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Fine structural characterization of the elements of a Nb-Ti superconducting cable

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\textbf{ABSTRACT}

A niobium-titanium alloy (Nb + 47\%Ti) superconducting cable that acts as a current-carrying element in the magnetic system of the international thermonuclear experimental reactor (ITER) has been subjected to cold deformation by drawing. Its microstructure, phase composition and properties have been monitored via atomic force microscopy, electron microscopy and optical microscopy methods at the intermediate stage of cold drawing upon the transition of $\varnothing 1.3 \rightarrow \varnothing 1.2$ mm. Plastic deformation centers are established to be localized at the breakage sites of the cable, and the shape and chemical composition of Nb-Ti fibers are determined at the defect-free region and breakage sites, as well. A partial lack of diffusion Nb barriers is also highlighted around Nb-Ti fibers at the breakage sites.

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1. Introduction

Practical application of superconductors allows essential engineering problems to be solved in areas where the use of traditional electrotechnical materials is economically non-expedient or fundamentally impossible. First of all, the speech is about the fabrication of magnetic systems (including those intended for ITER), accelerating storage complexes, powerful generators and electrical motors, transformers, and other electrotechnical devices.

In a variety of superconductors [1–11], Nb-Ti alloys utilized in the fabrication of current-carrying elements in the magnetic system of the International thermonuclear experimental reactor (ITER) occupy a position of particular importance for researchers. A superconducting cable comprises several thousands of superconducting cores with diameters of 2–5 \( \mu \)m that are fixed by a copper matrix [12]. High demands are placed on these cables, such as magnitude and stability of critical parameters, current characteristics, non-fissility of superconducting fibers (cores), as well as their structural homogeneity over the wire length and small deviations from cross-section...
geometric sizes [3–8].

A great deal of data in this field of research has been obtained in the works dedicated to characterization of superconductivity features of materials upon their structural modification under temperature and force impacts. As is established, the current-carrying capability of a superconductor depends on its microstructure, i.e., shape, dimensions and volume contents of particles that are released in the solid solution decomposition [4–6]. For these reasons, structural failure (defectiveness) of a superconducting composite blank must be taken into consideration at each of the stages of plastic deformation in order to develop successfully the process of fabrication of superconducting wires. First, the requirements for technological plasticity stock of all components of the wire at various stages of the process stay at the forefront of the regulations which needs in-depth knowledge of their deformation behavior and hardening owing to deformation-induced defects that arise during the formation of multifilament Nb-Ti superconductors.

In the production of Nb-Ti-based wires, the most essential stage is cold drawing, whereby the initial composite blank is being deformed from 60–70 mm to ø0.1–1.0 mm. This necessitates ensuring non-fissility of the process and specified density of microdefects in a superconductor, which control the formation of pinning centers (fastening of Abrikosov magnetic vortices in type-II superconductors [1]). The present work is therefore aimed at elucidating the effect of cold deformation by drawing on the structure of a stranded (multifilament) superconductor made from Nb-Ti alloy.

2. Materials and experimental procedure

The synthesis of Nb-Ti alloy-based composite superconductor takes the following steps:

(i) formation of a primary composite blank that is composed of an outer shell of the stabilizing Cu material, a Nb diffusion barrier in the form of a rolled sheet (shell), a ribbon or a thin-wall tube, and an axial cylindrical block of a Nb-Ti alloy;
(ii) extrusion and deformation of the blank to obtain a hexagonal rod;
(iii) cutting a hexagonal rod by measured lengths;
(iv) re-assembling into cushions of the stabilizing Cu material;
(v) extrusion of a secondary composite blank;
(vi) deformation with intermediate thermal processing at temperatures of 370–400°C to the final size of the cable.

Deformation structure and sub-structure of a multifilament cable with superconducting cores of Nb-47.5 wt% Ti alloy were monitored during the intermediate stage of drawing upon the transition of ø1.3 → ø1.2 mm at which failure arose. Composition and structure of a stranded wire were inspected using techniques providing necessary spatial resolution, such as optical microscopy (Neophot-21 and Olympus GX 71), scanning electron microscopy (Philips SEM 515), and atomic force microscopy (Solver PH47-PRO).

