Fatigue Behavior of Nb#Sn Composite Strands Used for Iter Magnets

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FATIGUE BEHAVIOR OF Nb$_3$Sn COMPOSITE STRANDS USED FOR ITER MAGNETS

By

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A Thesis submitted to the
Department of Mechanical Engineering
in partial fulfillment of the
requirements for the degree of
Master of Science

Degree Awarded:
Summer Semester, 2011
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I dedicate this manuscript to my parents and my guru, for everything, they bestowed on me.
ACKNOWLEDGMENTS

I am extremely thankful to so many people in ASC and NHMFL. First of all I am grateful to both of my advisers David Larbalestier and Peter Lee who have trusted me and gave me opportunity to work with ASC. They have been very kind and learning institute for me during my research effort at ASC. Peter Lee is the most exceptional microscopist I ever met (or will be meeting in future) and he taught me everything I know about microscopy and analysis of micrograph using Photoshop. He was always available to help me whenever I needed and guided me throughout my thesis. David Larbalestier taught me how to approach the problem and how to present it to make maximum sense out of it, which I believe will be helpful to me throughout my professional carrier. Both of them are great leaders and materials scientists and far better human beings, I was lucky to have both of them as my supervisors. Bill starch has been very generous to me and always available for helping me out with my polishing and Heat treatment. He always maintains the polishing lab full equipped with polishing pads and other polishing solutions. Even sometimes he made special arrangement for me so that I can polish my samples on weekends and that shows his kindness to the students. I learned so many important polishing tips from him which even helped all of the Nb₃Sn strand polishers. I also would like to extend my special thanks to Connie, Charlotte and Hui to make my stay comfortable at ASC. Connie, thanks for all those 25 cents change (sometimes vending machine accepts only exact change) that made my evenings at ASC.

I would also like to acknowledge Matthew Jewell (whose Ph.D. thesis was the very first literature I reviewed as a beginner in this field and was extremely helpful for this work) and Arnaud Devred form ITER International Organization for their guidance and financial support for this work.

I am also thankful to Bob Walsh and Dustin McRae for helping me out with my fatigue testing and allow me to use their Lab. Mr. Bob was always available to answers all my questions regarding fatigue testing which helped me to learn more about mechanical testing. Dustin became such a dear friend of mine during my experiments as we spend so many hours together to make each and every experiment close to perfect (as Dr. David says no experiment is perfect). He taught me all the fatigue test software settings and especially how to stop machine without spoiling your experiment at the end which is quite possible with fatigue test. We often had a
great (technical) talk regarding rotary internal combustion engine (as we both are big fans of rotary engine over conventional piston-cylinder engine).

I am also grateful to Bob Goddard who taught me the SEM and sometimes lent me SEM room keys to run my long hours macro in the evening. My dear friend, Carlos Sanabria was exceptionally helpful to overcome all my Photoshop challenges. Sung and Fumitake were always available for instant SEM help and you can find them on 1-911-SEM-HELP.

I also wish to thank my committee members, Dr. William Oates and Dr. Juan Ordonez, for their time and guidance.

Last but not least, I would like to thank my family for their long distance support and encouragement from India. I am also thankful to Deven Shah & family to allow me to be a part of his family and for all kindness and support throughout my stay in Tallahassee. It would not have happened without you, I love you all.

Manan K. Sheth
Tallahassee, FL, July 2011
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ABSTRACT

In tokamak fusion reactors, such as ITER, superconducting strands are subjected to repeated Lorentz force loading and unloading which may degrade performance over time. In addition to Lorentz force when cooling from reaction heat treatment to cryogenic operation temperature and in any subsequent operational thermal cycles, the different thermal contraction between conduit material and strand bundle introduces further strain loading and unloading due to contraction of strands. The Cu matrix which surrounds the brittle Nb$_3$Sn filaments allows the possibility of some elastic-plastic degradation that can initiate filament cracking and we are seeking to understand if there are design variables that might ameliorate such degradation. In this study we used advanced metallographic techniques to observe the effect of 1000 to 30,000 loading cycles on filament cracking at axial strains from 0.4% to 1.14%. The strands examined include both bronze process and internal tin ITER production strands. After fatigue testing at 77 K the strands were polished in longitudinal cross section and imaged by Scanning Electron Microscopy (SEM). Crack densities within filaments as a function of strain and number of fatigue cycles were quantified from large montages covering $\approx$ 20 mm length of strand. The observed filament crack density is highly sensitive to the Kirkendall voids produced in the Nb$_3$Sn reaction. Although in bronze process strands we saw many cracks in filaments surrounded by voids at lower strains, we rarely observed filament cracks away from voids until the axial strain reached 1%. Similarly for internal tin process strands we found few cracks in filaments surrounded by voids at lower strains but we rarely observed filament cracks away from voids until the axial strain exceeded 0.6%. Contribution of void/filament cracks was found highest among all types of cracks. Moreover, significant cracking of Nb$_3$Sn filaments always starts at axial strain close to the fracture limit in both bronze and internal tin route strands. Bronze process strands showed significant loading cycle effect on filament cracking as crack density increased significantly by increasing loading cycles from 1000 to 10,000. In contrast, internal tin strand did not show any significant loading cycle effect as compared to bronze process strands. A detailed comparison of the development of cracking under different axial strains and increasing loading cycles for different ITER strands will be presented.
CHAPTER 1

**Nb$_3$Sn SUPERCONDUCTORS FOR FUSION REACTOR MAGNET SYSTEM**

Composite superconductors, which consist of fine filaments of superconductors in a normal metal matrix, have gained universal acceptance in magnet construction. In order to increase their electromagnetic stability and reduce the energy loss in time-varying magnetic field applications these composites are usually twisted. The most superconducting magnets have used composites made using ductile superconducting alloys, mainly Nb-Ti [1]. Nb-Ti’s benefits are its low cost, reproducible critical current densities ($J_c$), long piece lengths and ductility. However, the conductor is best suited for performing at lower fields, in the order of 5 to 7 T at 4.2 K. Nb-Ti can supply fields higher than 7 T, as most recently for the Large Hadron Collider where it supplies 8.3 T, but the operating temperature of the system has to be reduced to 1.9 K to achieve this. Thus from earlier research it became clear that Nb-Ti had reached its $J_c$ limit at high fields and higher field magnets such as those to be utilized in International Thermonuclear Experimental Reactor (ITER) and Very Large Hadron Collider (VLHC) require a new choice of superconductor [2].

To improve the design of magnets for superconducting synchrotrons and for magnetic fusion reactors, metallurgical methods for producing filamentary conductors from brittle A15 structured superconductors were developed over the last 40 years. In 1969-1970, a method for fabricating such conductors with Nb$_3$Sn and V$_3$Ga was successfully developed and is now called the bronze process [1]. This brought us Nb$_3$Sn as the best conductor option for high field magnets, whose upper critical field (~30 T) and critical temperature (18.3 K) values are nearly double to those achieved in Nb-Ti; 15.4 T and 9.3 K, respectively. Today, Nb$_3$Sn is used in superconducting magnets to enable diverse technologies like nuclear magnetic resonance (NMR) at 11-23 T, nuclear fusion reactors at 5-13 T, particle accelerator dipoles and quadrupoles at 12-16 T etc [3]. By far the most ambitious (in terms of scale and cost) superconducting magnet system currently under development is the ITER (International Thermonuclear Experimental Reactor), which is scheduled to burn its first plasma in 2019. ITER is a large scale scientific experiment with the intention to demonstrate that it is possible to produce commercial energy from fusion. From 50 MW of input power, the ITER machine is designed to produce 500 MW of
fusion power, the first of all fusion experiments to produce a Q factor (the ratio of energy input to energy output) greater than or equal to 10 [4].

1.1 The ITER superconducting magnet system:

ITER is based on the ‘tokamak’ concept of magnetic confinement, in which the plasma is contained in a doughnut shaped vacuum vessel. The fuel – a mixture of Deuterium and Tritium, two isotopes of hydrogen – is heated to temperatures in excess of 150 million °C, forming a hot plasma. Strong magnetic fields produced by superconducting coils surrounding the vessel and by electric current driven through the plasma are used to keep the plasma away from the walls [4].

Figure 1.1: Main components of the ITER magnet systems. Adopted from [5].

The ITER magnet system consists of 18 superconducting toroidal field (TF), a central solenoid (CS), 6 poloidal field (PF) coils and a set of correction coils that magnetically confine, shape and control the plasma inside the vacuum vessel [4]. In the ITER magnet system, the central solenoid (CS) magnet system drives the plasma, while the toroidal field (TF) and poloidal field (PF) magnets confine and shape the plasma. Table 1.1 presents the technical parameters of these three systems [3].
The Cable-In-Conduit (CIC) conductor configuration for the ITER TF coil is shown in Figure 1.2 A). It consists of around 900 superconducting Nb$_3$Sn strands and around 500 pure Cu strands in a stainless steel jacket, with a small central cooling channel that allows the flow of liquid helium through the conductor. To increase heat removal rate the cables have a void fraction of about 25% to 33%. The CS uses a conductor with a thick ‘circle in square’ Incoloy 908 jacket as shown in Fig 1.2 B).

### Table 1.1: Technical parameters of ITER superconducting magnet systems [3].

<table>
<thead>
<tr>
<th></th>
<th>Peak field (T)</th>
<th>Operating Current (kA)</th>
<th>Conductor choice</th>
<th>Mass of conductor required (tons)</th>
<th>Estimated cost of conductor procurement (M€)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PF</td>
<td>6</td>
<td>45</td>
<td>NbTi</td>
<td>237</td>
<td>N/A</td>
</tr>
<tr>
<td>CS</td>
<td>12.5</td>
<td>41.5</td>
<td>Nb$_3$Sn</td>
<td>123</td>
<td>94</td>
</tr>
<tr>
<td>TF</td>
<td>11.8</td>
<td>68</td>
<td>Nb$_3$Sn</td>
<td>393</td>
<td>300</td>
</tr>
</tbody>
</table>

#### 1.2 Nb$_3$Sn Cable-In-Conduit conductor for ITER TF and CS coils:

The Cable-In-Conduit (CIC) conductor configuration for the ITER TF coil is shown in Figure 1.2 A). It consists of around 900 superconducting Nb$_3$Sn strands and around 500 pure Cu strands in a stainless steel jacket, with a small central cooling channel that allows the flow of liquid helium through the conductor. To increase heat removal rate the cables have a void fraction of about 25% to 33%. The CS uses a conductor with a thick ‘circle in square’ Incoloy 908 jacket as shown in Fig 1.2 B).

Figure 1.2: ITER Cable-In-Conduit conductors. Figure 1.2 A) ITER TF Conductor [3, 5, 6] and figure 1.2 B) ITER CS conductor.
Table 1.2: Parameter for ITER TF and CS windings. Adopted from [5].

<table>
<thead>
<tr>
<th></th>
<th>TF coil</th>
<th>CS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Number of coils</td>
<td>18</td>
<td>6</td>
</tr>
<tr>
<td>Max coil current (MA)</td>
<td>9.1</td>
<td>21.9 at 1M (13.0T)</td>
</tr>
<tr>
<td>Maximum field T/temperature K</td>
<td>11.8/5.0</td>
<td>13.0/4.7</td>
</tr>
<tr>
<td>Turns TF/CS coil</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Radial</td>
<td>11</td>
<td>14</td>
</tr>
<tr>
<td>Toroidal/vertical</td>
<td>14</td>
<td>40</td>
</tr>
<tr>
<td>Total</td>
<td>134</td>
<td>549</td>
</tr>
<tr>
<td>Conductor unit length (m)</td>
<td>760 (double pancakes)</td>
<td>895 (hexa pancakes) 594 (quadra pancakes)</td>
</tr>
</tbody>
</table>

Parameters for TF and CS windings are outlined in Table 1.2. Figure 1.3 shows representative cross-section of CIC conductor. Different parts of CIC are shown in Figure 1.3.

Figure 1.3: Cable-in-Conduit conductor consists of both superconducting and pure Cu strands. Adapted from [3].
1.3 Nomenclature:

Following are the terminologies regarding cable-in-conduit conductor.

1. **Cable**: This is the primary current carrying unit in a magnet. It normally consists of both Cu and superconducting strands. It contains ~ 1000 superconducting strands in a large CIC conductor [3].

2. **Strand**: It is a wire (~ 0.81 mm in diameter) which contains Nb$_3$Sn filaments, one or more diffusion barriers and stabilization Cu [3].

3. **Bundle**: This is the set of all Nb$_3$Sn filaments in the strand. The filaments can be all contained within a single diffusion barrier, or segregated by multiple diffusion barriers [3].

