a) Self-field critical current at 77 K as a function of the wire strain. (b) Permanent wire elongation after stress relaxation as a function of the maximum wire stress. Stress and strain have been recorded simultaneously with the  $I_c$  measurements shown in (a).

### ***n*-value and wire stress as a function of wire strain**

In Figure 3 the *n*-value and the wire stress as a function of wire strain are compared. Three simultaneous  $I_c$ , *n*-value and stress vs wire strain measurements have been performed at CERN, and in all cases a stress plateau is reached at about 0.65% strain. At the same strain where the stress plateau is reached, a drastic decrease in *n*-value and  $I_c$  is observed, presumably indicating that a strong damage of the Bi-2212 filaments occurs when the strain exceeds 0.65%. This suggests that stress-strain measurements may provide a good indication of the strain and stress limit at which strong Bi-2212 filament damage occurs. A similar stress plateau is observed in Nb<sub>3</sub>Sn powder-in-tube wires when the Nb<sub>3</sub>Sn filaments fracture [viii].

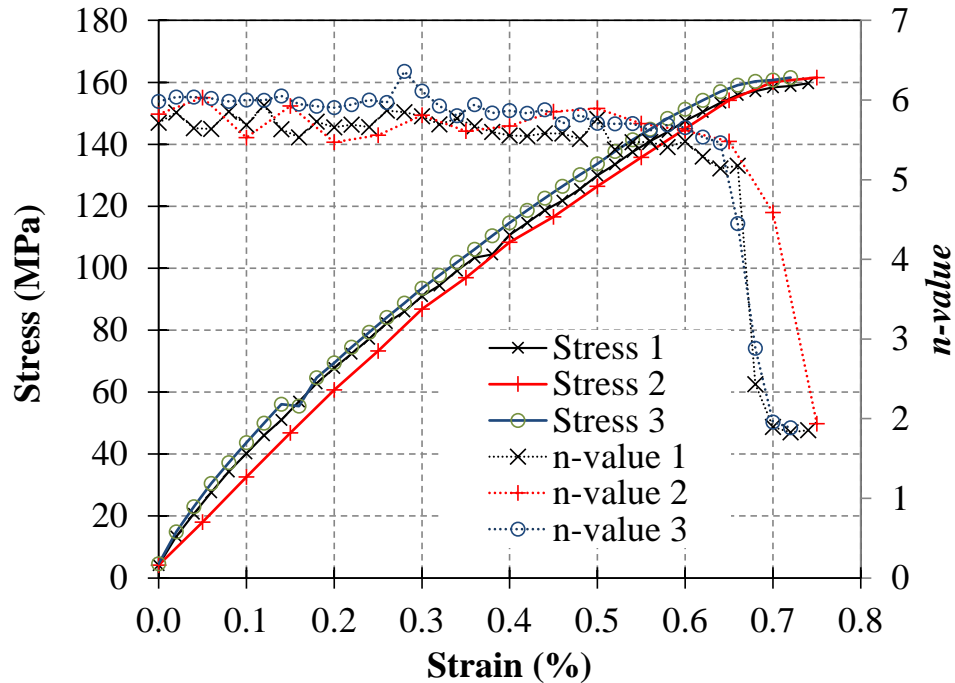
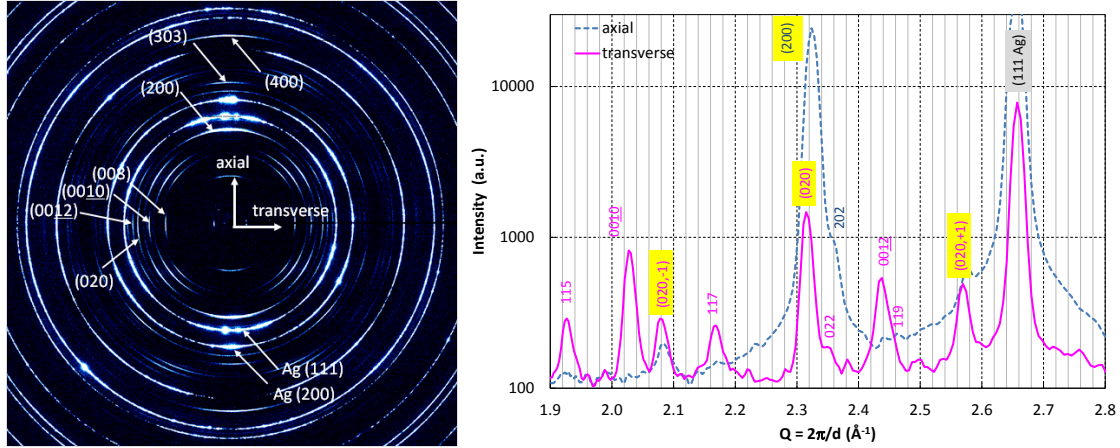


Figure 3: Comparison of Bi-2212 wire stress and *n*-value vs strain curves acquired at 77 K.