To study the fine structure of superconducting cable’s elements, a thin foil was cut off the sample with a focused ion beam in a Quanta 200 3D scanning ion-electron microscope. Structural characterization at the breakage sites of Nb-Ti fibers was implemented via transmission electron microscopy (Jeol 2100, 200 kV).

The cross-section relief peculiarities of the cable’s elements were probed via atomic force microscopy in the constant-force contact mode [9,10]. The essence of the method is that the feedback signal arising in scanning is preset so that a system is able to process the relatively smooth relief’s features rapidly and, on the other hand, to be slow enough to work off steep stairs of the surface. This allows a weak display of smooth relief’s features against the background of high-amplitude maxima of the diffusion barrier. Such a way of visualization may be used for the detection of small structural elements within a large area of the relatively smooth surface.

The chemical composition homogeneity of Nb-Ti fibers, as well as the element distribution at the interface between Nb-Ti alloy core and copper matrix, was controlled via scanning electron microscopy in secondary and back-scattered electron modes on a Quanta 200 3D ion-electron microscope. The element composition of alloy was determined via the energy-dispersion X-ray microanalysis using an EDAX console on the same microscope from the amount of characteristic X-ray photons with energies referred to Ti, Nb and Cu along the line passing through a fiber-matrix boundary. Scanning electron microscopy on a Carl Zeiss EVO 50 setup with an Oxford Instruments X-ray dispersion microanalysis console enabled us to establish structural parameters, such as sizes of grains and subgrains, and their homogeneity with respect to chemical and phase compositions with a necessary accuracy.

The phase composition of elements in the Nb-Ti multifilament superconductor was analyzed via X-ray diffraction on a Shimadzu XRD6000 system where the beam was monochromatized by a curved crystal monochromator. This monochromator allows a signal-to-background ratio to be improved and the weak X-ray lines of minor phases to be distinguished. A superconducting wire sample was exposed to a narrow beam (0.5 mm × 12 mm) at a voltage on the X-ray tube of 40 kV and a current of 30 mA. The exposure time per point was 5 s and the scanning increment was 0.02°. The entire X-ray diffractogram was recorded within 8 h. Experimental data was processed using the PDF+ software completed with the inorganic structure database that comprises 310,000 cards.

3. Results

Combining various analytic methods was aimed at establishing structural peculiarities of a superconducting composite wire and distribution of main chemical elements within it. Nb-Ti stranded superconductor cable is a three-layer system with a buffer (intermediate) layer of Nb-Ti fibers in a copper matrix (composite) between the copper core and the outer shell (Fig. 1). The composite may also include resistive or diffusion barriers that stabilize shells and strong reinforcing elements [12,13].

Metallography applied to cross sections of conductors reveals that Nb-Ti filaments in the intermediate layer at the
interface with a copper superconducting core are initially rounded with the average diameter of \( \sim 10 \mu m \). After exposed to deformation by drawing, all filaments become rhombohedral with diagonals of \( \sim 13 \) and \( 11 \mu m \), respectively.

A diffusion Nb barrier is found around the Nb-Ti fibers placed inside the copper matrix, which is clearly pronounced in the form of relief protrusions at the interface between filament and matrix. In the profilogram constructed using a secant method, the Nb barrier manifests itself by the high-amplitude maxima with widths of 250–260 nm, separated by the low-amplitude relief lines from Nb-Ti fibers and copper matrix. Against the background of a smooth relief of Nb-Ti fibers and copper matrix, one observes a high-amplitude niobium barrier in both transverse (Fig. 2) and longitudinal sections (Fig. 3).

Subjected to severe plastic deformation, copper in the core acquires a submicrocrystalline structure with the average grain size of \( \sim 800 \) nm, where single grains alternate with their conglomerates of up to 8 grains. The deformation of copper in the core is observed to the greatest extent along the core-intermediate layer boundary, where the maximum and minimum grain sizes are found to be respectively \( \sim 2120 \) nm and \( \sim 310 \) nm. In the intermediate layer between fibers in the matrix, copper is represented by uniaxial grains with average sizes of \( \sim 800 \) nm. On the other hand, the average size of copper grains in the conducting shell was \( \sim 1050 \) nm [9,10].