4. **Sub-bundle**: Small grouping of Nb$_3$Sn filaments within the cable in almost all bronze and internal tin strands is called sub-bundles this sub-bundle is normally the principal unit drawn to fine size before restacking to make the final billet [3].
5. Filaments: This is the individual Nb$_3$Sn fiber that carries electric current. Filaments usually vary in diameter from 3-6 μm depending on the strand architecture [3].

1.4 Problem statement:

As Nb$_3$Sn has a high superconducting transition temperature (18.3 K), high upper critical field $H_{c2}$ (~ 29T for doped A15) and high critical current density $J_c$, multifilamentary Nb$_3$Sn is a good choice for high field superconducting magnets (>10 T) [14]. ITER nuclear fusion reactor is by far the most ambitious (in terms of scale and cost) superconducting magnet system currently under development. Nb$_3$Sn conductor has been chosen by ITER for the toroidal field (TF) and central solenoid (CS) CICCs coils. In ITER during magnet operation the conductors carry up to 68 kA in a magnetic field of ~11-13 T [15] and therefore are subjected to heavy transverse loading due to the large Lorentz forces in the coils. These high stress fatigue conditions may lead to filament cracking. Such behavior is thought to be one cause of the irreversible current degradation [11, 16-19] observed in cable-in-conduit conductors. The experiments reported here study the fatigue behavior of individual Nb$_3$Sn strands in a controlled fashion, in the hope of providing strand level understanding of the degradation phenomena seen in ITER conductors. The goal of this research effort is to understand and answer the following three questions:

- 1) At what axial strain does fatigue start to produce observable cracking of Nb$_3$Sn filaments in strand?
- 2) Does repeated axial loading exacerbate the filament cracking?
- 3) Does the strand architecture play any role in fatigue related cracking?

Approach to answer these questions involves three steps: 1) Fatigue testing of Nb$_3$Sn strands 2) Sample preparation of fatigued strands and 3) Analysis of polished longitudinal cross section of fatigued strands using SEM.

Our goal is to develop a systematic and cost effective approach to analyze filament level cracking of fatigued (at different strains and different cycles) Nb$_3$Sn strands. To accomplish this, all fatigue tests will be done at 77 K and after fatigue testing the strands will be polished and analyzed using SEM and image analysis software [20-22]. Fatigue testing will be done at axial strain ranging from 0.4% to 1.14% for bronze process route strands and 0.4% to 0.7% for internal tin route strands. Strain effects will be also observed at different loading cycles, 1000,
10,000 and 30,000. The effect of strain and number of loading cycles on filament cracking will be studied for two bronze route ITER production strands (B4 and B5) and one internal tin route ITER production strand (IT1).
CHAPTER 2

STUDY OF DIFFERENT Nb₃Sn STRAND ARCHITECTURES

Nb₃Sn is a brittle intermetallic A15 phase superconducting compound. Sn atoms occupy the corners and body centered positions in this cubic structure, while the Nb atoms lie on three orthogonal chains on each face of the cube, as shown in figure 2.1. Nb₃Sn is a brittle material and thus cannot be easily drawn into a wire, which is necessary for winding superconducting magnets. To overcome this, wire manufacturers typically draw down composite wires containing ductile precursors such as Nb and Sn, from which the Nb₃Sn is formed by heat treatment at final size (usually after winding into the magnet). These Nb filaments are embedded in a Cu-based matrix for adiabatic and cryogenic stability and to provide medium for Sn diffusion. The greater

![Figure 2.1: Nb₃Sn crystal unit cell (A15).](image)

thermal contraction of the Cu also places the brittle Nb₃Sn filaments under compression and greatly reduces the likelihood of tensile cracking. There are three common wire fabrication techniques: the bronze process, Internal tin (Sn) process and powder in tube (PIT) process.
2.1 Bronze process:

In the area of technological uses of superconductors, one of the most significant developments was the discovery of the so-called “Bronze process” for fabrication of multifilamentary superconducting Nb\(_3\)Sn and V\(_3\)Ga [8]. The process consists of the coreduction in size of a filamentary composite wire (e.g. Nb in a Cu-Sn alloy) and of heat treating it to form the desired superconducting compound Nb\(_3\)Sn at the interface of Nb and Cu-Sn. In this way, fine (~ 3 - 5μm) multifilamentary superconducting wires can be produced using very brittle Nb\(_3\)Sn [8]. In the bronze process, Nb filaments are embedded in a Cu-Sn bronze matrix as shown in figure 2.2, whose maximum Sn content is ~9 at.% [3]. Heat treatment at 650° C for ~ 200 hours is needed to react through few microns of Nb as Sn activity is relatively low in Cu. Without the presence of Cu, Nb\(_3\)Sn can only be produced at very high temperature (> 800 °C) which produces a grain that is much larger and is impractical for wind and react magnet construction.

The bronze process is the Nb\(_3\)Sn fabrication method of choice for achieving small uniform filament size (~3 μm diameter) which results in low hysteretic losses (190 mJ/cm\(^3\) ± 3 T). Bronze process wires, however have low non- Cu \(J_c\) values (< 1000 A/mm\(^2\)) compare to those produced by other fabrication methods [2].

Figure 2.2: Schematic of the bronze process Nb\(_3\)Sn wire architecture. Image courtesy Peter J. Lee. Adapted from [3].
Because the hysteretic losses are proportional to the effective filament diameter, bronze strands are in primary use where low hysteretic loss is required. E.g. rapidly cycled magnets. Another important application is where high field homogeneity and very low persistent-current losses are required. E.g. nuclear magnetic resonance imaging (NMR) magnets [3].

Two bronze route strands (manufactured by B4 and B5) for the ITER toroidal field (TF) coils will be investigated here.

2.1.1 B4 and B5 strands heat treatment

The B4 and B5 strands have almost identical heat treatment schedule ends with a final reaction stage at 650 °C for 100 Hr. As shown in Fig.2.2 both bronze processed strands have uniformly distributed Nb filaments and they remain well separated without any agglomeration throughout the heat treatment. However heat treatment does not produce fully reacted cross-section for both B4 and B5 strands.

2.2 Internal tin

In the internal tin process, a pure (or alloyed) Sn-source is surrounded by Cu-clad, Nb rods in a pure Cu matrix or Nb rods inserted into a pre-drilled Cu can. The low melting point of the Sn means that any warm extrusion (typically required for good composite bonding) must be performed prior to the insertion of the Sn into the composite. The final composite packages can be cold drawn to final size and heat treated to form Nb₃Sn at the site of the Nb rods. Multiple sub-bundles of the Cu, Nb and Sn components may be enclosed by a single diffusion barrier (figure 2.3), which is preferred for fusion and other low-hysteretic loss applications, or each sub-bundle may be enclosed by an individual diffusion barrier, which is preferred for high \( J_c \) applications. In this type of strand, low-temperature (200 °C – 500 °C) heat treatment steps are used to mix the Sn from the core with the interfilamentary Cu. A subsequent higher-temperature step (~650 °C) is then used to convert the Nb to Nb₃Sn [3]. This process has the advantage of allowing more Sn into the overall package and increasing the Sn activity at the Nb-Cu interface (which increases the reaction rate) and does not require multiple anneals during the wire drawing process but has the disadvantage of being more difficult to uniformly reduce from billet to final.
size as a result of the large difference in mechanical properties amongst Nb, Cu and elemental Sn [3].

IT1 strand uses an internal tin design for its TF coil for ITER.

2.2.1 IT1 strand heat treatment

The IT1 strand uses a multiple step heat treatment schedule to mix the Sn from the core (200 °C – 500 °C) with the interfilamentary Cu, followed by a higher-temperature step (~650 °C) to convert Nb to Nb₃Sn. The filaments are ~5-6 μm after full heat treatment (almost twice the diameter of the filaments in the bronze processed strands). In this type of strand more agglomeration of filaments is typically found after full heat treatment.
CHAPTER 3

SAMPLE PREPARATION TECHNIQUE

3.1 Introduction:

Proper sample preparation is critical in obtaining consistent crack density statistics without significant errors being introduced by polishing artifacts. Nb₃Sn strand can be examined in two different cross-sections called transverse and longitudinal cross-section. We have developed very robust polishing procedure at ASC which minimizes the polishing artifacts at great extent. Matthew Jewell has put tremendous effort to develop polishing procedure as a part of his Ph.D. thesis.

3.2 Transverse sample preparation:

The transverse cross-section (region a) as shown in figure 3.1 is perpendicular to the wire axis (indicated by the arrow) and longitudinal cross-section (region b) is parallel to the wire axis. Since the filaments run parallel to the wire axis, transverse cracks will appear in the longitudinal cross-section and longitudinal cracks will appear in transverse cross-section. Transverse cracks are primarily responsible for current degradation since they physically obstruct the axial flow of

Figure 3.1: Schematic of round wire that has been sectioned transversely (region a) and longitudinally (region b). The arrow indicates the direction of the wire axis. Adopted from [3].
electric current along the filament. This implies that longitudinal cross-section will be the primary orientation of focus to identify degraded filaments.

The transverse cross-section is very important in obtaining accurate strand geometric information such as number of filaments, filament size and spacing, Cu-non Cu ratio, barrier thickness etc. The usual procedure to mount a transverse cross section is to first cure an empty, 1 inch (25.4mm) metallographic puck made from conductive phenolic resin (such as Buehler Konductomet or similar), and then drill a hole just large enough to fit one or (more) of each strand being investigated and then re-cure the puck with a small amount of additional resin. A very close approximation to a true transverse cross-section can be obtained with a properly sized drill hole and samples at least 5mm long [3].

The typical polishing procedure for a transverse cross-section of Nb$_3$Sn strand involves polishing with 400 grit paper (4 -5 minutes at 150 rpm) and 600 grit paper (5-6 minutes at 150 rpm) to remove the damaged layer introduced by sectioning the wire with cutters as well as flatten the sample and then 8 micron apex blue (30 minutes at 100 rpm) will be helpful to remove major scratches developed during 400 and 600 grit paper. To get fine polishing surface, diamond polishing is preferred after all these steps. 6um diamond suspension (15 minutes at 50 rpm), 3um diamond suspension (15 minutes at 50 rpm) and 1um diamond suspension (10 minutes at 50 rpm) are required steps before final polishing at 0.05um using colloidal silica on either a medium nap cloth or a napless synthetic “sponge” cloth such as the Buehler Chemomet pad on vibromet. In addition to the other polishing steps, vibrational polishing is very effective in removing damage caused by previous polishing steps.

To observe the progression of a transverse cross-section polished we imaged cross-sections using Laser confocal microscopy after each polishing step. All images were taken by undergraduate student Eric Sloan.

1) Post 400 Grit Paper (~ 22 μm) polishing
2) Post 600 Grit paper (~ 14 μm) polishing

Figure 3.2: Transverse cross section of ITER B4 strand post 400 grit paper polishing. Image was taken at 20x.

Figure 3.3: Transverse cross section of ITER B4 strand post 400 grit paper polishing (zoom in view of red box in Fig. 3.2).
3) Post 8 μm fixed diamond pad polishing (such as the Buehler Apex DGD pad)
4) Post 3 μm diamond suspension polishing
5) Post vibromet 1 hour
Figure 3.10: Transverse cross section of ITER B4 strand post vibromet 1 hour polishing. Image was taken at 20x. Some unusual observable white round spots on image are due to image processing.

Figure 3.11: Transverse cross section of ITER B4 strand post vibromet 1 hour polishing (zoom in view of red box in Fig. 3.10).

6) Post vibromet 6 hours
Figure 3.12: Transverse cross section of ITER B4 strand post vibromet 6 hours polishing. Significant increase in voids at this stage because the vibrational technique reveals the interfilamentary void structure by removing debris and smeared Cu. Image was taken at 20x.

Figure 3.13: Transverse cross section of ITER B4 strand post vibromet 6 hours polishing (zoom in view of red box in Fig. 3.12).
7) Post vibromet 12 hours

Figure 3.14: Transverse cross section of ITER B4 strand post vibromet 12 hours polishing. Image was taken at 20x.

Figure 3.15: Transverse cross section of ITER B4 strand post vibromet 12 hours polishing (zoom in view of red box in Fig. 3.14).
8) Post vibromet 24 hours

Figure 3.16: Transverse cross section of ITER B4 strand post vibromet 24 hours polishing. Image was taken at 20x.

Figure 3.17: Transverse cross section of ITER B4 strand post vibromet 24 hours polishing (zoom in view of red box in Fig. 3.16).
Figure 3.2 to 3.17 show the progression of a common sub-bundles as they progress from grit paper polishing to diamond suspension to the final vibromet polishing. Vibrational polishing reduces the instances of fractured filaments and also clears smeared Cu and polishing debris from the interfilamentary voids in the sample [3]. Figure 3.18 shows the ITER B4 transverse cross section after final polishing.

