Study of reliability of the HTS tapes in electrical circuit at 77 K

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Abstract. We investigated the stability of the critical parameters $I_c$ and $T_c$ of commercial HTS wires relative to the application of a transport current of 0.7 $I_c$ for extended periods of time in liquid nitrogen at 77K. The voltage-current characteristics $U(I)$ and critical current value of samples of Bi-2223-based tape (hermetic multi-filamentary in silver matrix and laminated with brass) and Dy-123 tape (on a Hastelloy C 276 steel substrate with MgO buffer layer) covered with a thin layer of Ag were examined. After 323 hours of applied current the critical current of the Bi-2223 tape decreased substantially and the sample expanded. A reference sample without applied current, but equally immersed in liquid nitrogen for 700 h showed a similar behavior. SEM investigations (JEOL JMS 5910-LV with LINK) revealed a change of the microstructure and the chemical composition of the Bi-2223 tape. The formation of microvoids inside the superconducting phase was discovered in the samples studied. The reason of the reduction of the critical current in Bi-2223 could be caused by the effect known as “bubbling”. No changes in the voltage-current characteristics $U(I)$ and the critical current value of the Dy-123 tape, manufactured with an epitaxial deposition technology, were found after applying a current for 400 hours and 1000 hours in liquid nitrogen.

1. Introduction
The progress in manufacturing of commercial HTS wires opens new horizons for applications like power cables, transformers, motors, and fault current limiters. However, the long-term reliability and the stability of HTS wires at working conditions need to be studied.
One of the effects that might worsen the superconducting properties of HTS wires is the well-known effect of “bubbling” [1]. This effect is connected with penetration liquid nitrogen inside of a superconductor, which evaporates at warming, that caused a strong deformation of
a material. An alternative effect is the electromigration of heavy ions and oxygen. The effect was discovered in thick ceramic films of Y-123, Bi-2212 and Bi-2223 compounds. Previously it was shown that irreversible local changes of a structure and phase constitution of HTS take place as a result of long-term continuous current transport at T=4.2-300 K [2-6]. More recently the electrical degradation of a HTS wire by continuous current transport was described [7].

The purpose of the work presented here was to study the stability of $I_c$ and $T_c$ of Bi-2223-based and Dy-123-based commercial wires relative to the long-term transport of superconducting current ($\approx 0.7 I_c$) at a temperature of 77 K.

2. Experimental details and results

The first conductor investigated was a HTS tape based on (BiPb)$_2$Sr$_2$Ca$_2$Cu$_3$O$_{10-x}$ (Bi-2223, American Superconductor), a so-called first generation (1-G) wire. It is a multi-filamentary wire encased in a silver matrix and laminated with brass to increase the mechanical strength and provide a hermetic seal.

The other series of experiments were carried out with a HTS coated conductor, a 2-G DyBa$_2$Cu$_3$O$_{7-x}$ tape (Dy-123), manufactured with an epitaxial deposition technology (THEVA, Germany).

The following destinations are used below: Sample B1: Bi-2223 with long-time current load in liquid nitrogen; Sample B2: no current in liquid nitrogen; sample B0 – virgin. Sample D1 and D2 correspondingly for Dy-123.

2.1. Bi-2223 – 1G

The investigated straight sample of Bi-2223 tape (B1) with the dimensions of $108 \times 4 \times 0.4$ mm$^3$ had an $I_c = 126$ A ($j_c = 7360$ A/cm$^2$) at 77 K and a $T_c = 110$ K. It was immersed in a cryostat filled with liquid nitrogen, ready for the application of transport currents for extended time periods. The current passed via indium contacts under compression into the sample, while a pair of voltage taps completed the set-up for a four-point measurement of IV-curves. A reference sample (B2) was placed into the cryostat as well.

![Figure 1. $U(I)$ before and after the application of 85 A through Bi-2223 tape at 77 K.](image)

1. Initial $U(I)$ and after 100 hours, $I_c = 126$ A. (Sample B1).
2. Initial $U(I)$, $I_c = 119$ A. (Sample B2).
3. $U(I)$ after current passing for 323 h, $I_c = 85$ A. (Sample B1).
4. $U(I)$ after a month in a liquid nitrogen, $I_c = 55$ A. (Sample B2).
5. Electric field criterion $1 \mu$V/cm.

Direct current of 85±2A passed through the sample B1 and the voltage-current characteristics $U(I)$ of the sample was recorded from time-to-time. After the first 100 hours of current application $U(I)$ and $I_c$ had not changed. The total time of current passing through the B1 sample was 323 hours, after which the critical current had decreased from 126 A to 85 A (figure 1, curves 1,3).
Figure 1 (curves 2, 4) shows the voltage-current characteristics $U(I)$ of the reference sample B2, which remained in liquid nitrogen for 700 hours without applied current. The critical current of the reference sample B2 decreased from 119 A to 55 A and expansion of the tape was observed. The critical temperature of both samples (B1 and B2) remained unchanged at 110 K.

2.2. Dy-123 tape – 2G

The superconducting DyBCO layer was deposited by e-beam evaporation on a Hastelloy C 276 steel substrate with an oriented MgO buffer layer. A 350 nm thin layer of Ag covered the superconductive layer. The straight sample D1 with the dimensions of $108 \times 4 \times 0.1 \text{ mm}^3$ had an $I_c$ of 63 A and a $T_c$ of 90 K. Also for the DyBCO samples compressed indium helped to reduce the contact resistance.

