ABSTRACT

ROGERS, SAMUEL. Effects of Tensile Fatigue on Critical Current and n-value of \((\text{RE})\text{Ba}_2\text{Cu}_3\text{O}_{7-x}\) Superconductors (Under the direction of Prof. Justin Schwartz)

Superconductivity, first discovered in 1911, has been one of the most promising yet misunderstood phenomena of the 20th century. There are still competing theories on why superconductors, especially high temperature superconductors, pass current with no electrical losses. This lack of knowledge translates into a lack of understanding about what causes damage in these materials. Slight perturbations in grain orientation, stresses, or stoichiometry can cause a loss of superconductivity and render the material useless for its intended application. During manufacturing and use, the superconducting material experiences many stresses that can lead to electromechanical failure.

High temperature superconductors (HTS) have been touted as a game changer for energy and magnet applications. Their higher critical temperatures and increased current carrying capability show great promise. While research is being done to manufacture new devices that employ HTS, they are extremely costly and it is almost prohibitively expensive to construct a full scale device simply for experimental purposes.

The material properties of HTS must be fully understood in order to manufacture devices on a large scale. These devices will be in service for many years, and performance of these devices must be retained throughout their operation. It is known that materials behave differently in monotonic loading than in fatigue, and the same applies for HTS composite conductors.
To understand how (RE)Ba$_2$Cu$_3$O$_{7-x}$ (REBCO), a type of HTS, survives fatigue loading; tensile fatigue loading up to $10^5$ cycles and 0.50 % strain has been performed. Following this loading, SEM was used to investigate the damage on a microstructural level and measure crack growth. Critical current and the transition index ($n$-value) were measured before and after each fatigue loading, in order to understand a what point degradation occurs in REBCO conductors.
Effects of Tensile Fatigue on Critical Current and n-value of (RE)Ba$_2$Cu$_3$O$_{7-x}$ Superconductors

by
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A dissertation submitted to the Graduate Faculty of North Carolina State University in partial fulfillment of the requirements for the degree of Doctor of Philosophy Materials Science and Engineering Raleigh, North Carolina 2016

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BIOGRAPHY

Sam Rogers was born in San Diego, California in 1989. After finishing high school he attended Occidental College in Los Angeles, California for his bachelor’s degree in chemistry. He was admitted in 2012 to the PhD program at North Carolina State University to study materials science and engineering. After a semester as a teaching assistant, he joined Professor Justin Schwartz’s group to further the understanding of high temperature superconductors and their electromechanical properties. In 2015 he co-founded a university spinout focused on the large scale production of gold nanorods and was awarded three startup awards through the Lulu e-games business plan competition. In 2016 he received a Technology Entrepreneurship and Commercialization graduate certificate, focused on bringing technology innovations to market. Sam is currently working towards his MBA at North Carolina State University. In his free time, Sam enjoys golfing, camping and restoring older vehicles with modern technologies.
ACKNOWLEDGMENTS

I would like to thank my thesis advisor, Dr. Justin Schwartz, for his continued support and critique of my work that allowed me to complete my studies. His method of advising allowed me to pursue opportunities that shaped who I am as a scientist today. I am also indebted to my committee members, Dr. Hunte, Dr. Zikry, and Dr. Scattergood, for their advice and knowledge that made this work possible. Their classes prepared me for this work, and gave me the underlying knowledge needed to hit the ground running in lab.

I would like to thank my group members, both past and present, for their fruitful discussions on all topics; you provided the motivation on days when the tasks in lab seemed impossible. Last but certainly not least, I would like to thank all the MSE staff members who assisted me during my time here at NC State.
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Chapter 1

Introduction and Overview

Motivation

Ceramic materials with favorable electronic properties are extremely important in next generation technologies. Semiconducting and superconducting ceramic materials have changed the world, and continued development of these materials will likely lead to more game-changing discoveries. In order to apply these functional ceramics to applications, more must be understood about their mechanical properties. Since ceramics have a low toughness compared to high strength metals, it is common to produce a composite material that has a functional ceramic layer with a high strength metallic substrate. Studies have been performed on the stress/strain behavior of these composites over a single cycle, however detailed fatigue understanding is limited.