![Image](image1.png)

Figure 3.18: Transverse cross-section of ITER B4 after final polishing. Notice the smaller interfilamentary voids are clearly visible. Image was taken at 20x.

### 3.3 Longitudinal sample preparation:

Analysis of longitudinal cross section is very important in the case of fatigued sample analysis as transverse cracks reveal in longitudinal cross section are the primary current blocking feature. Longitudinal sample preparation was very well summarized in Matthew Jewell’s Ph.D. thesis [3] and some of his points will be repeated here again for better understanding. Polishing on longitudinal cross-section requires a careful optimization between polishing rates and
polishing damaged induced. Some general principles that should guide the design of a polishing
procedure for composites with brittle filaments such as Nb$_3$Sn are provided in Matthew Jewell
Ph.D. thesis [3]. These guidelines were developed in experimental context of his PhD thesis but
it was mentioned that they are in good agreement with suggestions provided in metallography
handbooks, such as that of Vander Voort. The polishing procedure mentioned in [3] was
modified to some extent as a new polishing machine was installed in ASC in 2010 which was
more reliable in polishing a flat surface and to a desired depth under only 1 pound force; this
allowed us to eliminate all manual polishing steps. For longitudinal polishing proper material
removal is essential in order to image the full mid-diameter cross-section and some direct
method of specifying the amount of material removed is advisable. Measuring puck thickness at
each step could be the simplest and best method for direct measurement. There was a strict
tolerance on grinding depth to be kept as the samples used in this study were 0.81 mm diameter
and the metallographic puck does a poor job of holding once > 50% of the diameter has been
polished. In this study a polishing depth of $350 \mu m \pm 50 \mu m$ was targeted for each
metallographic puck based on [3].

Considering all these facts, general polishing procedure should be as follow:

- **First**, start polishing with 600 grit paper size with water as lubricant at 150 rpm speed.
  Total 7-8 minutes of polishing by this grit paper is needed to remove at least 0.3 mm of
  material as rest of the smaller grit size steps do not remove sufficient material. It is
  recommended to use new abrasive pad for every two minutes of polishing.

- **Second** step uses an 8 μm fixed diamond pad (such as the Buehler Apex DGD pad) with
  water as a lubricant at 100 rpm speed. At this stage thick damage layer developed during
  the previous step must be removed without introducing a thick damage layer of its own.
  This step requires 1 hour polishing time. It is recommended to dress the pad every 5
  minutes for about 30 - 60 seconds using an 8 μm stick to improve the grinding surface of
  apex pad.

- **Next** three steps are polishing with 6 μm, 3 μm and 1 μm diamond suspension at 50 rpm
  speed to get fine polishing surface and to make sample ready for final polishing in
  vibromet. At each step it is recommended to spend 20-30 minutes of polishing time.
These steps remove materials at very slow rate and produce little change in sample thickness.

- Final polishing involves 48-60 hours of vibromet polishing with 0.05 μm colloidal silica at only 20%-30% of the maximum vibrational amplitude. Many hours of vibrational polishing are required to completely remove the polishing damage in longitudinal cross-section.

After vibromet polishing, the sample is cleaned with DI water using a Buehler pad in vibromet for 30 minutes. After DI water cleaning to keep the sample surface stain free, a high pressure filtered air jet is used for 5 minutes continuously to remove all moisture from the surface. With this polishing procedure clean, flat, damage free longitudinal cross sections of Nb$_3$Sn composite wires can be prepared.

To observe the progression of a longitudinal cross-section during the polishing procedure we imaged cross-sections using Laser confocal microscopy after multiple polishing steps. All images were taken by undergraduate student Eric Sloan.

1) Post 600 grit paper (~14 μm)

Figure 3.19: Longitudinal cross section of ITER B4 strand post 600 grit paper polishing. Image was taken at 50x.
2) Post 8 μm fixed diamond pad polishing (such as the Buehler Apex DGD pad)

Figure 3.20: Longitudinal cross section of ITER B4 strand post 600 grit paper polishing (zoom in view of red box in Fig. 3.19).

Figure 3.21: Longitudinal cross section of ITER B4 strand post 8 μm polishing. Image was taken at 50x.
3) Post 3 μm diamond suspension polishing

Figure 3.23: Longitudinal cross section of ITER B4 strand post 3 μm polishing. Image was taken at 50x.
Figure 3.24: Longitudinal cross section of ITER B4 strand post 3 μm polishing (zoom in view of red box in Fig. 3.23).

4) Post vibromet 1 hour

Figure 3.25: Longitudinal cross section of ITER B4 strand post vibromet 1 hour. Image was taken at 50x.
5) Post vibromet 6 hours

Figure 3.26: Longitudinal cross section of ITER B4 strand post vibromet 1 hour (zoom in view of red box in Fig. 3.25).

Figure 3.27: Longitudinal cross section of ITER B4 strand post vibromet 6 hours. Image was taken at 50x.
Figure 3.28: Longitudinal cross section of ITER B4 strand post vibromet 6 hours (zoom in view of red box in Fig. 3.27).

6) Post vibromet 12 hours

Figure 3.29: Longitudinal cross section of ITER B4 strand post vibromet 12 hours. Image was taken at 50x.
Figure 3.30: Longitudinal cross section of ITER B4 strand post vibromet 12 hours (zoom in view of red box in Fig. 3.29).

7) Post vibromet 24 hours

Figure 3.31: Longitudinal cross section of ITER B4 strand post vibromet 24 hours. Image was taken at 50x.
8) Post vibromet 48 hours
Figure 3.34: Longitudinal cross section of ITER B4 strand post vibromet 48 hours (zoom in view of red box in Fig. 3.33).

9) Post vibromet 66 hours

Figure 3.35: Longitudinal cross section of ITER B4 strand post vibromet 66 hours. Image was taken at 50x.
Figure 3.36: Longitudinal cross section of ITER B4 strand post vibromet 66 hours (zoom in view of red box in Fig. 3.35).

Figure 3.19 to 3.36 show the progression of polished longitudinal cross-section as they progress from grit paper polishing to diamond suspension to the final vibromet polishing.
CHAPTER 4

FATIGUE TESTING OF Nb$_3$Sn COMPOSITE STRANDS

4.1 Fatigue phenomena due to Lorentz force in ITER magnets:

In ITER, during magnet operation, the cable-in-conduit conductors (CICCs) carry current up to 68 kA in a magnetic field of ~11-13 T. The CICCs are composed of more than 1000 strands with a diameter of 0.81 mm and they have a nominal void fraction of 33%-36% [9]. This large current and magnetic field creates Lorentz force always acting perpendicular to conductor axis. Symbolically,

$$ F_L \approx B \times I $$

Where, $F_L$ = Lorentz force, $B$ = Magnetic field intensity, $I$ = electrical current.

Since this force acts perpendicular to the current direction, it necessarily acts perpendicular to the strain axis as well. Thus Lorentz force is constantly trying to displace the superconducting strand from its neutral position, which can cause Nb$_3$Sn (a classic brittle intermetallic) filaments to fracture. Even though the cabled strands have little space to move, Nb$_3$Sn strands may bend sufficiently under Lorentz forces to introduce the possibility of local strain values exceeding the irreversible strain limit of Nb$_3$Sn. The complex cabling geometry not only imposes distributed magnetic loads along each strand but also cumulated loads from other strands transferred by the strand to strand contacts. These bending and contact loads on the Nb$_3$Sn strands affect the critical current and create a periodic strain variation along the filaments [10]. In addition, differential thermal contraction under cooling from reaction heat treatment to cryogenic operation temperature and subsequent operational thermal cycling introduces additional strains on the strand. Different approaches have been used to analyze bending load [9, 10], contact load [9, 10] and axial load effect [11] but surprisingly less effort has been put on investigation of loading cycle effect (Fatigue effect) on transport properties of conductor even though the ITER coils are expected to be cycled thousands of times. Y. Miyoshi in [11] nicely presented the effect of axial strain on transport properties of strand but the study was focused on filament damage at increasingly applied axial strain for 1 cycle only. Our research effort is also focused on filament damage due to axial strain but here the main aim was to study the effect of number of loading
cycles rather than just one cycle on filament damage. Understanding of the effect of loading cycles is very important as central solenoid is pulse magnet and plasma cycles in central solenoid over the time period create fatigue in the conductor. The Cu matrix which surrounds the brittle Nb$_3$Sn filaments allows the possibility of some elastic-plastic degradation that can initiate filament cracking and it is important to understand if there are design variables that might ameliorate such degradation.

4.2 Fatigue behavior in different types of materials:

Fatigue is a form of failure that occurs in structures subjected to dynamic and fluctuating stresses (e.g. bridges, aircraft and machine components). Under these circumstances it is possible for failure to occur at a stress level considerably lower than the tensile or yield strength for a static load. The term “fatigue” is used because this type of failure normally occurs after a lengthy period of repeated stress or strain cycling. Fatigue is important as it is the single largest cause of failure in metals, estimated to comprise approximately 90% of all metallic failures. Furthermore, it is catastrophic and insidious, occurring very suddenly and without warning. Fatigue failure is brittle like in nature even in normally ductile metals, in that there is very little, if any, gross plastic deformation associated with failure [13]. The process occurs by initiation and propagation of cracks and ordinarily the fracture surface is perpendicular to the direction of an applied tensile stress [13]. The nominal maximum stress values are less than the ultimate tensile stress limit and may be below the yield stress limit of the material [12]. If the loads are above a certain threshold, microscopic cracks will begin to form at the surface. Eventually a crack will reach a critical size, and the structure will suddenly fracture. In ITER strands, we have a complex cyclic strain environment in which a very ductile alloy (Cu matrix) surrounds a very brittle metallic (Nb$_3$Sn filaments) and holds it under compression and this environment is further complicated by the presence of voids at the filament/matrix interference and often a ductile unreacted Nb core at the center of the filaments.
4.3 Fracture in materials:

Fracture is considered the end result of plastic-deformation processes, and there are many different ways that plastic deformation leads to failure. In fracture an object or material separate into two or more pieces under the action of stress. Here, ductile material fracture and brittle material fracture will be discussed.

4.3.1. Ductile fracture:

Figure 4.1: Stress-strain curve of ductile material (structural steel). Adopted from [12].

A. Apparent stress (F/A0)
B. Actual stress (F/A)
1. Ultimate strength
2. Yield Strength
3. Rupture
4. Strain hardening region
5. Necking region
Ductile materials generally exhibit a very linear stress-strain relationship up to a well defined yield point (Figure 4.1). The linear portion of the curve is the elastic region and the slope is the modulus of the elasticity or Young’s modulus. After the yield point, the curve typically decreases slightly because of dislocations escaping from Cottrell atmospheres. As deformation continues, the stress increases on account of strain hardening until it reaches the ultimate strength. Until this point, the cross-sectional area decreases uniformly. Beyond this point a neck forms where the local cross-sectional area decreases more quickly than the rest of the sample resulting in an increase in the true stress [12]. However if the curve is plotted in terms of true stress and true strain the stress will continue to rise until failure. Eventually the neck becomes unstable and the specimen ruptures (fractures).

In ductile fracture, extensive plastic deformation takes place before fracture. A ductile crack spreads as a result of intense localized plastic deformation of the metal at the tip of the crack.

Figure 4.2: Schematic representations of the steps in ductile fracture (in pure tension) and ductile failure of specimen strained axially. Adopted from [12].
The basic steps are void formation, crack formation (void coalescence), crack propagation and resulting in a cup and cone shaped failure surface as shown in Figure 4.2.

4.3.2. Brittle fracture:

Brittle materials do not have a yield point and do not strain harden. Therefore the ultimate strength and breaking strength are the same. A typical stress-strain curve for a brittle material will be linear (Figure 4.3). Usually, there is no necking of brittle specimen under axial strain. In brittle fracture, no apparent plastic deformation takes place before fracture. In brittle crystalline materials, fracture can occur by cleavage as the result of tensile stress acting normal to crystallographic planes [12]. In amorphous solids, by contrast, the lack of a crystalline structure results in a conchoidal fracture (brittle materials fracture this way when they do not follow any natural planes of separation), with cracks proceeding normal to the applied tension [12] (Figure 4.4). In brittle fracture the movement of the crack involves very little plastic deformation of the metal adjacent to the crack.