The plastic deformation regions in the form of specific defects or breakage sites of superconducting Nb-Ti filaments were detected via microscopic methods in the cross (Fig. 4a) and longitudinal (Fig. 4b) sections within the intermediate layer of Nb-Ti fibers at the boundary with the copper core.

At the cable's failure site in the matrix of the intermediate layer between Nb-Ti fibers, the average copper grain size was \( \sim 850 \) nm. According to statistical data processing, the average copper grain size of \( \sim 800 \) nm in the matrix is comparable to that of \( \sim 850 \) nm at the point of cable failure. As follows from the double \( t \)-criterion, for this pair of values \( |t| = 1.69 \), and the Student's coefficient for the confidence probability \( \alpha = 0.9 \) is \( t_{\alpha,1} = 1.89 \), i.e., \( |t| < t_{\alpha,1} \) and the difference in average copper grain sizes is thus negligible.

Within the plastic deformation region, Nb-Ti fibers are strongly deformed and lose their regular cross-sectional shape (Fig. 4a), exhibiting different widths within the length of the cable in the longitudinal section (Fig. 5a). Nevertheless, their chemical composition remains unchanged. As is evident from the experimental chemical element distribution maps, Ti and Nb are in Nb-Ti fibers, copper is distributed between Nb-Ti fibers in the copper matrix at the level of 59.18 at\% Cu and the niobium content (as a diffusion barrier around fibers) is about 39.83 at\% Nb (Fig. 5a).

To study the in-depth morphology of the plastic deformation regions, \( \sim 0.5 \)-mm-thick layers were successively ground several times. In accordance with metallographic data, all Nb-Ti fibers have the rounded shape at the inner surface adjacent to the copper core. The first traces of plastic deformation by drawing are observed in the intermediate layer in the region contiguous with the copper shell. On the outer surface side, Nb-Ti fibers possess the diamond-like shape.

The cross section topography after polishing to a depth of 0.5 mm at the cable's breakage site was inspected via optical and atomic force microscopy (Fig. 6). As is evident, the nearby Nb-Ti fibers in the area adjacent to the copper core are irregular in shape and form the deformation localization zone, like in the state prior to polishing. At the point of cable failure, within the matrix of the intermediate layer between Nb-Ti fibers, the average copper grain size is \( \sim 850 \) nm, while statistical processing highlights the average grain size of copper in the matrix of \( \sim 800 \) nm in the defect-free region that is comparable to the copper grain size of \( \sim 850 \) nm at the cable's breakage site. Polishing the cross section to a depth of 0.5 mm at the failure point was found to cause the emergence of the Nb barrier around Nb-Ti fibers in the copper matrix, which manifests itself by the 250-nm-thin high-amplitude maxima in the profilogram and is similar to that in the initial (unpolished) state. The same Nb barrier was detected around Nb-Ti fibers at the breakage point after polishing to a depth of 1 mm.

Metallographic characterization of the cross sections subjected to etching and polishing to a depth of 1 mm reveals that these Nb-Ti fibers are similar in appearance to those unpolished. Nevertheless, the defect of Nb-Ti fibers localized at the cable's breakage site takes on a different form with petal-like fibers merging in a single fiber that evidences fiber thickness.

Fig. 2 – (a) Niobium barrier around fibers in the conducting matrix within the cross section at the defect-free area; (b) profilogram of this domain; (c) 3D image: 1 – copper matrix, 2 – Nb-Ti fibers; 3 – niobium barrier.

variations within the length of the cable. The nearest Nb-Ti fibers at the plastic deformation zone become thus rounded.

Optical microscopy characterization of the cut surface after its polishing to a depth of 2 mm evidences severe changes within the plastic deformation defect-like zone compared to the initial state. The Nb-Ti fibers gradually merge at the breakage site, and the nearest Nb-Ti fibers around the defect possess a rounded shape that is typical of Nb-Ti fibers in the defect-free area. It is worth mentioning that one more defect has been detected in another domain near the “intermediate layer of Nb-Ti fibers in the copper matrix-copper core” interface, which comprises two Nb-Ti fibers with smaller dimensions and irregular shape over against the nearest Nb-Ti fibers. Scanning the cross section surface of the cut via atomic force microscopy in the contact mode after etching and grinding to a depth of 2 mm reveals the presence of the Nb barrier around Nb-Ti fibers in the copper matrix, which has been detected earlier in the initial state through the defect-free area without grinding. In the profilograms, it manifests itself by the high-intensity sharp maxima with a width to 250 nm. The Nb barrier is also present on the inner and outer surfaces of all superconductor Nb-Ti fibers in the copper matrix.