There is an industry need for high current density conductors that only superconductors can achieve. Low temperature superconductors (LTS) have changed the way we practice medicine, and perform scientific research. A class of materials, high temperature superconductors, allows for even greater current density and operation in high magnetic fields. This opens the door for exciting energy opportunities that would transform the landscape of society as we know it today. However, these materials are expensive (millions of dollars for some applications) and little is known about their mechanical properties. In order to receive support for the applications, it must be shown that the materials used will
withstand the stresses over the lifetime of the device. This work aims to understand at what point high temperature superconductors (HTS) materials suffer fatigue failure, and why. By answering these questions, HTS will have one less barrier to entering the market.

**Basics of superconductivity**

Normal electrically conductive materials such as copper require a voltage, or potential, to pass electric current. This occurs because the current is carried by “conduction electrons” which are free to move throughout the material. Thermal vibrations of atoms, impurities, and defects can scatter these moving electrons and give rise to resistance. This heat, or resistance, is what limits the ability of normal conductors to pass large amounts of current. When the temperature of materials is lowered, the thermal vibration is reduced, and this accounts for why electrical resistance decreases with decreasing temperature. Some applications take advantage of this resistance, such as electric heaters, however for magnet and energy applications this resistance is an inefficiency that limits the usefulness of ordinary conductive materials. Figure 1 plots resistance vs. temperature for both normal metals and superconductors.
In 1911 superconductivity was discovered by H. Kamerlingh Onnes in the Netherlands; he found that mercury had zero electrical resistance when cooled below 4 K [1]. Since this discovery, over 6,000 compounds have been found to be superconductors. Only a handful of these are technologically viable, and can be seen in figure 2. [2]
Superconductors are elements, alloys, or compounds that have zero electrical resistance when cooled below their critical temperatures, $T_c$. The physics behind superconductivity was misunderstood for 50 years after their discovery, and this stalled their use in applications.\cite{2} BCS theory, by Bardeen, Cooper, and Schrieffer, describes in detail the phenomenon of superconductivity. When a material is in its superconductive state, pairs of electrons (cooper pairs) are formed at the lowest energy level. The strong interaction between these electrons does not allow scattering and results in zero resistivity. If a temperature above $T_c$ is reached, the cooper pairs break apart and superconductivity is lost. The exact interaction mechanism for superconductivity in HTS is not fully understood yet. Figure 3 shows when one electron attracts the nearest positively charges ions, there is an attractive force that causes the second electron to be pulled in by the localized positive charges.\cite{3} This attractive force between
electrons is greater than the local repulsion and explains how electron pairs form in LTS. There are many cooper pairs throughout the lattice in a superconductive material and they all have a wave function of the same amplitude and phase which overlap and result in a wave function. The coherence length, $\xi$, is a measure of the spatial variation of the density of superconducting electrons. Thus, a small coherence length means that the density is rapidly changing.

Figure 3: Formation of Cooper pairs in the lattice of a material [3]

$T_c$ is one value that affects superconductivity, the other parameter is the critical magnetic field, $H_c$. If the electric field in a superconductor becomes non zero, then the current flow is a finite value. Maxwell’s equation (1) shows that the magnetic field cannot vary with time when $E=0$. If there is a magnetic field is presented to a superconducting material, the magnetic flux does not penetrate the material and is completely expelled from the superconductor. This is called the Meissner effect and is what allows superconductors to levitate over a magnet. [4]

$$\nabla \times E = -\frac{\partial B}{\partial t} \quad (1)$$
Based on the Meissner effect, superconductors can be grouped as two types, Type I and type II. Figure below shows the magnetization vs field data for a type II superconductor.