Figure 4.3: Stress ($\sigma$) – strain ($\varepsilon$) curve for brittle materials. Adopted from [12].
4.4 Tensile test of ITER strands:

The tensile test is a fundamental test in which a specimen is subjected to uniaxial tension until failure. The tensile test measures important properties such as the ultimate tensile strength, yield strength, Young’s modulus and strain hardening characteristics.

Tensile test experimental setup:

We have developed a reliable tensile test setup as shown in Figure 4.5 for tensile testing of ITER strands. All tensile and fatigue tests have been performed at Materials Development and Characterization Laboratory at NHMFL. As shown in Figure 4.5, the specimen (strand) was held between a top and bottom holder firmly with a small load of 20 N to keep it straight during tensile or fatigue testing. An extensometer was attached to the specimen to measure the applied strain. During the tensile test machine was run under stroke control at displacement rate 0.5 mm/min until the specimen broke. The same procedure was followed for all the tensile testing of the ITER strands. This test gives us the stress-strain curve from which we can obtain the stress corresponding to each desired strain. All tensile tests have been performed at 77 K. Here the stress-strain curves of the ITER B4, B5 and IT1 strands are presented.
The tensile test was a necessary first step to perform fatigue testing because it provides us required stress (indirectly force) corresponding to the strain for fatigue test. For example, from the stress-strain curves of the ITER strands (Figure 4.6) we obtained the data shown in Table 4.1. Table 4.1 shows us how much force required to produce a particular strain. The fatigue test is more complex than the tensile test; the biggest challenge is to apply the same force for each of the many cycles.

Figure 4.5: Tensile test apparatus for testing Nb$_3$Sn strands with the extensometer attached to the specimen.

4.5 Fatigue testing of ITER strands:

The tensile test was a necessary first step to perform fatigue testing because it provides us required stress (indirectly force) corresponding to the strain for fatigue test. For example, from the stress-strain curves of the ITER strands (Figure 4.6) we obtained the data shown in Table 4.1. Table 4.1 shows us how much force required to produce a particular strain. The fatigue test is more complex than the tensile test; the biggest challenge is to apply the same force for each of the many cycles.
Figure 4.6: Stress-strain curves for ITER B4, B5 and IT1 strands. An unloading was performed to obtain the Young’s modulus of the Nb$_3$Sn composite strands.
Table 4.1: Comparison of force (N) – strain values for two bronze route (B4 and B5) strands and one internal tin route strand (IT1) from the stress-strain curves of each individual strand. The stress was converted into force by multiplying 0.515 \text{mm}^2 (the cross-section area of strand).

<table>
<thead>
<tr>
<th>B4 strand</th>
<th>B5 strand</th>
<th>IT1 strand</th>
</tr>
</thead>
<tbody>
<tr>
<td>Strain</td>
<td>Force (N)</td>
<td>Strain</td>
</tr>
<tr>
<td>0.40%</td>
<td>83</td>
<td>0.40%</td>
</tr>
<tr>
<td>0.60%</td>
<td>110</td>
<td>0.60%</td>
</tr>
<tr>
<td>1.00%</td>
<td>164</td>
<td>1.00%</td>
</tr>
<tr>
<td>1.22%</td>
<td>192</td>
<td>1.16%</td>
</tr>
</tbody>
</table>

Figure 4.7: The data from Table 4.1 is plotted to compare the required force (N) and corresponding strain values for two bronze route (B4 and B5) strands and one internal tin route strand (IT1).
The values in red/bold color in Table 4.1 indicate the force-strain values corresponding to the breaking point under tensile test. Figure 4.7 shows that to apply the same strain, internal tin route IT1 strand requires more force compared to both bronze route strands B4 and B5. While the IT1 strand requires more force to apply the same strain than the bronze strands, it breaks at a lower strain value (~ 0.83%) than either of the bronze route strands (B4 ~ 1.22% and B5 ~ 1.16%). This may be because of the fact that internal tin route IT1 strand has more Nb before heat treatment and due to that more Nb$_3$Sn (brittle material) after heat treatment which experiences less strain hardening before fracture compare to bronze route strands.

The fatigue test can be performed under strain control or load control. For fatigue testing under strain control we need to attach an extensometer to the specimen (the same experimental setup as shown in figure 4.5 for the tensile test). The extensometer has a sharp needle edge grips by which it is attached to the specimen. Now concern here is that the sharp needle edge grips may create stress concentrations on the specimen and it may also slip in the liquid nitrogen during the fatigue test; thus we chose to use load control. However, it is still important to have a record of displacement during the fatigue test to make sure that applied strain does not runaway during cyclic loading and keep the applied strain constant for particular number of cycles. For this purpose we added a separate extensometer holder with 3 extensometers at the top end of the specimen holder as shown in Figure 4.8.

**Fatigue test procedure:**

As shown in figure 4.8, three extensometers were attached to the specimen holder in the fatigue test setup. We track the record of displacement by running the first cycle of the fatigue test under stroke control as shown in figure 4.9 and then the rest of the cycles in force control (load control). Here only fatigue test procedure for B4 strand 1% strained for 10,000 cycles is discussed but we followed the same procedure for all other tests. Table 4.1 shows that 164 N corresponds to 1% strain for the B4 strand. The first cycle was run in stroke control mode at stroke of 0.5mm/min up to 164 N force and then back to 20 N force (the initial load to keep the sample straight). During this cycle we record two displacement values 0.7383 mm (Max. Displacement) and 0.443 mm (Min. Displacement) corresponding to 164 N and 20 N respectively as shown in Figure 4.9. After the first cycle in stroke control, force control was used for the rest of the cycles, oscillating between two loads of 164 N and 20 N.
During force control we periodically monitored the two displacement values (Maximum and Minimum) (e.g. after every 500 cycles in this case, Table 4.2). Figure 4.10 is the plot of (Max. Displacement minus Min. Displacement) versus number of cycles which shows some strain hardening because of the ductile copper matrix of the strand. Because of strain hardening the Max. displacement values decrease as the number of cycles increases and minimum displacement values increase, which leads to a constant displacement range (Figure 4.10) (Max. displacement – Min. displacement) after a certain number of cycles.

Figure 4.8: Fatigue test setup, with three extensometers attached to the specimen holder. The average of readings from all three extensometers provides feedback for the load cycle.
Table 4.2: Max. and Min. displacement data from extensometer.

<table>
<thead>
<tr>
<th>Cycle No.</th>
<th>Max. Disp.(mm)</th>
<th>Min. Disp.(mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.7383</td>
<td>0.443</td>
</tr>
<tr>
<td>1</td>
<td>0.714</td>
<td>0.49</td>
</tr>
<tr>
<td>50</td>
<td>0.716</td>
<td>0.49</td>
</tr>
<tr>
<td>500</td>
<td>0.716</td>
<td>0.49</td>
</tr>
<tr>
<td>850</td>
<td>0.706</td>
<td>0.51</td>
</tr>
<tr>
<td>1550</td>
<td>0.7034</td>
<td>0.514</td>
</tr>
<tr>
<td>2150</td>
<td>0.7014</td>
<td>0.515</td>
</tr>
<tr>
<td>2650</td>
<td>0.7013</td>
<td>0.516</td>
</tr>
<tr>
<td>3150</td>
<td>0.7007</td>
<td>0.516</td>
</tr>
<tr>
<td>3650</td>
<td>0.7</td>
<td>0.516</td>
</tr>
<tr>
<td>4150</td>
<td>0.698</td>
<td>0.518</td>
</tr>
<tr>
<td>4650</td>
<td>0.6975</td>
<td>0.518</td>
</tr>
<tr>
<td>5150</td>
<td>0.6979</td>
<td>0.518</td>
</tr>
<tr>
<td>5650</td>
<td>0.6977</td>
<td>0.5193</td>
</tr>
<tr>
<td>6150</td>
<td>0.6975</td>
<td>0.5187</td>
</tr>
<tr>
<td>6650</td>
<td>0.6972</td>
<td>0.5189</td>
</tr>
<tr>
<td>7150</td>
<td>0.6969</td>
<td>0.5195</td>
</tr>
<tr>
<td>7650</td>
<td>0.6974</td>
<td>0.52</td>
</tr>
<tr>
<td>8150</td>
<td>0.6954</td>
<td>0.521</td>
</tr>
<tr>
<td>8650</td>
<td>0.6951</td>
<td>0.5214</td>
</tr>
<tr>
<td>9150</td>
<td>0.695</td>
<td>0.5213</td>
</tr>
<tr>
<td>9650</td>
<td>0.6955</td>
<td>0.5211</td>
</tr>
<tr>
<td>10,000</td>
<td>0.6956</td>
<td>0.5206</td>
</tr>
</tbody>
</table>
Figure 4.9: First cycle under stroke control.

Figure 4.10: (Max. disp. - Min. disp.) versus number of cycles.
From Table 4.2 it can be concluded that there was not any strain run away (strain value was approximately 1% (0.01) throughout the experiment) since all Max. and Min. displacement values during experiment are within the displacement range [0.443, 0.7383]. All fatigue tests were performed at 1Hz frequency. Once the desired numbers of loading cycles were attained, the machine was stopped and the specimen was cut from the top and bottom holders and kept it in a straight 0.82 mm diameter quartz tube for polishing. Figure 4.11 to Figure 4.13 illustrate the effect of the number of loading cycles on strain hardening for the different ITER strands. All displacement data are the average of the displacement values from the three extensometers.

**B4 strand:**

![Graph](image)

**Figure 4.11:** ITER B4 strand displacement data plotted as (Max. disp. – Min. disp.) versus number of loading cycles. A) 1% strain applied for 1000 cycles. B) 1% strain applied for 30,000 cycles. From figure A) and B) it is clear that as the number of cycles increases the displacement decreases because of the strain hardening. To see the effect of 10,000 cycles please refer to Figure 4.10.
B5 strand:

Figure 4.12: ITER B5 strand displacement data plotted as (Max. disp. – Min. disp.) versus number of loading cycles. A) 1% strain applied for 1000 cycles. B) 1% strain applied for 10,000 cycles. C) 1% strain applied for 30,000 cycles. From all three figures it is clear that as the number of cycles increases the rate of strain hardening decreases until there is no further hardening (~ 10-20 K cycles).

In Figure 4.12 C) there is a noticeable difference in (Max.disp. – Min.disp.) for the first three data points because one of the three extensometers slipped during the fatigue test after first cycle in force control so it just decreased the value of (Max. disp. – Min. disp.) from third data point onwards during force control mode and that is why there is noticeable difference between second and third data point. However, this slippage did not affect the experiment.
IT1 strand:

Figure 4.13: ITER IT1 strand displacement data plotted as (Max. disp. – Min. disp.) versus number of loading cycles. A) 0.7% strain applied for 1000 cycles. B) 0.7% strain applied for 10,000 cycles. C) 0.7% strain applied for 30,000 cycles.

Figures 4.11 to 4.13 show that both the bronze route (B4 and B5) strands and the internal tin route (IT1) strand experienced strain hardening over the first 10,000 – 20,000 loading cycles. However, none of the strands broke under cyclic loading even after 30,000 cycles. The effect of loading cycles on filament cracking for both bronze route and internal tin route ITER strands is summarized in Chapter 5.
CHAPTER 5

STUDY OF FILAMENT CRACKING UNDER UNIAXIAL STRAIN AND INCREASING LOADING CYCLES

After fatigue testing of the Nb$_3$Sn strands, the next step was to analyze the filament integrity in the fatigued strands. Fully heat treated ITER bronze route strands (B5 and B4) were fatigued at 0.4%, 0.6% and 1% strain for 1000, 10,000 and 30,000 loading cycles. ITER internal tin route strand (IT1) was fatigued at 0.4%, 0.6% and 0.7% strain for 1000, 10,000 and 30,000 loading cycles. Fatigue testing was performed at 77 K and at 1 Hz frequency. To analyze the fatigued samples, a systematic sample preparation approach was developed.

5.1 The sample preparation approach to analyze the filament integrity in fatigued Nb$_3$Sn strands:

Sample preparation is critical step to obtain the reproducible crack density statistics. A fully reacted, but unstrained strand provides control on polishing procedure. So it is recommended to keep the unstrained strand along with strained (fatigued) strands into the same puck (1 inch). For example as shown in Figure 5.1, B4 strands strained at 0.4%, 0.6% and 1% strain for 1000 cycles were polished along with unstrained fully heat treated strand. The lengths of the strands are

![Figure 5.1: Polishing puck with embedded B4 strands of different applied strains. Strains were applied for 1000 cycles.](image)
varied from 12-20 mm and the different lengths are used to identify different samples. These strands were embedded in a conducting Bakelite metallographic puck and carefully polished as described in Chapter 3 using grit papers, diamond suspensions to remove half the thickness of the embedded strands. Final polishing was conducted with 0.05 μm silica suspension to permit high resolution imaging in SEM. To measure the distributions of crack in the Nb$_3$Sn strand under uniaxial cyclic loading following approach was followed:

The ITER B4 strand was strained at 1.1% and 1.14% strain for 1000 cycles. To see the effect of fatigue on crack distribution within Nb$_3$Sn strand, both End and Center pieces of fatigued strands were analyzed using the SEM. The following results were found at the end of the analysis.