### **Bi-2212 lattice parameter changes as a function of wire strain**

The diffraction pattern of the Bi-2212 wire acquired at RT is shown in Figure 4(a). In agreement with previous results [xiv,xv], it shows significant texture effects. Strong arcs of (00*L*) reflections are observed only in the transverse direction indicating an out-of-plane texture with the *ab* planes of Bi-2212 grains preferentially aligned (FWHM =  $14 \pm 1^\circ$ ) parallel to the wire axis. Our data is also consistent with the presence of in-plane texture [xv]. This follows from the strongly non-uniform intensity distribution in the 200/020 ring with the intensity in the axial direction (200) about an order of magnitude stronger than in the transverse direction (020).



**Figure 4:** (a) Diffraction pattern acquired in transmission geometry at RT. (b) Radially integrated axial and transverse patterns, showing that the relative intensity of modulation reflections (020, $\pm$ 1) with respect to (200/020) is much higher in the transverse direction, which allows unambiguously index the transverse direction as [010] (modulation direction) and the axial direction as [100] in agreement with previous results [xv, xvi].

Figure 4(b) also shows that the 020 and 200 lines are significantly shifted with respect to each other. The Bi-2212  $d$ -spacing calculated from the position of 020 reflection in the transverse direction is about 0.3% larger than that calculated from the position of (200) reflection in the axial direction, which is at least a factor of three larger than the reported orthorhombicity of the Bi-2212 phase [xvii]. This indicates that the Bi-2212 filaments are in axial precompression inside the wire [xviii]. However, because of unknown orthorhombicity, assessing the exact precompression is not straightforward. Therefore, below all  $d$ -spacing values are normalised to those measured at the zero external strain.

The Bi-2212 (200), (303) and (400) reflections in the axial direction and the (020), (020,+1), (020,-1), (008), (0010) and (0012) reflections in the transverse direction were fitted to follow the relative  $d$ -spacing changes as a function of the wire strain measured with the extensometer at RT. The results are presented in Figure 5. Initially the axial  $d$ -spacing values increase and the transverse values decrease linearly with increasing wire strain. Above about 0.55% external strain the Bi-2212  $d$ -spacing changes only slightly, indicating that the filaments cannot carry higher loads anymore, presumably because of filament fracture. For comparison the simultaneously acquired stress-strain curve is shown as well. It can be seen that a stress plateau is reached at the same wire strain where the  $d$ -spacing plateau is observed.