If there is a single, sharp, transition to the normal state, then the superconductor is said to be type I. These materials are typically elemental superconductors. For type II
superconductors, there are two critical field values that are of interest. The lower critical field \( (H_{c1}) \), below which the field is completely expelled, Meissner effect, from the material as in a type I superconductor. Below the upper critical field \( (H_{c2}) \), the magnetic field partially penetrates the material. Above values of \( H_{c2} \) the type II superconductor transitions to the normal state. If the magnetic field is between \( H_{c1} \) and \( H_{c2} \), this is called the mixed state. In the mixed state, superconductivity is preserved. Because \( H_{c2} \) values are usually much larger than \( H_{c1} \) values, type II superconductors are useful for applications involving high magnetic fields. [5]

A second explanation for superconductivity is given by Ginzburg and Landau (GL) using the order parameter below where the shield current is the wave function \( \psi \), \( \xi \) is the GL coherence length (spatial change in order parameter).

\[
\psi = |\psi_0| \exp \left( -\frac{x}{\xi} \right)
\]

Abrikosov solved the GL equations with a two-dimensional periodic approach and through this work suggested that there was a periodic array of flux lines, the flux-line lattice (FLL). This predicted the occurrence of vortices in superconducting materials which is shown in the figure below. When an external magnetic field is applied to the superconducting material, vortices invade the superconductor and superconductivity is suppressed in the \( \xi \) radius. Magnetic field energy is reduced in the \( \lambda \) radius. [3]
Shielding current flows in the $\lambda$ radius, and each fluxon carries one magnetic flux quantum, $\Phi_0 = 2.07 \times 10^{-15}$ Tm$^2$. This is due to the supercurrent circulating in the vortex. Due to the passage of current, $J$, the Lorentz force ($f_l = J \times e_2 \Phi_0$) causes the vortices to move. This motion of vortices is limited by the friction force. Efforts to reduce the motion (pinning) of said vortices have been under development in past years to improve in field $J_c$. [6]

There are two main groups of superconductors, high temperature (HTS), and low temperature (LTS). Materials that are superconductive above 77 K, boiling point of liquid nitrogen, are classified as HTS, and those that require cooling below 77 K to become superconductive are classified as LTS. LTS materials generally require liquid helium or cryocoolers to operate, while HTS can operate in liquid nitrogen. LTS superconductors include NbTi$_2$ and Nb$_3$Sn, which are used extensively in MRI systems and other high field applications. These materials have been well studied and have a stake in the current market. [7][8]
Properties of Superconductors

Simply because a superconductor is cooled, does not mean it will remain superconductive. A transition from the superconducting to the normal state can occur in three different ways: by raising the temperature above the critical temperature ($T_c$), by raising the applied magnetic field above the upper critical field value ($H_{c2}$), and finally by raising the current density above the critical current value ($J_c$). A combination of these changes can trigger a transition to the normal state at a value less than the “critical values” as seen in figure 7. If the material stays within the bounds of the critical surface, it will remain superconducting.

![Superconductor critical surface plot](image)

Figure 7: Superconductor critical surface plot

These parameters apply for a specific type of material, and can be changed due to processing, purity, stoichiometry, or mechanical damage. It is important to note that a superconductor is only as good as its worst area. That is to say, if a superconductor was locally damaged and
could only carry 50% of its rated current, the entire application would then be limited to 50% of its rated current because of a bottleneck restriction. It is for this reason that any damage, no matter how localized, must be mitigated.