Figure 5.2: ITER B4 strand fatigued (strained) at 1.1% and 1.14% strain for 1000 cycles. Both end and center pieces of strands were polished to see the crack distribution uniformly along the Nb$_3$Sn strand.
From Figure 5.3 we can conclude that the "center" piece of the strand has more filament cracks compared to the "end" piece of the strand for the same applied strain and number of cycles. Therefore, it is recommended to polish the center piece of the fatigued strand as that portion contains a higher density of cracks than the end of the strand. To illustrate this point see the Figure 5.4 and Figure 5.5, which show the crack distributions of the center piece and the end piece of 1.14% strained strand for 1000 cycles respectively.

Figure 5.3: Crack densities for ITER B4 strand. Strain applied for 1.1% and 1.14% for 1000 cycles. Both the Center and End pieces of fatigued strands were analyzed to observe the crack distribution uniformly along the Nb₃Sn strand.
Figure 5.4: Crack distribution for ITER B4 strand (Center piece), fatigued at 1.14% strain for 1000 cycles. On top of the bar-chart, each white dot in a black background marks the location of a crack. The X-position represents the location of the 1 mm wide analysis band along the length of the strand from the left end of the strand in the polishing puck.
In Figure 5.4 we can see that after the X-position of 10 mm, there are more cracks on the bottom side of the strand which suggests that there may have been some minor bending of the strand during experiment (although none was observed during the experiment) or may be because of large axial strain (1.14% strain, close to fracture limit) introduces a localized instability. The results presented in this thesis are based on the analysis of center pieces of the fatigued strands unless otherwise stated.

Figure 5.5: Crack distribution for ITER B4 strand (End piece), fatigued at 1.14% strain for 1000 cycles. On top of the bar-chart, each white dot in a black background marks the location of a crack. The X-position represents the location of the 1mm wide analysis band along the length of the strand from the left end of the strand in the polishing puck.
5.2 ITER B5 strand (bronze route) fatigue test crack density results:

Fully heat treated ITER B5 strands were fatigued for three different strains 0.4%, 0.6% and 1%. Each strain was applied for three different loading cycles 1000, 10,000 and 30,000.

<table>
<thead>
<tr>
<th>Strain</th>
<th>Number of loading cycles</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.40%</td>
<td>1000 10,000 30,000</td>
</tr>
<tr>
<td>0.60%</td>
<td>1000 10,000 30,000</td>
</tr>
<tr>
<td>1%</td>
<td>1000 10,000 30,000</td>
</tr>
</tbody>
</table>

Table 5.1: Fatigue test matrix for ITER B5 strand.

Table 5.1 shows the fatigue test matrix for ITER B5 strand. As shown in Figure 5.1, B5 strands were also polished in longitudinal cross section similarly to the B4 strand. Fatigued strands strained at different strain values (0.4%, 0.6% and 1%) for the same number of loading cycles were mounted in the same polishing puck with fully heat treated unstrained strand. The pucks were polished using the polishing procedure for longitudinal cross-section described in Chapter 3. After final polishing each sample was imaged using the SEM and the filament cracks were counted using image montages.

5.2.1 ITER B5 strand crack classifications:

During our crack analysis of fatigued strands we found primarily three different kinds of cracks: 1) Void/filament cracks, where the crack touches the void. 2) Non-void/filament cracks, where the crack does not touch a void and 3) Axial or radial crack, which runs parallel to filament axis. Axial cracks are rare and appear to initiate at unreacted Nb cores rather than voids; therefore axial cracks have not been separated out based on voids; Detailed SEM images of each type of crack are shown in Figures 5.6. Apart from these three primary types of cracks there are sometimes half-cracks (cracks that do not span the entire filament diameter) also happened to
occur but the contribution of the half crack was found negligible as compared to the other three types of cracks.

Figure 5.6: Crack classification for ITER B5 strand. Images of non-void/filament cracks and axial or radial crack were taken from the sample fatigued at 0.6\% strain for 1000 cycles whereas image of void/filament cracks was taken from the sample fatigued at 0.4\% strain for 1000 cycles. The jagged appearance of theses cracks is due to the crack running along the Nb$_3$Sn grain boundaries. In axial crack image, note that the widening of the crack at the unreacted Nb core suggest that it initiates from there.
5.2.2 Crack density (cracks/mm²) calculations:

Tables 5.2 to 5.4 show the crack density results for ITER B5 strand fatigued at 0.4%, 0.6% and 1% strains for 1000, 10,000 and 30,000 cycles respectively. Zero strain represents unstrained fully heat treated strand. Crack density measurements were calculated for the full non-Cu area (that includes barrier and area between the barrier) of imaged cross-section.

Table 5.2: Crack density results for ITER B5 strand for 1000 loading cycles.

<table>
<thead>
<tr>
<th>Strain applied (1000cycles)</th>
<th>Non-void/Filament cracks</th>
<th>Void/Filament cracks</th>
<th>Half Cracks</th>
<th>Axial cracks</th>
<th>Total Cracks</th>
<th>Area(mm²)</th>
<th>Crack density (cracks/mm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>2</td>
<td>5</td>
<td>0</td>
<td>3</td>
<td>10</td>
<td>8.49</td>
<td>1.18</td>
</tr>
<tr>
<td>0.004</td>
<td>0</td>
<td>7</td>
<td>1</td>
<td>0</td>
<td>8</td>
<td>4.50</td>
<td>1.78</td>
</tr>
<tr>
<td>0.006</td>
<td>0</td>
<td>12</td>
<td>1</td>
<td>2</td>
<td>15</td>
<td>5.99</td>
<td>2.50</td>
</tr>
<tr>
<td>0.01</td>
<td>4</td>
<td>16</td>
<td>0</td>
<td>2</td>
<td>22</td>
<td>7.97</td>
<td>2.76</td>
</tr>
</tbody>
</table>

Table 5.3: Crack density results for ITER B5 strand for 10,000 loading cycles.

<table>
<thead>
<tr>
<th>Strain applied (10,000cycles)</th>
<th>Non-void/Filament cracks</th>
<th>Void / Filament cracks</th>
<th>Axial cracks</th>
<th>Total cracks</th>
<th>Area(mm²)</th>
<th>Crack density (cracks/mm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0</td>
<td>5</td>
<td>1</td>
<td>6</td>
<td>6.24</td>
<td>0.96</td>
</tr>
<tr>
<td>0.004</td>
<td>5</td>
<td>10</td>
<td>0</td>
<td>15</td>
<td>4.20</td>
<td>3.57</td>
</tr>
<tr>
<td>0.006</td>
<td>1</td>
<td>17</td>
<td>1</td>
<td>19</td>
<td>4.63</td>
<td>4.10</td>
</tr>
<tr>
<td>0.01</td>
<td>11</td>
<td>67</td>
<td>2</td>
<td>80</td>
<td>4.86</td>
<td>16.46</td>
</tr>
</tbody>
</table>
Table 5.4: Crack density results for ITER B5 strand for 30,000 loading cycles.

<table>
<thead>
<tr>
<th>Strain applied (30000 cycles)</th>
<th>Non-void/Filament cracks</th>
<th>Void/Filament cracks</th>
<th>Half cracks</th>
<th>Axial cracks</th>
<th>Total cracks</th>
<th>Area (mm²)</th>
<th>Crack density (cracks/mm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0</td>
<td>0</td>
<td>14</td>
<td>15</td>
<td>9.80</td>
<td>1.53</td>
<td></td>
</tr>
<tr>
<td>0.004</td>
<td>0</td>
<td>5</td>
<td>0</td>
<td>6</td>
<td>7.45</td>
<td>1.48</td>
<td></td>
</tr>
<tr>
<td>0.006</td>
<td>0</td>
<td>4</td>
<td>1</td>
<td>11</td>
<td>8.08</td>
<td>1.98</td>
<td></td>
</tr>
<tr>
<td>0.01</td>
<td>44</td>
<td>87</td>
<td>3</td>
<td>23</td>
<td>8.55</td>
<td>18.35</td>
<td></td>
</tr>
</tbody>
</table>

Figure 5.7: ITER B5 strand crack density results are plotted for different number of loading cycles. It is noticeable that ITER B5 strand did not show any significant filament cracking up to 1% strain for 1000 loading cycles (~ 3 cracks/mm²) but crack density increases significantly (from ~ 3 cracks/mm² to ~ 17 cracks/mm²) with increase in number of loading cycles from 1000 to 10,000 at 1% strain and did not show any further significant increase in crack density (~ 19 cracks/mm²) with increase in number of loading cycles from 10,000 to 30,000.
Crack densities from Table 5.2 are plotted in Figure 5.7 (red small dash line) as a cracks/mm² versus strain to see the evolution of filament cracking as a function of strain.

Figure 5.7 (red small dash line) indicates that crack density increases somewhat with increase in strain for 1000 cycles. From Table 5.2 and Figure 5.7 it can be concluded that ITER B5 strand did not show any significant filament cracking (crack density <= 3 cracks/mm² which is not significant as unstrained strand cross-section itself had a crack density of ~ 1 cracks/mm²) up to 1% strain (which is, however, close to breaking point ~ 1.16% strain under tensile test) for 1000 loading cycles. This led us to increase the number of cycles from 1000 to 10,000 and then up to 30,000 to examine the effect of loading cycles on crack density. Table 5.3 and Figure 5.7 (blue long dash line) show the similar data for 10,000 cycles.

From Figure 5.7 it can be seen that there was noticeable increase in filament cracking found at 0.6% strain (crack density ~ 4 cracks/mm², unstrained strand has crack density of 1 cracks/mm²) for 10,000 loading cycles. However, at 1% strain for 10,000 loading cycles a significant increase in crack density was found (crack density ~ 17 cracks/mm²) which is not the case for 1000 loading cycles. This means that the ITER B5 strand showed a significant loading cycle effect at 1% strain as the crack density increased from ~ 3 cracks/mm² for 1000 loading cycles to ~ 17 cracks/mm² for 10,000 loading cycles. These results made it even more important to extend the experiment to 30,000 cycles, crack density results for 30,000 cycles are shown in Table 5.4 and Figure 5.7 (green solid line).

Table 5.4 and Figure 5.7 (green solid line) show that for 30,000 cycles the crack density does not seem to vary significantly from 10,000 loading cycles. The crack density up to 0.6% strain at 30,000 loading cycles did not show any significant filament cracking (crack density <= 2 cracks/mm², unstrained strand had a crack density of 2 cracks/mm²). Similar to 10,000 loading cycles, significant filament cracking for 30,000 loading cycles starts at 1% strain (crack density ~ 19 cracks/mm²) and the crack density at 1% strain was found to be almost same for both 10,000 and 30,000 loading cycles (~ 17 cracks/mm² and ~ 19 cracks/mm² respectively).
5.2.3 Contribution of different kinds of cracks to the total crack density:

As we are seeking to understand the design variables related to strand architecture that might be associated with filament cracking, it is important to see the contribution of each different kind of crack in the total crack density. Figure 5.8 shows the contribution of non-void/filament cracks, void/filament cracks and axial or radial cracks to the total crack density for 1000, 10,000 and 30,000 loading cycles of the B5 strand. Non-void/filament cracks are most likely to occur at high strain (1% or above) close to the fracture limit. Non-void/filament crack counts are very low up to 0.6% strain, irrespective of the number of loading cycles. Even at 1% strain for 1000 cycles, non-void/filament cracks have a low density (0.5 cracks/mm$^2$). Non-void/filament cracks start to increase noticeably at 1% strain for 10,000 loading cycles (~ 2 cracks/mm$^2$) and 30,000 loading cycles (~ 5 cracks/mm$^2$) but still these numbers are low compared to void/filament cracks. The void/filament crack density is higher than the non-void/filament and axial crack density for most of the time which is not surprising as voids allow more stress concentration and provide less compressive protection to the filaments. Void/filament crack counts also increase with increasing strain. Although void/filament cracks have highest contribution among all three types of crack, the void/filament crack counts were not found in significant number until 1% strain for 1000 cycles (~ 2 cracks/mm$^2$). Void/filament crack counts start to increase significantly at 1% strain for 10,000 cycles (~ 13 cracks/mm$^2$) and remain in a close range for 30,000 cycles. Axial crack densities appear to have no relationship with the applied strain and loading cycle numbers, as axial crack counts remain almost same for all strains and loading cycles. Axial cracks, however, are almost always associated with an unreacted Nb core.
Figure 5.8: Contribution of different kinds of crack to the total crack density for ITER B5 fatigued strands. It is clear that contribution of void/filament cracks stands highest among all kinds of cracks at all strain values and different loading cycles. Non-void/filament crack density = Non-void/filament cracks / Area. E.g. for 1% strain for 30,000 cycles non-void/filament cracks are 44 and therefore non-void/filament crack density = 44/8.55 = 5.14 cracks/mm². Similar procedure was followed for calculating crack densities of other types of cracks as well.
5.3 ITER IT1 strand (internal tin route) fatigue test crack density results:

The approach that employed to prepare fatigue test crack density results for IT1 strands was exactly similar to B5 strand but the highest strain value for fatigue test was restricted to 0.7% as IT1 strand breaks at ~ 0.83% axial strain under tensile test. The fatigue test matrix for ITER IT1 strand is shown in Table 5.5.