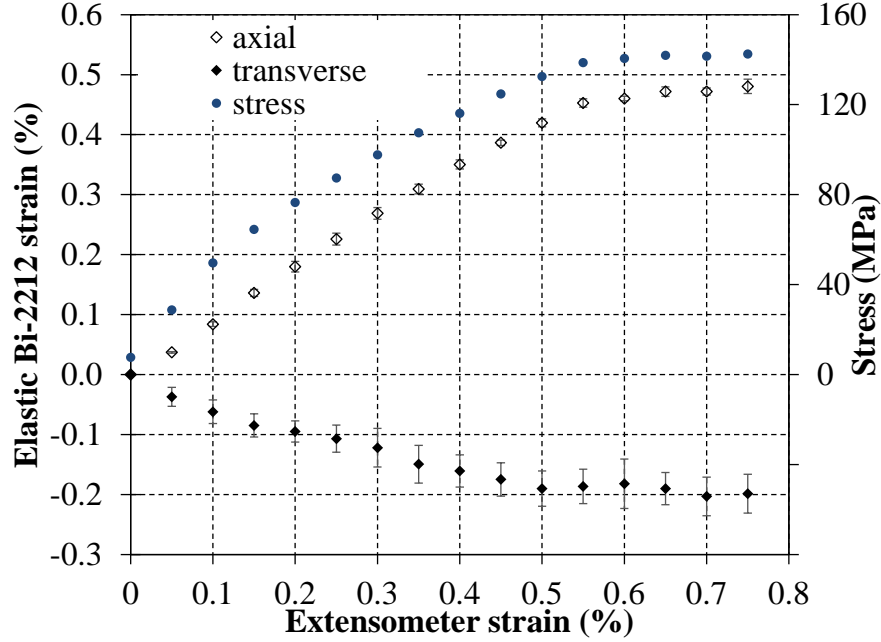
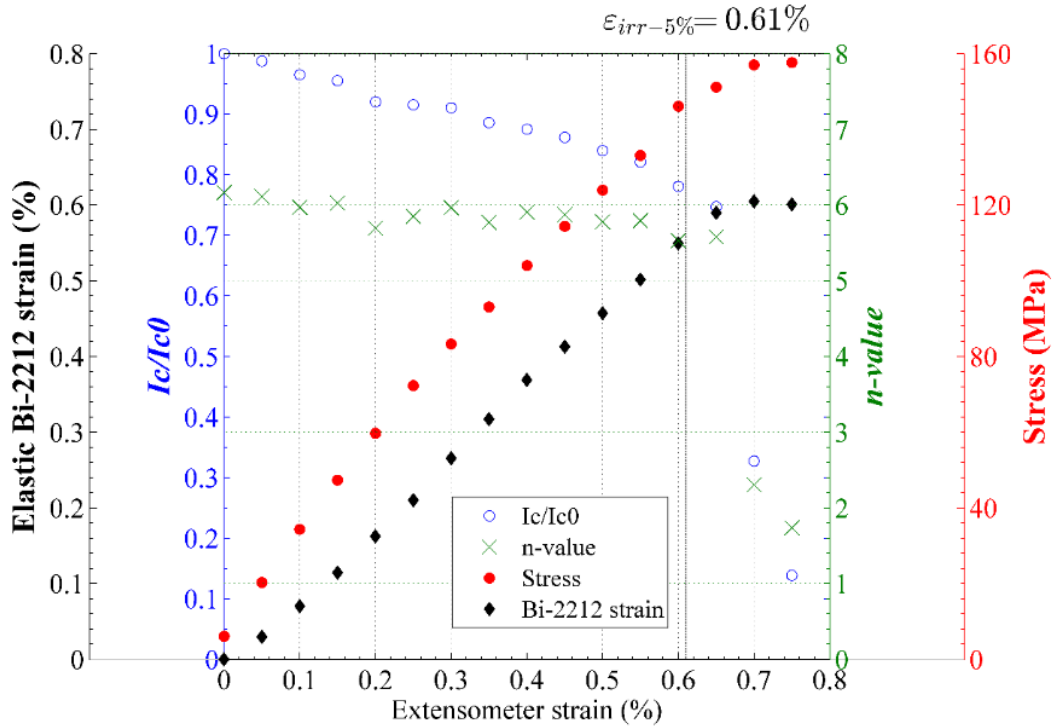


Figure 5: Elastic strain calculated from the relative Bi-2212 *d-spacing* changes with respect to the *d-spacing* at zero strain in axial and transverse direction as a function of the wire strain at RT. The strain values are the average for all reflections in a given direction, and the error bars show the scatter of the results ( $\pm 1 \sigma$ ). The stress-strain curve is shown for comparison.

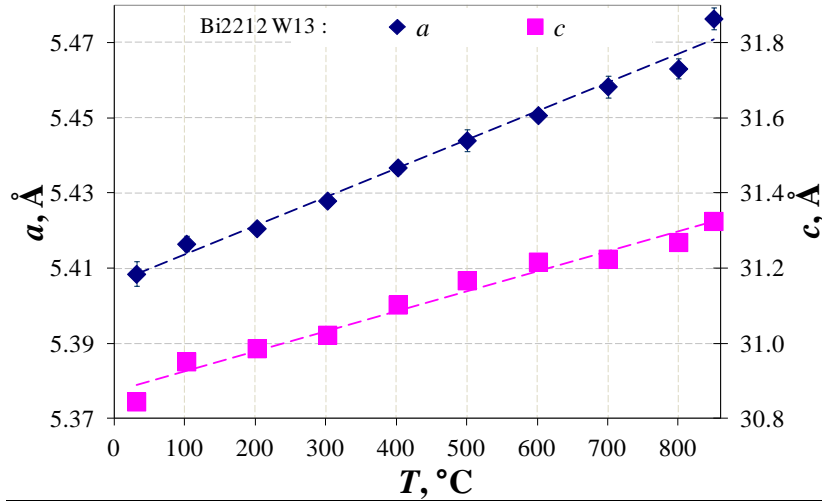
In Figure 6 the relative axial Bi-2212 lattice strain is compared with the simultaneously measured relative critical current  $I_c/I_{c0}$ , *n-value* and stress evolution as a function of the uniaxial wire strain that has been measured with the extensometer in liquid nitrogen. It can be seen that the Bi-2212–axial lattice parameter reaches a plateau at the same wire strain where the  $I_c$  and *n-value* drop drastically, and where the stress reaches a plateau as well. All these observations can be explained by filament fracturing at a wire strain of about 0.65% at 77 K.