Also a DyBCO reference sample (D2) was placed into the cryostat. Figure 2 shows the voltage-current characteristics $U(I)$ of the coated conductor sample. After passing a current of 44 A for 300 hours through the sample at 77 K, no changes in critical current, critical temperature and geometry of the tape have been detected in both samples (experimental and reference, D1, D2).

![Figure 2. $U(I)$ before and after the application of a 44 A current to the Dy-123 tape sample at 77 K. In all cases $I_c$ is ~ 63A.](image)

Applying a transport current of 80 A, i.e. exceeding $I_C$, to the Dy-123 tape (D1) for 100 hours in liquid nitrogen did not change the VI-characteristic either.

Thus the experiments showed degradation of the Bi-2223 tape (Samples B1, B2). These results allow us to suppose that the decrease of the critical current in the Bi-2223 samples could be caused by the effect known as “bubbling” rather than by electromigration of ions.

The investigated samples of Dy-123 tapes (Samples D1, D2) were not affected by transport currents applied for 400 h and neither by remaining in liquid nitrogen for 1000 h.

3. SEM investigation

To check the assumption above we investigated the microstructure and the chemical composition of the Bi-2223 tape with the help of a scanning electron microscope (JEOL JSM 5910 – LV) and a Energy Dispersive X-ray Microanalysis (EDS) system. The cross-sections of the Bi-2223 tape samples were polished before microanalysis. To reveal changes in the microstructure and the chemical composition of the tape we compared the images in secondary and back scattering electrons. For characterization of the chemical composition
changes, maps of element distribution were constructed in certain area sample cross-section.

The first line in table 1 lists the average chemical composition of the Bi-2223 phase in the virgin sample (Sample B0). The values were obtained by averaging the analysis results of five arbitrarily chosen points. The remaining four lines (No 1-4) in Table 1 show the element distribution for four selected parts of the studied sample B1 (323 h current and 700 h in liquid nitrogen). The maximum content variation at the chosen points occurred for cooper and calcium. The data given in the Table 1 show that the average chemical composition are similar (within the limits of errors) in all samples.

| Sample | Bi (±0.39) | Pb(±0.37) | Ca(±0.1) | Sr(±0.23) | Cu(±0.23) | O(±0.32) |
|--------|------------|-----------|----------|-----------|-----------|----------|
| Virgin | 10.16      | 0.89      | 10.26    | 10.77     | 15.37     | 52.54    |
| 1      | 9.61       | 0.94      | 9.53     | 10.52     | 12.98     | 56.42    |
| 2      | 10.05      | 0.95      | 9.57     | 11.27     | 13.84     | 54.31    |
| 3      | 10.72      | 0.85      | 11.1     | 11.39     | 16.24     | 49.7     |
| 4      | 10.22      | 0.8       | 10.43    | 11.18     | 14.7      | 52.67    |

* Instrument errors in brackets (σ %).

Figure 3 shows micrographs in back scattering electrons of four different cross-sections of the Bi-2223 tape: of the virgin sample B0(1), of the current sample B1 (2,3) (323 h current and 700 h in liquid nitrogen) and of the references sample B2 (4) (only nitrogen).

![Figure 3](image)

**Figure 3.** SEM micrographs of cross-sections of Bi-2223 hermetic multi-filamentary tape. 1- virgin sample (B0); 2 and 3 – sample B1 (after 323 h current passing); 4- references sample B2 (no current, 700 h in liquid nitrogen).

The micrographs represent cross-sections of a composite multi-filamentary superconductor Bi-2223 in a matrix from silver. Here light sites are silver, grey sites are superconductor. Dark defect fields are the porous regions. Darkly grey strips are brass at the
bottom and top of the images. Attention drawn to the fact, that the area engaged by microporous considerably has increased in Figure 3 (2,3,4) in comparison with Figure 3 (1) - virgin sample.

Chemical element spatial distribution maps taken on an area of \(60 \times 60 \, \mu \text{m}^2\) revealed a tendency to form microareas with a changed content of calcium and cooper relative to the virgin sample. Mapping of the surface showed that these areas of altered composition had a size in the range from 1 to 5 \(\mu \text{m}\). In the current sample B1 the change of chemical composition is more marked. In the reference sample B2 (no current) chemical composition is more homogeneous. The data are not presented here.

4. Conclusion
Investigations of the long-term stability of the critical parameters (\(T_c\) and \(I_c\)) of \(1\)-\(G\) Bi-2223 tape from American Superconductor showed that critical current of the sample decreased from 126 A to 85 A and expension of the tape occurred after 323 hours of applied transport current and 700 hours immersion in liquid nitrogen. The reference sample B2 showed a very similar behaviour. \(T_c\) was not changed by the long-term immersion in liquid nitrogen. Formation of cavities inside of the composite superconductor and its size increased were observed by EDS X-ray microanalysis in both samples B1, B2. The result can be easily explained by generation of deformations inside of the composite HTS at thermocycling. Therefore the decrease of the critical current and formation of microvoids in the Bi-2223 samples could be caused by the effect known as “bubbling” rather than by electromigration of ions.

However data on mapping point to some increase of the size of areas with the changed element constitution in the current sample, it is the characteristic property of an electrostimulated diffusion of ions in HTS. Until now SEM investigation and microanalysis could not give clear answer on the questions. Future detail SEM investigation would be clarified the nature of long-term unstability of the samples.

Good high-current stability (0.7 \(I_c\)) of a 2-\(G\) Dy-123 tape (THEVA) at 77 K was demonstrated during a 400 h long-term test.

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