Table 5.5: Fatigue test matrix for ITER IT1 strand.

<table>
<thead>
<tr>
<th>Strain</th>
<th>Number of loading cycles</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.40%</td>
<td>1000 10,000 30,000</td>
</tr>
<tr>
<td>0.60%</td>
<td>1000 10,000 30,000</td>
</tr>
<tr>
<td>0.70%</td>
<td>1000 10,000 30,000</td>
</tr>
</tbody>
</table>

5.3.1 ITER IT1 strand crack classifications:

Examples of the same 3 types of cracks seen in B5 are shown in Figure 5.9.
In IT1 strand, an unusual feature was that the axial cracks were sometimes found without any unreacted Nb core (Figure 5.9) which is not the case for either bronze route strands (B5 and B4).
5.3.2 Crack density (cracks/mm$^2$) calculations:

Tables 5.6 to 5.8 show the crack density results for ITER IT1 strands fatigued at 0.4%, 0.6% and 0.7% strain for 1000, 10,000 and 30,000 cycles.

Table 5.6: Crack density results for ITER IT1 strand for 1000 loading cycles.

<table>
<thead>
<tr>
<th>Strain Applied (1000 cycles)</th>
<th>Axial Cracks</th>
<th>Half Cracks</th>
<th>Non-void/Filament Cracks</th>
<th>Void/Filament Cracks</th>
<th>Total</th>
<th>Area (mm$^2$)</th>
<th>Crack Density (Cracks/mm$^2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>12</td>
<td>0</td>
<td>0</td>
<td>12</td>
<td>24</td>
<td>8.42</td>
<td>2.85</td>
</tr>
<tr>
<td>0.004</td>
<td>4</td>
<td>0</td>
<td>0</td>
<td>10</td>
<td>14</td>
<td>6.42</td>
<td>2.18</td>
</tr>
<tr>
<td>0.006</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>13</td>
<td>16</td>
<td>4.09</td>
<td>3.91</td>
</tr>
<tr>
<td>0.007</td>
<td>8</td>
<td>8</td>
<td>46</td>
<td>70</td>
<td>132</td>
<td>5.30</td>
<td>24.92</td>
</tr>
</tbody>
</table>

Table 5.7: Crack density results for ITER IT1 strand for 10,000 loading cycles.

<table>
<thead>
<tr>
<th>Strain applied (10,000 cycles)</th>
<th>Axial Cracks</th>
<th>Half Cracks</th>
<th>Non-void/Filament Cracks</th>
<th>Void/Filament Cracks</th>
<th>Total</th>
<th>Area (mm$^2$)</th>
<th>Crack density (Cracks/mm$^2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>10</td>
<td>1</td>
<td>0</td>
<td>11</td>
<td>22</td>
<td>6.46</td>
<td>3.41</td>
</tr>
<tr>
<td>0.004</td>
<td>2</td>
<td>0</td>
<td>1</td>
<td>7</td>
<td>10</td>
<td>2.73</td>
<td>3.66</td>
</tr>
<tr>
<td>0.006</td>
<td>12</td>
<td>2</td>
<td>6</td>
<td>37</td>
<td>57</td>
<td>5.36</td>
<td>10.63</td>
</tr>
<tr>
<td>0.007</td>
<td>12</td>
<td>1</td>
<td>34</td>
<td>72</td>
<td>119</td>
<td>6.26</td>
<td>19.01</td>
</tr>
</tbody>
</table>
Table 5.8: Crack density results for ITER IT1 strand for 30,000 loading cycles.

<table>
<thead>
<tr>
<th>Strain Applied (30,000 cycles)</th>
<th>Axial Cracks</th>
<th>Half Cracks</th>
<th>Non-void/Filament Cracks</th>
<th>Void/Filament Cracks</th>
<th>Total</th>
<th>Area (mm²)</th>
<th>Crack Density (Cracks/mm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>7</td>
<td>2</td>
<td>1</td>
<td>7</td>
<td>17</td>
<td>9.55</td>
<td>1.78</td>
</tr>
<tr>
<td>0.004</td>
<td>10</td>
<td>3</td>
<td>0</td>
<td>25</td>
<td>38</td>
<td>7.96</td>
<td>4.77</td>
</tr>
<tr>
<td>0.006</td>
<td>15</td>
<td>3</td>
<td>10</td>
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<td>84</td>
<td>8.56</td>
<td>9.81</td>
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<tr>
<td>0.007</td>
<td>16</td>
<td>8</td>
<td>42</td>
<td>120</td>
<td>186</td>
<td>8.68</td>
<td>21.43</td>
</tr>
</tbody>
</table>

Figure 5.10: ITER IT1 strand crack density results are plotted for different numbers of loading cycles. It is noticeable that ITER IT1 strand did not show any significant filament cracking up to 0.6% strain for 1000 loading cycles (~ 4 cracks/mm²) but crack density increases significantly (from ~ 4 cracks/mm² to ~ 25 cracks/mm²) with an increase in strain to 0.7% for 1000 loading cycles. Increasing the number of loading cycles from 1000 to 10,000 and 30,000 did not show any significant increase in filament cracking except there is some noticeable increase in crack density at 0.6% strain which indicates some sensitivity at 0.6% strain for loading cycles > 10,000.
In figure 5.10 (red small dash line) the crack density versus strain plotted for 1000 loading cycles to see the evolution of filament cracking with increasing strain values.

Figure 5.10 (red small dash line) and Table 5.6 indicate that ITER IT1 strand did not show any significant filament cracking up to 0.6% strain (crack density <= 4 cracks/mm$^2$ which is not significant as unstrained strand has crack density of 3 cracks/mm$^2$) for 1000 loading cycles. Once strain increases from 0.6% to 0.7% (which is close to breaking point ~ 0.83% strain under tensile test) significant filament cracking was noticed and crack density went to ~ 25 cracks/mm$^2$. This was not the case for ITER B5 strand since it did not show any significant filament cracking (crack density ~ 3 cracks/mm$^2$) at 1% strain for 1000 loading cycles. To get a more clear understanding of the loading cycle effect, the IT1 strand was fatigued at the same strain values, 0.4%, 0.6% and 0.7%, for 10,000 and 30,000 loading cycles. The crack density results for 10,000 loading cycles are shown in Table 5.7 and Figure 5.10 (blue long dash line).

Table 5.7 and Figure 5.10 (blue long dash line) show that there is a noticeable increase in crack density at 0.6% strain (~ 4 cracks/mm$^2$ to ~ 11 cracks/mm$^2$) with increasing number of loading cycles from 1000 to 10,000. At 0.7% strain for 10,000 loading cycles crack density increased significantly to ~ 19 cracks/mm$^2$. This number is close to the crack density number at 0.7% strained for 1000 loading cycles (~ 25 cracks/mm$^2$). This reverse result (more cracks at 1000 cycles compared to 10,000 cycles for the same applied strain) could have been due to the cross-sectional variations or from polishing variability but in general it can be said that crack density did not change significantly at 0.7% strain by increasing the loading cycles from 1000 to 10,000. A similar set of crack density results for 30,000 loading cycles are shown in Table 5.8 and Figure 5.10 (solid green line).

The crack density results for 30,000 loading cycles demonstrate the fact that there is no significant increase in filament cracking with increase in loading cycles from 10,000 to 30,000 at any strain value. Moreover, crack density remains almost same as of 10,000 loading cycles. In general, in IT1 strand we saw noticeable increase in filament cracking by increasing loading cycles from 1000 to 10,000 and 30,000 at 0.6% strain but other than that there is no significant loading cycle effect found unlike the ITER B5 strand.
5.3.3 Contribution of different kinds of cracks to the total crack density:

As mention in section 5.2.3 it is important to see the contribution of each type of crack to the total crack density as sometimes it reveals the effect of geometrical variables of the strand on the crack density. Figure 5.11 shows the contribution of non-void/filament cracks, void/filament cracks and axial or radial cracks to the total crack density for 1000, 10,000 and 30,000 loading cycles.

Figure 5.11: Contribution of different kinds of crack to the total crack density for ITER IT1 fatigued strands. It is clear that contribution of void/filament cracks stands highest among all kinds of cracks at all strain values and different loading cycles.
It can be seen from Figure 5.11 that non-void/filament cracks did not show any significant contribution up to 0.6% strain regardless of number of loading cycles (crack density <= 1 cracks/mm²). A significant increase in non-void/filament crack count starts at 0.7% strain for 1000 cycles (non-void/filament crack density ~ 9 cracks/mm²) and remains in the range of ± 4 cracks/mm² for 10,000 (non-void/filament crack density ~ 5 cracks/mm²) and 30,000 loading cycles (non-void/filament crack density ~ 5 cracks/mm²). Similar to the ITER B5 strand, the contribution of void/filament cracks was found to be higher than the both the non-void/filament cracks and the axial cracks. A noticeable increase in void/filament crack density was found at 0.6% strain for 10,000 loading cycles (void/filament crack density ~ 5 cracks/mm²) and remains almost same for 30,000 loading cycles (void/filament crack density ~ 7 cracks/mm²). A significant increase in void/filament crack density starts at 0.7% strain for 1000 loading cycles (void/filament crack density ~ 13 cracks/mm²) and remains almost the same for 10,000 (void/filament crack density ~ 12 cracks/mm²) and 30,000 loading cycles (~ 14 cracks/mm²). The contribution of axial cracks was not significant compared to the void/filament and non-void/filament cracks at all strains and loading cycles. At low strain values there were sometimes more axial cracks than non-void/filament cracks. In B5 strand axial cracks were always found in filaments with unreacted Nb core, and always appeared to have initiated from the unreacted Nb core. In IT1 strand that is not the case all the time, sometimes axial cracks were found in filaments without any unreacted Nb core (Figure 5.9).
5.4 ITER B4 strand (bronze route) fatigue test crack density results:

As explained in section 5.1, for the B4 strands only end pieces of the fatigued samples were initially polished and analyzed for 0.4%, 0.6% and 1% strain for 1000, 10,000 and 30,000 cycles. Some of these samples were polished by early, not-fully-optimized polishing techniques and have more polishing artifacts. As B4 strand did not show any significant filament cracking up to 1% strain regardless of higher number of loading cycles, we increased the strain to 1.1% and 1.14% but at the same time we consider that there might be a variation in crack distribution along the length of the strand so it would be better to analyze both end and center pieces of the same fatigued strand. Detailed results for this analysis are shown in section 5.1, Figure 5.3. Because of these ambiguities, crack density results for B4 strand are excluded from this thesis.

As discussed in section 5.2 ITER B5 strand experienced significant loading cycle effect at high strain (~1% strain) which makes it important to analyze the effect of loading cycles in B4 strand as both strands have same bronze route architecture. To analyze the loading cycle effect following approach was used.

ITER B4 strands fatigued at 1% strain for 1000 and 10,000 cycles were repolished (using center pieces of the fatigued strands only, same as for B5 and IT1 strands) along with unstrained fully heat treated strand as shown in Figure 5.12.
5.4.1 ITER B4 strand crack classifications:

Examples of each of the three types of cracks are shown in Figure 5.13.

Figure 5.13: Crack classification for ITER B4 strand. Images of non-void/filament cracks and void/filament crack were taken from the sample fatigued at 1.14 % strain for 1000 cycles whereas image of axial or radial crack was taken from the fully heat treated unstrained sample. Notice that the axial crack appears to have initiated from the unreacted Nb core.
5.4.2 Crack density (cracks/mm²) calculations:

Crack density results for ITER B4 strand fatigued at 1% strain for 1000 and 10,000 cycles are presented in Table 5.9 and Figure 5.14.