**High Temperature Superconductors**

This work will be focused on high temperature superconductors, mainly a specific type of HTS with the chemical formula \( \text{REBa}_2\text{Cu}_3\text{O}_{7-x} \) (REBCO) where RE stands for a rare earth element. REBCO was discovered in 1987 and had a \( T_c \) of 93 K, at the time this discovery lead to a renewed interest in superconducting materials and many new HTS compounds were discovered in the following years[9]. REBCO, like all cuprate superconductors, are type II. The properties of REBCO are sensitive to the oxygen content, and only materials with \( 0 \leq x \leq 0.65 \) are superconducting below \( T_c \). REBCO is also extremely sensitive to microstructural changes, grain misalignments of greater than 2 degrees can significantly affect the critical current density, \( j_c \). Because of this, the manufacturing of REBCO is extremely expensive and difficult. YBCO is a defect perovskite structure with multiple layers as shown in figure 8, where the CuO\(_2\) planes are the basic structural element.
In order to successfully manufacture REBCO conductors on a large length scale while retaining high $J_c$, manufacturers must retain a bi-axial growth template for the REBCO layer. This is achieved through the deposition of multiple buffer layers that both allow for texture and also act as diffusion barriers so the REBCO is not poisoned by the high strength substrate.[11] The architecture of a SuperPower REBCO conductor is shown in figure 9, note the intricate buffer stack made up of various oxides.
Manufacturing Process

It is important to understand the architecture and complexity of REBCO conductors, as their manufacturing process has a direct correlation to their mechanical strength. The first step is selecting a substrate, which can be Hastelloy C-276, Stainless Steel, Ni-W, or another high strength alloy. This substrate is electropolished before additional layers can be deposited. An alumina and yttria diffusion layer is deposited on the polished substrate, followed by IBAD MgO to provide a texture template for growing the REBCO layer. The final buffer stack layer is SrTiO$_3$ (STO) to provide a lattice match between the buffer stack and the superconductor. It is clear that a lot of engineering is needed in order to preserve both the correct texture and stoichiometry of the REBCO layer. The REBCO layer is deposited using Metal Organic Chemical Vapor Deposition (MOCVD). This layer can contain BaZrO$_3$ (BZO) nano-columns to provide increased flux pinning for operation at elevated magnetic fields.[12] The REBCO layer is coated with a silver cap layer that provides good electrical
transfer and oxygen diffusion during anneal. A final copper or brass layer is then electroplated or soldered onto the conductor for increased stabilization during operation and quench conditions.

Figure 10: Showing the advantages that flux-pinning has on transport in a magnetic field[13].

Long length manufacturing has been an issue for conductor companies due to instability in the process. In years past it was difficult to manufacture more than 100 m of continuous conductor. Current operations can produce ~500 m of usable conductor that meets or exceeds specifications, however this value is less than the amount of conductor needed for larger applications. In order to join these pieces of tape, lap joints are created and soldered together in order to create a low loss, but resistive, splice. Researchers are currently investigating methods to make superconducting joints and splices, while manufacturers are attempting to produce km long runs of continuous conductor.
Applications of Superconductors

LTS has been extensively used in MRI magnets to generate fields of 3 T at 4.2 K, and due to the ability to recycle the liquid helium with a cryocooler and the low cost of LTS, MRI manufacturers see no need to use HTS[14]. However, there are some applications where the properties of LTS make them unusable. These applications operate at elevated temperatures (>50 K), extremely high current densities, high magnetic fields, and see large stresses during operation. Such applications include but are not limited to, high field magnets, accelerator magnets, transformers, fault current limiters (FCLs), superconducting magnetic energy storage (SMES), Superconducting Quantum Interference Devices (SQUID), and cellular communication filters. [14][15] [16][17][18] One common theme of these applications is their extremely high cost, usually on the order of millions of dollars. Therefore, these devices must operate with a long service life in order to balance the high price tag. One step to achieving reliable operation is a better understanding of the mechanical properties of the materials used in the devices.
Stresses in Superconductor Applications

These superconducting materials are subject to extremely high stresses, there are three main types of stresses that a superconducting material will be exposed to:

Fabrication Stresses

A large majority of applications require that the superconducting material be wound in coil form, and during the winding of the coil the superconducting material is subject to a tensile and bending stress. The tensile stress is from the coil winding process that requires a certain line tension be maintained during fabrication, and the bending stress is due to the winding
around a coil former. As the coil diameter becomes smaller, the bending stresses will increase substantially.