SEM data allow construction of the element distribution maps, where the brightness of images displays qualitatively the chemical element distribution through the area scanned. Such an analysis reveals that titanium is only present in Nb-Ti and copper is localized between fibers, whereas niobium is found in Nb-Ti fibers and between them in the matrix.

At the breakage site, the Nb-Ti fibers loses the regular shape (Fig. 2a), but retain their chemical composition at the level of 63.33 at% Ti and 35.57 at% Nb. As is seen in the element distribution maps, Ni and Nb are localized in fibers, while Cu and Nb are distributed between fibers in the matrix.
Fig. 3 – (a) Niobium barrier around fibers in the conducting matrix within the cross section of the defect-free area; (b) profilingram of this domain; (c) 3D image.

In order to evaluate the Nb barrier thickness, titanium and niobium element distributions were plotted along the line through a fiber-matrix boundary in the defect-free area where Nb-Ti fibers have the regular round shape. The diffusion barrier thickness is established to be 1 μm. Superposing Ti and Nb distribution maps displays a partial lack of the niobium barrier at the cable’s breakage sites around Nb-Ti fibers in the copper matrix (Fig. 5b).

As is aforesaid, the fine structure of the elements in the cable has been studied on a thin foil cut off the sample with a focused ion beam. TEM characterization of the breakage zone of Nb-Ti fibers evidences severe deformation of their structure with neither relaxation traces nor indistinct grain boundaries. No individual dislocations inside the grains of Nb-Ti fiber are resolved, as well. The fiber-matrix interface at the breakage site exhibits a partial lack of the niobium barrier. In turn, the copper structure in the matrix is deformed and relaxed. The Cu grains highlight individual dislocations with no agglomerations. After a while, one observes the niobium barrier with a submicrocrystalline structure that comprises fine and slightly uneven grains within the matrix-fiber boundary (Fig. 7).

The energy-dispersion X-ray microanalysis of the element composition along the line exhibits the presence of Nb, Ti and Cu elements across the selected area that covers the “Nb-Ti fibers–copper matrix” intermediate layer.

The distribution spatial uniformity of the main elements in the fiber and in the matrix has then been estimated using a number of characteristic X-ray photons with the energy $N$ attributed to Ti, Nb, Cu as a function of the measuring point position. The non-uniformly arranged number of characteristic X-ray photons for Ti, Nb, Cu in the fiber (zone I) and in the matrix (zone III) evidences the diffusion layer (zone II) in a segment passing through the fiber–matrix boundary (Fig. 8). It is clear that in the Nb-Ti fiber the characteristic X-ray photons for Ti and Nb are much more predominant than for Cu,
and vice versa in the copper matrix. For the intermediate diffusion layer (Fig. 8) a number of characteristic X-ray photons of Nb remain first at the constant level that confirms the existence of the Nb barrier, as detected by AFM (Figs. 2 and 3). The amount of characteristic X-ray photons for Ti and Nb becomes then lessened with the increasing number of photons for Cu.

The X-ray diffractogram contains the high-intensity reflexes referred to crystal phases, such as pure copper-(Cu) and Nb-Ti alloy. A third phase is associated with intermetallic tetragonal CuTi$_2$ (Fig. 9). There is also a small amount of lines that allow assuming the presence of α-titanium and copper oxide in the compound. The contents of the predominant phases were estimated from the integral intensity ratio to be: Cu – 63.54 wt%; Nb-Ti 35.46 wt%; and CuTi$_2$ – 1 wt%. The crystal lattice parameters were evaluated for each of phases, as well, and are listed in Table 1.