Table 5.9: Crack density results for the ITER B4 strand fatigued at 1% strain for 1000 and 10,000 loading cycles.

<table>
<thead>
<tr>
<th>Number of cycles</th>
<th>Half Cracks</th>
<th>Non-void/Filament cracks</th>
<th>Void/Filament cracks</th>
<th>Axial cracks</th>
<th>Total</th>
<th>Area (mm²)</th>
<th>Crack density (cracks/mm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0</td>
<td>0</td>
<td>5</td>
<td>18</td>
<td>23</td>
<td>10.05</td>
<td>2.29</td>
</tr>
<tr>
<td>1000</td>
<td>0</td>
<td>4</td>
<td>11</td>
<td>18</td>
<td>29</td>
<td>7.67</td>
<td>3.78</td>
</tr>
<tr>
<td>10,000</td>
<td>1</td>
<td>42</td>
<td>54</td>
<td>7</td>
<td>104</td>
<td>6.58</td>
<td>15.81</td>
</tr>
</tbody>
</table>

Figure 5.14: Crack density versus number of loading cycles plotted for ITER B4 strand fatigued at 1% strain for 1000 and 10,000 cycles.
Figure 5.14 shows that ITER B4 strand did not show any significant filament cracking at 1% strain for 1000 cycles (crack density ~ 4 cracks/mm$^2$ which is not significant as unstrained strand has crack density of ~ 2 cracks/mm$^2$). Filament cracking increases significantly (crack density ~ 16 cracks/mm$^2$) at 1% strain with an increase in the number of loading cycles to 10,000. Here also the void/filament crack density was the highest among all other types of cracks. This behavior is very similar to ITER B5 strand in which at 1% strain for 1000 cycles the crack density was ~ 3 cracks/mm$^2$ and increased to ~ 17 cracks/mm$^2$ at 1% strain for 10,000 loading cycles. These results indicate that both bronze route strands (B4 and B5) experienced significant loading cycle effect at 1% strain.

5.5 Comparison of fatigue behavior between ITER strands in terms of cracks/Nb$_3$Sn length (mm):

To differentiate the behavior of each ITER strand under fatigue, we compared the crack density results of ITER strands with each other. But this comparison is only valid if all three strands (B5, B4 and IT1) have same Nb$_3$Sn fraction area and filament count inside the barrier. These numbers might be similar for both bronze route (B4 and B5) strands but it is not the case when comparing bronze route and internal tin route strands. To overcome this issue we normalized the area by the total Nb$_3$Sn filament length in each cross section, so as to present the crack counts as the cracks per Nb$_3$Sn length (mm) instead of cracks per mm$^2$. Carlos Sanabria, undergraduate student at ASC, generated a normalization index (mm/mm$^2$) (Nb$_3$Sn filament length (mm) per area (mm$^2$) between the barrier) by image analysis of the SEM cross-section for each ITER strand. Multiplying this normalization index (mm/mm$^2$) with the area (mm$^2$) will generate the available Nb$_3$Sn filament length (mm) in longitudinal cross section. Total number of cracks divided by this Nb$_3$Sn length (mm) will give us cracks/mm number which we can use to compare the filament cracking in different ITER strands. The normalization index for ITER B4 and B5 strand was the same (169 mm/mm$^2$), which is not surprising as both B4 and B5 are bronze route strands and have almost same superconducting properties. The normalization index for IT1 strand was only 59 mm/mm$^2$ due to the much larger filament diameter and much smaller number of filaments.
5.5.1 Cracks/Nb$_3$Sn length (mm) comparison between ITER B5 and ITER IT1 strands:

Now using the normalization index for B5 (169 mm/mm$^2$) and IT1 (59 mm/mm$^2$) strands, Tables 5.10 to 5.15 can be recalculated for direct comparison. Results from Tables 5.10 - 5.15 are plotted as cracks/mm versus strain in Figures 5.15 and 5.16.

Table 5.10: Cracks/mm results for ITER B5 strand. All strains were applied for 1000 cycles. The Nb$_3$Sn filament length can be calculated by multiplying respective area by 169 mm/mm$^2$ (B5 normalization index).

<table>
<thead>
<tr>
<th>Strain applied (1000 cycles)</th>
<th>Void/Filament cracks</th>
<th>Half Cracks</th>
<th>Axial Cracks</th>
<th>Total</th>
<th>Area (mm$^2$)</th>
<th>Nb$_3$Sn length (mm)</th>
<th>B5 strand (cracks/mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>2</td>
<td>5</td>
<td>0</td>
<td>3</td>
<td>10</td>
<td>8.49</td>
<td>1434.99</td>
</tr>
<tr>
<td>0.004</td>
<td>0</td>
<td>7</td>
<td>1</td>
<td>0</td>
<td>8</td>
<td>4.50</td>
<td>760.69</td>
</tr>
<tr>
<td>0.006</td>
<td>0</td>
<td>12</td>
<td>1</td>
<td>2</td>
<td>15</td>
<td>5.99</td>
<td>1012.95</td>
</tr>
<tr>
<td>0.01</td>
<td>4</td>
<td>16</td>
<td>0</td>
<td>2</td>
<td>22</td>
<td>7.97</td>
<td>1346.86</td>
</tr>
</tbody>
</table>

Table 5.11: Cracks/mm results for ITER IT1 strand. All strains were applied for 1000 cycles. The Nb$_3$Sn filament length can be calculated by multiplying respective area by 59 mm/mm$^2$ (IT1 normalization index).

<table>
<thead>
<tr>
<th>Strain applied (1000 cycles)</th>
<th>Axial Cracks</th>
<th>Half Cracks</th>
<th>Void/Filament cracks</th>
<th>Total</th>
<th>Area (mm$^2$)</th>
<th>Nb$_3$Sn length (mm)</th>
<th>IT1 strand (cracks/mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>12</td>
<td>0</td>
<td>0</td>
<td>12</td>
<td>24</td>
<td>8.42</td>
<td>496.86</td>
</tr>
<tr>
<td>0.004</td>
<td>4</td>
<td>0</td>
<td>0</td>
<td>10</td>
<td>14</td>
<td>6.42</td>
<td>378.69</td>
</tr>
<tr>
<td>0.006</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>13</td>
<td>16</td>
<td>4.09</td>
<td>241.52</td>
</tr>
<tr>
<td>0.007</td>
<td>8</td>
<td>8</td>
<td>46</td>
<td>70</td>
<td>132</td>
<td>5.30</td>
<td>312.53</td>
</tr>
</tbody>
</table>
Table 5.12: Cracks/mm results for ITER B5 strand. All strains were applied for 10,000 cycles. The Nb$_3$Sn filament length can be calculated by multiplying respective area by 169 mm/mm$^2$ (B5 normalization index).

<table>
<thead>
<tr>
<th>Strain applied (10,000 cycles)</th>
<th>Non-void/Filament cracks</th>
<th>Void/Filament cracks</th>
<th>Axial cracks</th>
<th>Total</th>
<th>Area (mm$^2$)</th>
<th>Nb$_3$Sn length (mm)</th>
<th>B5 strand (cracks/mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0</td>
<td>5</td>
<td>1</td>
<td>6</td>
<td>6.24</td>
<td>1054.42</td>
<td>0.00569</td>
</tr>
<tr>
<td>0.004</td>
<td>5</td>
<td>10</td>
<td>0</td>
<td>15</td>
<td>4.20</td>
<td>709.09</td>
<td>0.02115</td>
</tr>
<tr>
<td>0.006</td>
<td>1</td>
<td>17</td>
<td>1</td>
<td>19</td>
<td>4.63</td>
<td>782.60</td>
<td>0.02428</td>
</tr>
<tr>
<td>0.01</td>
<td>11</td>
<td>67</td>
<td>2</td>
<td>80</td>
<td>4.86</td>
<td>821.57</td>
<td>0.09737</td>
</tr>
</tbody>
</table>

Table 5.13: Cracks/mm results for ITER IT1 strand. All strains were applied for 10,000 cycles. The Nb$_3$Sn filament length can be calculated by multiplying respective area by 59 mm/mm$^2$ (IT1 normalization index).

<table>
<thead>
<tr>
<th>Strain applied (10,000 cycles)</th>
<th>Axial Cracks</th>
<th>Half Cracks</th>
<th>Non-void/Filament cracks</th>
<th>Void/Filament cracks</th>
<th>Total</th>
<th>Area (mm$^2$)</th>
<th>Nb$_3$Sn length (mm)</th>
<th>IT1 strand (Cracks/mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>10</td>
<td>1</td>
<td>0</td>
<td>11</td>
<td>22</td>
<td>6.46</td>
<td>381.16</td>
<td>0.05772</td>
</tr>
<tr>
<td>0.004</td>
<td>2</td>
<td>0</td>
<td>1</td>
<td>7</td>
<td>10</td>
<td>2.73</td>
<td>161.09</td>
<td>0.06208</td>
</tr>
<tr>
<td>0.006</td>
<td>12</td>
<td>2</td>
<td>6</td>
<td>37</td>
<td>57</td>
<td>5.36</td>
<td>316.41</td>
<td>0.18014</td>
</tr>
<tr>
<td>0.007</td>
<td>12</td>
<td>1</td>
<td>34</td>
<td>72</td>
<td>119</td>
<td>6.26</td>
<td>369.30</td>
<td>0.32223</td>
</tr>
</tbody>
</table>
Table 5.14: Cracks/mm results for ITER B5 strand. All strains were applied for 30,000 cycles. The Nb₃Sn filament length can be calculated by multiplying respective area by 169 mm/mm² (B5 normalization index).

<table>
<thead>
<tr>
<th>Strain applied (30000cycles)</th>
<th>Non-void/Filament cracks</th>
<th>Void/Filament cracks</th>
<th>Half Cracks</th>
<th>Axial Cracks</th>
<th>Total</th>
<th>Area(mm²)</th>
<th>Nb₃Sn length (mm)</th>
<th>B5 strand (cracks/mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0</td>
<td>1</td>
<td>0</td>
<td>14</td>
<td>15</td>
<td>9.80</td>
<td>1656.97</td>
<td>0.00905</td>
</tr>
<tr>
<td>0.004</td>
<td>0</td>
<td>5</td>
<td>0</td>
<td>6</td>
<td>11</td>
<td>7.45</td>
<td>1259.08</td>
<td>0.00874</td>
</tr>
<tr>
<td>0.006</td>
<td>0</td>
<td>4</td>
<td>1</td>
<td>11</td>
<td>16</td>
<td>8.08</td>
<td>1365.01</td>
<td>0.01172</td>
</tr>
<tr>
<td>0.01</td>
<td>44</td>
<td>87</td>
<td>3</td>
<td>23</td>
<td>157</td>
<td>8.55</td>
<td>1445.72</td>
<td>0.10860</td>
</tr>
</tbody>
</table>

Table 5.15: Cracks/mm results for ITER IT1 strand. All strains were applied for 30,000 cycles. The Nb₃Sn filament length can be calculated by multiplying respective area by 59 mm/mm² (IT1 normalization index).

<table>
<thead>
<tr>
<th>Strain applied (30000 cycles)</th>
<th>Axial Cracks</th>
<th>Half Cracks</th>
<th>Non-void/Filament Cracks</th>
<th>Void/Filament Cracks</th>
<th>Total</th>
<th>Area(mm²)</th>
<th>Nb₃Sn length (mm)</th>
<th>IT1 strand (cracks/mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>7</td>
<td>2</td>
<td>1</td>
<td>7</td>
<td>17</td>
<td>9.55</td>
<td>563.56</td>
<td>0.03017</td>
</tr>
<tr>
<td>0.004</td>
<td>10</td>
<td>3</td>
<td>0</td>
<td>25</td>
<td>38</td>
<td>7.96</td>
<td>469.73</td>
<td>0.08090</td>
</tr>
<tr>
<td>0.006</td>
<td>15</td>
<td>3</td>
<td>10</td>
<td>56</td>
<td>84</td>
<td>8.56</td>
<td>505.10</td>
<td>0.16631</td>
</tr>
<tr>
<td>0.007</td>
<td>16</td>
<td>8</td>
<td>42</td>
<td>120</td>
<td>186</td>
<td>8.68</td>
<td>512.16</td>
<td>0.36317</td>
</tr>
</tbody>
</table>
Figure 5.15: Cracks/mm versus strain plotted for ITER B5 and IT1 strands for A) 1000 cycles B) 10,000 cycles and C) 30,000 cycles.
From Figure 5.15 A) it can be concluded that ITER IT1 strand seems to have many more cracks/mm compared to ITER B5 strand at any particular strain for 1000 loading cycles. Also note that more cracks/mm (0.0483 cracks/mm) was found in unstrained fully heat treated IT1 strand as compared to B5 strand (0.00697 cracks/mm). Similar data for 10,000 loading cycles is presented in Table 5.12 and Table 5.13. Cracks/mm versus strain results for 10,000 cycles are plotted in Figure 5.15 B).