**Figure 6: Comparison of Bi-2212/Ag wire relative  $I_c$ ,  $n$ -value, stress and axial Bi-2212 strain variations as a function of the composite wire strain all measured simultaneously at 77 K.**

### Thermal expansion of Bi-2212 and Ag

In previous work the microstructure and phase changes during Bi-2212 wire processing were studied using *in situ* XRD at ID15B [xix]. Using the XRD patterns from that study we assessed the lattice parameters of Bi-2212 and Ag in the green wire in the temperature range from RT to 850 °C. For Bi-2212, this is a rough estimate made assuming a tetragonal unit cell and using positions of the peak maxima of major reflections (002, 101, 008, 113, 115, 0010, 117, 200 and 220) without taking into account overlapping with lines of minor intensities. The Bi-2212  $a$  and  $c$  axis lattice parameter changes upon heating from RT to 850 °C are compared in Figure 7.



**Figure 7: Comparison of Bi-2212  $a$  and  $c$  axis lattice parameter changes upon heating of a multifilamentary Bi-2212 green wire to 850°C.**

The average linear coefficients of thermal expansion (CTE) determined for the whole temperature range of 30 to 850 °C are  $\alpha_{\langle a \rangle} = 1.4 \times 10^{-5} \text{ K}^{-1}$  and  $\alpha_c = 1.7 \times 10^{-5} \text{ K}^{-1}$ . These values are slightly lower than those reported for powder samples [xx], which may reflect the difference in the O exchange conditions in wire and powder. The CTE in the direction of the  $a$  axis ( $\alpha_{\langle a \rangle}$ ) is the most relevant in the case of a biaxially textured Bi-2212 wire. For Ag we have determined  $\alpha = 2.42 \times 10^{-5} \text{ K}^{-1}$ , which coincides relatively well with the literature data [xxi].

Extrapolating the data shown in Figure 7 to 4.2 K it can be estimated that during cool down from the processing peak temperature to the operation temperature Ag shrinks roughly 1% more than Bi-2212. The different thermal expansion coefficients can explain the Bi-2212 filament pre-compression in the composite wire. Apart from the thermal expansion coefficients, the filament pre-compression depends also on the elastic and plastic properties of the composite constituents.

## 4 Discussion and conclusion

Here we report to our knowledge for the first time the results of a simultaneous measurement of the superconductor critical current and  $n$ -value, the lattice parameter changes as well as the composite wire stress and strain.

The optimisation heat treatment of the overpressure processed Bi-2212 wires for maximizing  $I_c$  at 77 K has enabled the self-field  $I_c$  vs strain measurements in liquid nitrogen. In the test configuration using a straight wire that is free to contract during cooling down the stress state of the wire is not affected by the properties of a sample holder and solder materials. Wire bending and radial strain gradients are negligible.

Most studies of the Bi-2212 electromechanical properties have been performed at 4.2 K, the temperature at which Bi-2212 conductors are typically used. Since the reversible  $I_c$  degradation of cuprate superconductors is attributed to the stress dependence of their

critical temperature [xxii], it can be expected that at the comparatively high temperature of 77 K, which is very close to the Bi-2212 critical temperature, the initial reversible reduction of the critical current is much stronger than at 4.2 K. Indeed, as can be seen from Figure 6, the linear slope with which  $I_c$  is reduced up to a strain of 0.35% is  $dI_c/d\varepsilon = -28\%$  per percent strain, which is much steeper than the approximate slope of  $dI_c/d\varepsilon = -0.4$  that has been reported for another Bi-2212 wire at 4.2 K [xiii].