Fig. 5 – Scanning electron microscopy images of Nb-Ti filaments in copper matrix: (a) cross section (a number of spectrum indicates the area probed); (b) element composition distribution map of superposed Ti and Nb elements for the cross section at the breakage sites of Nb-Ti fibers in superconducting cable (light gray – Ti, dark gray – Nb, white – Cu).

4. Discussion

Nowadays, current-carrying capacity of superconducting Nb-Ti alloy wires can be improved by various ways that are typically in increasing the amount of fibers within the blank or in modifying the diffusion barrier structure. In the present study, a partial lack of the niobium barrier and the plastic deformation zone at Nb-Ti fiber breakage sites are localized near to the copper core. This evidences non-uniform deformation of the first and the presence of Nb-Ti layers with the diffusion barrier from the “copper core–intermediate layer of Nb-Ti fibers across the “copper–matrix” interface. One reason of fiber failure in the cable is assumed to be a kind of assembly of a composite blank used for the multifilament wire production. An increase in the content of fibers through the composite cable is due to their diminishing sizes and, consequently, to a rising amount of voids [11]. In a lack of the reliable contact between fibers, as well as of the interplay between fibers and core, the deformation is almost similar to the longitudinal bending that causes non-uniform deformation of
Fig. 6 – AFM of a plastic deformation zone at Nb-Ti fiber breakage sites (shown with arrow) over the depth of polishing: (a) 0 mm, (b) 0.5 mm, (c) 1 mm; (d) 2 mm; 1 – copper matrix, 2 – Nb-Ti fibers.

Fig. 7 – Electron microscopy images of the fine structure of engineering superconducting cable elements: (a) Nb-Ti fiber-copper matrix interface with a lack of Nb barrier; (b) copper matrix-niobium barrier interface: 1 – Nb-Ti fibers; 2 – copper matrix of Nb-Ti filament; 3 – Nb barrier.

Table 1 – Phase composition and crystal lattice parameters.

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<th>Phase</th>
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<th>Space group</th>
<th>a, nm</th>
<th>c, nm</th>
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<tr>
<td>Cu</td>
<td>A1</td>
<td>Fm3m</td>
<td>0.3619</td>
<td></td>
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<tr>
<td>Nb-Ti</td>
<td>A2</td>
<td>Im3m</td>
<td>0.3228</td>
<td></td>
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<tr>
<td>CuTi2</td>
<td>t16</td>
<td>I4/mmm</td>
<td>0.29686</td>
<td>1.06861</td>
</tr>
<tr>
<td>α-Ti</td>
<td>A3</td>
<td>P65/mmc</td>
<td>0.29812</td>
<td></td>
</tr>
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</table>
fibers and of the diffusion barrier. Creating niobium diffusion barriers in Nb-Ti superconducting structures enables one to avoid the interaction at the Nb-Ti alloy/copper interface, which is favorable for the formation of brittle intermetallic Ti-Cu compounds and, consequently, for the breakage of individual superconducting fibers and of the wire as a whole, deteriorating thus the electrical and physical characteristics of the latter. In this work, CuTi₂ inclusions were detected at the cable breakage sites in a superconductors based on Nb-Ti alloys.

It is noteworthy that the deformation heating differs for the core and the intermediate layer owing to multiple drawing. The deformations at the core-intermediate fibrous layer interface at a radius 𝑟 = 𝑅c at the output of the drawing tool are herewith of interest. The calculation of thermoelastic state of the core, intermediate layer and shell is described in detail in [12].

When producing superconducting materials, neither the emergence of pores nor exfoliation of the outer fibrous layer in the matrix is unwanted. An increase in temperature on account of the deformation heating brings thermoelastic deformation of the composite blank’s components, at which the increment of the core radius is

\[ \Delta R_C = \alpha_1 \Delta T_1 R_C \]  \hspace{1cm} (1)

where \( \alpha_1 \) is the linear expansion coefficient of the core material; \( \Delta T_1 \) is the deformation heating of the core material.

On the other hand, the inner radius of the intermediate fibrous layer increases as follows

\[ \Delta R_{I2} = \alpha_2 \Delta T_2 R_c \]  \hspace{1cm} (2)

where \( \alpha_2 \) is the linear thermal expansion coefficient of the material of the intermediate fibrous layer in the matrix; \( \Delta T_2 \) is the deformation heating of the material of the intermediate fibrous layer in the matrix.