**Thermal Cooldown Stress**

Once the coil has been manufactured, at room temperature, the coil must be cooled to its operating temperature. In the instance of HTS, the conductor is a composite metal/ceramic where the ceramic is the superconducting material and the metal is a high strength alloy that provides strength. These materials have different coefficients of thermal expansion, with the metal being a higher value, thus the ceramic HTS is subject to a compressive strain during cooling. Figure 12 shows the residual stresses in the different layers of the conductor after a cool down from deposition temperature (1073 K) to 77 K.

Figure 12: Residual stress in the composite YBCO conductor after cooldown from the manufacturing temperature.[19]
Operating Stresses

After manufacturing and cooldown, the coil is then energized which causes a large Lorentz force to act on the material. This Lorentz force acts as a hoop stress on the conductor, and tries to push the magnet apart in the radial direction. At a local level, this hoop stress can be modeled as a tensile stress on the individual turns of conductor.

Lifetime Operation (Fatigue)

Taking into account these three stresses, it is important to now consider how the application is used over its entire lifetime. Magnets typically do not remain at cryogenic temperatures for their entire lifetime, service procedures and power outages can cause the magnet to warm up and require another cooldown. This may happen on the order of 10-100 times during the lifetime of a magnet, however the operating stresses will fluctuate much more often. Magnets used for research purposes are ramped and de-ramped for each individual experiment, and researchers at the NHMFL postulate that their most recent research magnet will be subject to 50,000 charge/discharge cycles over its lifespan[20]. Magnets that are used with alternating current (AC), will see a variation in the hoop stress due to the changing magnetic field. And superconducting motors will see centrifugal stresses when starting and stopping, and when the load on the motor is varied.

Introduction to Fatigue Failure

It is clear that superconducting applications will see alternating stresses, which can cause materials to fail at a stress much lower than needed for fracture during a single cycle[21][22].
This type of failure is called fatigue failure, and it will be the main topic of the thesis. Fatigue was first observed when metals that were subjected to repeated stress failed at a much lower stress required for failure on a single load. Fatigue is caused from the plastic strain, cyclic stress and tensile stress all acting in conjunction. Researchers have determined the number of cycles needed to fracture many common structural materials, this value is commonly known as the fatigue life[21].

Fatigue can be performed in multiple methods, with a reversed cycle, repeated cycle, or a random cycle. The most commonly used types of loading are reversed and repeated cycles because they give meaningful data that can be used for materials selection. Fatigue experiments generally requires certain parameters in order to understand the loading conditions, the most common parameters are explained here. The stress range, $\sigma_r$, is the difference from the minimum and maximum stress.

$$\sigma_r = \sigma_{max} - \sigma_{min}$$

The mean stress, $\sigma_m$, is the average of the maximum and the minimum stress.

$$\sigma_m = \frac{\sigma_{max}+\sigma_{min}}{2}$$

The alternating stress is equal to

$$\sigma_a = \frac{\sigma_{max}-\sigma_{min}}{2} = \frac{\sigma_r}{2}$$

The stress ratio, $R$, is an important factor used when describing fatigue data, and is the ratio of minimum stress to maximum stress

$$R = \frac{\sigma_{min}}{\sigma_{max}}$$
This means for a fatigue experiment that starts at zero stress, and loads to a certain stress value, then back to zero, the R value is zero. For a fully reversed cycle, the R value is -1. Work for this thesis will be performed using strain controlled fatigue, and the value for strain replaces the values for stress, for example $\sigma_{\text{min}}$ is would be replaced by $\varepsilon_{\text{min}}$, etc. The common terms then take on the word strain, so mean stress would be called mean strain.

![Fatigue loading graph](image)

Figure 13: Fatigue loading graph that shows the parameters of interest [23].