Figure 5.15 B) illustrates the same point as of 1000 loading cycles that IT1 strand has more cracks/mm compared to B5 strand at any particular strain for 10,000 loading cycles. It should be noted that at 0.6% strain for 10,000 cycles difference between B5 cracks/mm and IT1 cracks/mm (0.15586 cracks/mm) increased (Notice the gap between the red and blue lines at 0.6% strain in Figure 5.15 B) as compared to 1000 loading cycles (0.0514 cracks/mm). In this case also the unstrained IT1 strand has many more cracks/mm (0.0577 cracks/mm) than the B5 strand (0.0056 cracks/mm). Table 5.14 and Table 5.15 present similar data for 30,000 loading cycles. In Figure 5.15 C) cracks/mm versus strain results are plotted from this data.

Figure 5.16: Cracks/mm versus strain plotted in a single plot for ITER B5 and IT1 strands for different loading cycles.
Figure 5.15 C) shows that IT1 strand has more cracks/mm as compared to B5 strand at any particular strain for 30,000 loading cycles which is the same result as of 1000 and 10,000 loading cycles.

Figure 5.16 shows the cracks/mm results for different numbers of loading cycles in the same plot, which indicates that IT1 strand showed more cracks/mm as compared to B5 strand at any strain (including fully heat treated unstrained strand) and loading cycles.

5.5.2 Cracks/Nb₃Sn length (mm) comparison between ITER B5 and ITER B4 strands:

As mentioned in the section 5.4, ITER B4 strand has only two center piece strand crack density results and they are at 1% strain for 1000 and 10,000 cycles and therefore only these two results will be compared with corresponding B5 strand results. Table 5.16 and 5.17 are generated using crack density results presented in section 5.2 and section 5.4. Both B4 and B5 strand have the same normalization index 169 mm/mm².

Table 5.16: Cracks/mm results for ITER B4 strand. Fatigued at 1% strain for 1000 and 10,000 cycles.

<table>
<thead>
<tr>
<th>Number of cycles</th>
<th>Total cracks</th>
<th>Area(mm²)</th>
<th>Nb₃Sn length (mm)</th>
<th>B4 strand (cracks/mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>23</td>
<td>10.05</td>
<td>1697.96</td>
<td>0.01355</td>
</tr>
<tr>
<td>1000</td>
<td>29</td>
<td>7.67</td>
<td>1295.88</td>
<td>0.02238</td>
</tr>
<tr>
<td>10,000</td>
<td>104</td>
<td>6.58</td>
<td>1111.98</td>
<td>0.09353</td>
</tr>
</tbody>
</table>
Table 5.17: Cracks/mm results for ITER B5 strand. Fatigued at 1% strain for 1000 and 10,000 cycles.

<table>
<thead>
<tr>
<th>Number of cycles</th>
<th>Total cracks</th>
<th>Area (mm²)</th>
<th>Nb₅Sn length (mm)</th>
<th>B5 strand (cracks/mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>10</td>
<td>8.49</td>
<td>1434.99</td>
<td>0.00697</td>
</tr>
<tr>
<td>1000</td>
<td>22</td>
<td>7.97</td>
<td>1346.86</td>
<td>0.01633</td>
</tr>
<tr>
<td>10,000</td>
<td>80</td>
<td>4.86</td>
<td>821.57</td>
<td>0.09737</td>
</tr>
</tbody>
</table>

Figure 5.17: Cracks/mm versus number of cycles at 1% strain plotted for B4 and B5 strands. Notice that both strands have a very similar behavior under fatigue.
Cracks/mm versus number of cycles results for B4 and B5 strands at 1% strain are plotted in Figure 5.17. From Figure 5.17 it can be said that both B4 and B5 strand almost behaved same under 1% strain for 1000 and 10,000 loading cycles. Here this conclusion was made based on only these two results but we hope to verify this conclusion with more results (included in future work).
CHAPTER 6

CONCLUSION AND FUTURE WORK

Conclusion:

A systematic approach for the fatigue testing of ITER strands has been developed. Evolution of filament cracking under different uniaxial strain and different loading cycles has been studied for two bronze route strands (B4 and B5 - fully reacted filaments of ~ 3 micron size) and one internal tin route strand (IT1 - fully reacted filaments of ~ 6 micron size).

At 77 K ITER B5 strand breaks at ~ 1.16% strain (varied from strand to strand between 1.15% and 1.18%) under tensile test. ITER B5 strand, as shown in Table 5.1 was fatigue tested for three different strains 0.4%, 0.6% and 1% and for three different loading cycles 1000, 10,000 and 30,000. The results show that ITER B5 strand did not show any significant filament cracking up to 0.6% strain, regardless of number of loading cycles. Even at 1% strain for 1000 loading cycles, the crack density was measured to be ~ 3 cracks/mm$^2$, which is not significant as the unstrained strand polished cross-section had a crack density of ~ 1 crack/mm$^2$. Significant filament cracking starts at 1% strain for 10,000 loading cycles (crack density ~ 17 cracks/mm$^2$) and remains almost same at 1% strain for 30,000 loading cycles (crack density ~ 19 cracks/mm$^2$). These results demonstrate the fact that ITER B5 strand experienced significant loading cycle effect at 1% strain as crack density increases with increasing number of loading cycles from 1000 to 10,000. Furthermore, ITER B5 strand did not experience any further loading cycle effect with increasing number of cycles from 10,000 to 30,000 as crack density did not increase significantly.

The ITER B4 strand breaks at ~ 1.22% strain under tensile testing at 77 K. As mentioned in Chapter 5, section 5.4, to compare the effect of loading cycles on crack density with B5 strand, two fatigued (at 1% strain for 1000 and 10,000 loading cycles) samples (center pieces) were repolished and results were compared. Not surprisingly, the results were found similar to the ITER B5 strand for the same strain and loading cycles. Like B5, B4 strand also did not show any significant filament cracking at 1% strain for 1000 cycles, the crack density was found to be ~ 4 cracks/mm$^2$, which is not significant as the unstrained strand itself has crack density of ~ 2 cracks/mm$^2$. 

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cracks/mm$^2$. Here also significant filament cracking starts at 1% strain for 10,000 loading cycles (crack density $\sim$ 16 cracks/mm$^2$). Similar to B5 strand, B4 strand also experienced significant loading cycle effect at 1% strain as the crack density increases significantly with increase in number of loading cycles from 1000 to 10,000.

Internal tin route IT1 strand requires more force as compared to both bronze route strands to apply the same strain (Table 4.1 and Figure 4.7) but it breaks at lower strain ($\sim$ 0.83% ) under tensile test at 77 K. IT1 strand did not show any significant filament cracking up to 0.6% strain for 1000 loading cycles (crack density $\sim$ 4 cracks/mm$^2$). Significant filament cracking starts at 0.7% strain for 1000 loading cycles (crack density $\sim$ 25 cracks/mm$^2$). A further increase in the number of loading cycles to 10,000 (crack density $\sim$ 19 cracks/mm$^2$) and 30,000 (crack density $\sim$ 22 cracks/mm$^2$) at 0.7% strain did not show a significant increase in crack density. The reason for this reverse trend (decrease in crack density with increase in loading cycles) in the crack density could be that the variation in polishing artifacts in the samples. Noticeable increase in filament cracking was found at 0.6% strain for 10,000 loading cycles (crack density $\sim$ 11 cracks/mm$^2$) as well as for 30,000 loading cycles (crack density $\sim$ 10 cracks/mm$^2$). Based on all these results it can be said that unlike bronze route strands, Internal tin IT1 strand did not show any significant loading cycle effect on filament cracking as crack density does not seem to increase significantly by increasing number of loading cycles from 1000 to 10,000 and 30,000 except there was some sensitivity to loading cycles found at 0.6% strain for 10,000 and 30,000 cycles.

Primarily three different kinds of crack, void/filament cracks, non-void/filament cracks, axial or radial cracks were found during SEM analysis of fatigued samples for all three strands. Regarding the contribution of each kind of crack, the void/filament cracks stand out among all kinds of crack presumably because the void results in a higher stress concentration as well as reduces the compressive protection around the filament. The non-void/filament cracks only start at high strain values (close to fracture limit) and has a much lower contribution at low strain values. The axial crack density stays almost constant regardless of strain values and loading cycles. The axial cracks were most often associated with unreacted Nb cores. However, in the IT1 strand axial cracks were also found without any unreacted Nb core (image of axial crack without unreacted Nb core in Figure 5.9) suggesting that they may be polishing artifacts produced by some other factor than the tensile fatigue.
Regarding crack distributions along the full length of the fatigued strands, more cracks were found in the center part of the fatigued sample compared to end part (Figure 5.3) and that was the reason for using only the center piece of fatigued strands for crack density calculations in this thesis. We also observed qualitatively that dispersed filament cracking was found in both B4 and B5 strands (bronze route) but a more collective cracking was found in IT1 strand (Internal tin route) (image of non-void filament cracks in Figure 5.9).

The normalized results in section 5.5 indicate that the internal tin route IT1 strand had a much greater frequency of cracks per Nb$_3$Sn filament length compared to both bronze route strands, B4 and B5 (Figure 5.15 and 5.16). Both the B4 and B5 strands seem to have a similar behavior under fatigue testing (Figure 5.17).

It is very interesting to see that all three strands are (B4, B5 and IT1) designed for the same application and have similar superconducting properties but they vary greatly in mechanical properties, which may have a significant impact on conductor operational lifetime, considering the effect of Lorentz force and thermal cool down in ITER magnet systems.

In short, in response to questions posted in a problem statement in the beginning of thesis we can now provide the following answers.

1) At what strain does fatigue start to produce observable cracking of Nb$_3$Sn filaments in strand?

Our primary experiments were restricted to only three strain values and three different loading cycles for each strand; the results showed that measurable increase in filament cracking always starts close to the fracture limit. For example, in both the bronze route strands, significant filament cracking starts at 1% strain and 10,000 loading cycles (B5 strand’s fracture limit is ~ 1.16% strain and B4 strand’s fracture limit is ~ 1.22% strain) while in the IT1 (internal tin route) strand significant filament cracking starts at 0.7% strain and 1000 loading cycles (IT1 strand’s fracture limit is ~ 0.83% strain).

2) Does repeated axial loading exacerbate the filament cracking?

The B5 strand did not show any significant loading cycle effect on the crack density at low strains (up to 0.6% strain) but it did show a significant loading cycle effect at high strain ~ 1%. As already discussed before, the B5 strand crack density increases significantly at 1% strain by increasing the number of cycles from 1000 (~ 3 cracks/mm$^2$) to 10,000 (~ 17 cracks/mm$^2$). A similar loading cycle effect was also found for the B4 strand, where at 1% strain the crack
density increases significantly by increasing number of loading cycles from 1000 (~ 4 cracks/mm$^2$) to 10,000 (~ 16 cracks/mm$^2$). In contrast to both bronze route strands, the internal tin route IT1 strand did not show any significant loading cycle effect on crack density. The crack density for the IT1 strand remains almost constant regardless of different loading cycles.

3) Does the strand architecture play any role in fatigue related cracking?

By normalizing the longitudinal cross section area being analyzed with available Nb$_3$Sn length in that cross section area, fatigue performance of different strand architecture strands can be compared in terms of cracks per Nb$_3$Sn length (mm). This comparison shows that the internal tin route IT1 strand has many more cracks per length of filament than the bronze route B5 strand. This implies that the degradation of $J_c$ for the internal tin route IT1 strand should be much more catastrophic compared to the bronze route strands, B4 and B5. Both bronze route strands seem to have very similar performance under fatigue testing (based on two results).

**Future work:**

Here, two complete sets of fatigue test results at 77 K are presented, one for ITER B5 (bronze route) strand and another for ITER IT1 (Internal tin route) strand. Also few ITER B4 strand results were presented to compare the fatigue test performance with B5 strand but complete sets of results need to be prepared. This work can further be extended to fatigue testing of other internal tin route strands. The purpose of this extended work will be to prepare complete sets of results for two bronze route strands and two internal tin route strands. These results will provide us greater understanding of the comparative fatigue performance of each strand (indirectly the difference in mechanical properties and superconducting performance) and will be useful information for a conductor design point of view.
REFERENCES

[1] Chapter 4, Metallurgy of continuous filamentary A15 superconductors by Masaki Suenaga


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