The condition of the continuity preservation for a composite blank subjected to the deformation heating by drawing can be written as

\[ \Delta R_{C1} \geq \Delta R_{C2} \]  \hspace{1cm} (3)

It corresponds to a lack of tensile normal stresses at the core–intermediate fibrous layer interface in the matrix, which may drive the exfoliation of nearby Nb-Ti fibers from the copper core due to the thermoelastic strains. In this case, the condition (3) takes the form

\[ \alpha_1 \Delta T_1 \geq \alpha_2 \Delta T_2 \]  \hspace{1cm} (4)

The condition (4) is interpreted as the criterion of preservation of post-deformation continuity of a superconducting blank.

The in-depth characterization (Figs. 4 and 5) of the morphological features of the plastic deformation region at the nearby Nb-Ti fiber–copper core interface testifies to non-uniformity of plastic deformation of the cable and non-fulfillment of the condition (4).

The use of the niobium barrier in the form of sheet and the need for overlapping edges has caused its thinning and breakage upon subsequent deformation (i.e., its partial lack). In this connection, to be utilized in the fabrication of diffusion barriers in superconductors, rolled sheet barrier materials must fulfill strict requirements concerning their grain and mechanical characteristics. Videlicet, the barrier’s material is strongly recommended to have homogeneous fine grains with sizes of 20–60 μm, yield strength to 250 MPa and relative elongation of 45–60%, which would allow it to withstand further joint...
deformation, especially in the case of a composite comprising many other elements.

Approaches traditionally used in the analysis of plasticity of engineering superconductor Nb-Ti alloys [13,14] underline the concepts of uniformity and homogeneity of plastic deformation, as well as of macroscale localization immediately before destruction that contradicts to the modern ideas. According to experimental data on the plastic deformation nature, its intrinsic inhomogeneity from the onset of deformation can lead to the early formation of one or several stable foci of plastic flow localization [15,16] and to the further failure of the filament. Recent thorough studies of deformation macro-localization allowed one to establish the univocal correspondence between the plastic flow law at the given segment of the deformation curve and the type of space-time distribution of plastic distortion tensor components [17]. These facts must be taken into consideration, when elaborating the cold deformation technology by drawing until obtaining superconducting fibers of desired dimensions.

5. Conclusions

In the present study, combining various techniques that provide spatial resolution from micro- to macrolevel allowed establishing previously unknown evaluation peculiarities of localized plastic flow and microstructure formation at the intermediate stage of drawing of engineering Nb-Ti superconductors, which serve as the current-carrying elements for the magnetic system of ITER.

Deformation by drawing was found to exert a strong impact on the structure of a Nb-Ti alloy-based multifilament superconductor. First, Nb-Ti fibers undergo changes in their size and shape in the intermediate layer of superconductor – the fibers at the interface with a copper core possess the rounded shape and those contiguous with a copper shell are rhombic. Second, copper in the elements of the cable subjected to severe plastic deformation gains a submicrocrystalline structure. Third, a specific behavior of the diffusion niobium barrier was observed in the intermediate layer of the cable. As was found, the diffusion Nb barrier with a submicrocrystalline structure around Nb-Ti fibers in the copper matrix was 1-μm-thick in the defect-free region, whereas its partial lack was detected in the plastic deformation localization at the breakage sites of Nb-Ti fibers. Finally, the deformation localization at the macrolevel, as well as the formation of the cable's breakage center, was determined by microstructural evolution peculiarities: the deformation localizations were highlighted at the breakage sites of the cable, where the nearby Nb-Ti fibers exposed to the layer-by-layer grading were irregular in shape. Furthermore, inclusions – CuTi2 intermetallicides – that could cause failure of some of superconducting fibers and of the cable as a whole were discovered at the breakage sites of the cable.

Establishing the afore regularities enabled us to improve the cold deformation by drawing procedure and to reduce the amount of filament breakages within the composite at the relevant steps of cold drawing (direct drawing, railing, and multiple drawing) upon the fabrication of multifilament cable, without going beyond the current technical requirements.

Conflicts of interest

The authors declare no conflicts of interest.

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