Temperature can also influence the strain at which filament fracture occurs, because the axial pre-compression of the filaments and the mechanical properties of the different wire constituents depend on temperature. This can be seen for instance by comparing the RT and 77 K stress-strain and XRD data presented here.

At 77 K the axial Bi-2212 *d-spacing* increases linearly with the extensometer wire strain, indicating a linear elastic behaviour up to a wire strain of about 0.6%. Increasing the wire strain further does not change the Bi-2212 axial lattice parameter, indicating that the Bi-2212 filaments cannot carry higher loads anymore. This is also seen by the change in slope of the stress-strain curve that reaches a plateau at a wire strain of about 0.65%. A drastic *n* value decrease is another indication for a strong filament damage [xxiii] when the wire strain exceeds about 0.65% (Figure 6).

The XRD and stress-strain results show that at RT the filament damage occurs at roughly 0.1% lower strain than at 77 K (Figure 5). However, we believe that the irreversible strain limit at which strong filament cracking occurs at 77 K is similar to that at 4.2 K, because in this temperature range the magnitude of the axial Bi-2212 pre-compression [xxiv] and the Young's modulus of the Bi-2212 wire change only slightly [xxv,xxvi]. Assuming that the mechanical strength of the Bi-2212 may slightly improve during cooling from 77 K to 4.2 K, we conclude that for the overpressure processed Bi-2212/Ag composite wire the irreversible uniaxial tensile wire strain exceeds  $\varepsilon_{irr-5\%} = 0.60\%$ .

The  $\varepsilon_{irr-5\%}$  value that we found in our study is about 0.1% higher than the  $\varepsilon_{irr-5\%}$  limit of 0.50% found in a previous study using another 1 bar melt processed 37x18 Bi-2212 wire soldered onto a Cu-Be Walters spring [xxvii]. In another study using a Cu-Be Walters spring the onset of irreversible degradation of different 1 bar melt processed Bi-2212/Ag wires has been detected at 0.31% [xiii]. This difference is partly due to the higher sensitivity of the latter experiment to detect the onset of irreversible degradation. Indeed, from the Figures in [xiii] it can be seen that the strain at which an  $I_c$  degradation of 5% occurs is higher than 0.45%.  $I_c$  vs strain results obtained with a U-shaped brass spring have been reported for different Bi-2212 wires with a pure Ag matrix and with reinforced matrix. A 5% permanent  $I_c$  degradation was in this case obtained at strain values between 0.4-0.7% [xxviii].

A comparison of the  $\varepsilon_{irr}$  results of a straight wire that is free to contract during cool down with  $I_c$  vs strain results obtained with other test devices is not obvious. The strain dependence of superconducting properties is commonly studied by soldering the superconductor onto the sample holder of Walters springs [xxix] or bending springs [xxx], which allows to perform experiments in tension and in compression. Soldering the superconducting composite onto a sample holder invariably influences the strain state after cool down, and the tensile strain limit at which an irreversible degradation is observed is influenced by the materials properties of the sample holder and the solder

[xxx]. The bending of the wire on springs also influences the wire internal stress state [xxx,xxxii,xxxiii]. Thus, the difference in  $\epsilon_{\text{irr-5\%}}$  of Bi-2212 wires that have been reported could be due to the influence of the test configuration.

Different Bi-2212 wire geometries, such as differences in filament size and the Bi-2212 volume fraction, and different processing could be another reason for the different  $\epsilon_{\text{irr}}$  values that have been reported. Since OP processing largely eliminates the porosity in the Bi-2212 wires [v], it could be expected that this process also improves the strain resistance. However, recently it was reported that OP processing does not strongly influence the irreversible strain limit under tensile loading [xxxiv]. The influence of porosity on the Bi-2212 wire resistance against transverse compressive loads, as they are dominating in accelerator magnets, is a subject of further studies.

The simultaneous measurement of the superconducting properties, mechanical properties and lattice distortions in straight superconducting composites that can freely contract during cool down is particularly useful to gauge the intrinsic conductor strain limits and to compare them directly to the lattice distortions. This type of experiment has great potential for studying the electromagnetic properties and degradation mechanisms of a variety of superconducting composites including  $\text{MgB}_2$  and  $\text{Nb}_3\text{Sn}$ , provided that the set-up can be equipped with a cryocooler for  $I_c$  measurements at variable temperature. Other test configurations are needed to perform measurements in compression, in applied field and with higher test currents.

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