**What Causes Fatigue Failure**

Fatigue failure is broken down into four distinct areas: crack nucleation, stage 1 crack growth, stage 2 crack growth, and ultimate ductile failure. Crack nucleation is initiated by the movement of dislocations which eventually form persistent slip bands and become the nucleus of short cracks. Stage 1 and 2 crack growth proceed until the crack becomes a critical length, where the material ultimately mechanically fails.
Material defects such as cracks, scratches and notches are called stress concentrators, because they locally cause stress to concentrate around the defect. The presence of these defects can significantly reduce its fatigue strength. Other common sources of stress concentration can come from microstructural level defects such as inclusions and pores. The size of the specimen also has an effect on the fatigue life. Increasing the material size also increases the surface area and volume and gives a greater probability of internal and surface defects. This is why laboratory size specimens usually have a higher fatigue life then their real world counterparts. It is a factor to consider when we are using 5-10 cm long single tapes of HTS, and this will ultimately be constructed into a magnet built with kilometers of HTS conductor.
Surface condition plays a large role in the fatigue life, since most fatigue starts on the surface of the material. The roughness of the surface is very important, and the direction of the scratches on the surface play a role in fatigue life as well. If the polishing scratches on a specimen are oriented parallel to the applied load, the specimen will have a higher fatigue life than a specimen that has the scratches oriented perpendicular to the applied load. This is important to note for the HTS tapes since many of them have different surface conditions and cap layers, ranging from copper laminate to silver plating.

**Onset of Failure**

As with any loading regimen, it is important to clearly define when failure has been reached. The common stop criteria for fatigue experiments is when the material has mechanically failed and broken. This process is repeated multiple times for different stresses, and gives a $\sigma$-N curve. Some applications are complex loading scenarios and an average stress value cannot be easily determined, other cases the load may vary in a non-sinusoidal fashion. Some materials such as steels and high strength alloys have a fatigue limit, where below a certain stress value, the material will last indefinitely.

For the purpose of measuring HTS materials, it is imperative to set a failure threshold that is electrical, not mechanical. Once the conductor has mechanically failed, it will be well past the point of being electrically useful. Therefore, for this study, it has been set that a 10 % reduction in critical current will be “failure”. Magnets are usually operated with a significant
margin of error, that is the current in the superconductor during operation is 30-70 % of the rated capacity, in order to prevent quenching and operate in as stable a manner as possible.

![V vs. I plot for a superconductor during a transport measurement][25]

**Figure 15**: V vs. I plot for a superconductor during a transport measurement[25]

**Temperature Effects**

Another very important consideration is temperature effects. Since the HTS will be operated at 77 K and lower, it must be taken into consideration what is happening to the materials at these low temperatures.

For the purpose of this work, all fatigue loading will be performed at room temperature due to the complexity of a low temperature fatigue experiment apparatus. If an attempt was made to perform fatigue at 77 K, the frequency of the loading would have to be increased significantly in order to reduce the measurement time. With long measurement times, it
would be all but impossible to keep the sample submerged in the cryogen without boiling off, and the load cell would eventually freeze up.

Performing fatigue at room temperature will be inherently different than low temperature due to the material properties. At lower temperatures, materials’ fatigue strength increases with the exception of steel and other materials that undergo a ductile to brittle transition. The following experiments utilize Hastelloy and stainless steel, both of which do not undergo a ductile to brittle transition.

Another factor specific to HTS conductors at low temperatures, is the compressive stresses brought on by the difference in thermal expansion. As discussed above, the cooling of HTS conductors will place the superconducting layer in a compressive stress state. Since tensile fatigue loading is being utilized, the compressive stress upon cooldown would be beneficial and further increase the fatigue life of the conductor.

Ceramic Mechanical Behavior

It is necessary to give some background on how ceramic materials respond to an applied load in order to fully understand what causes them to fail. Compared to conventional metallic materials, ceramics exhibit poor toughness due to their inability to undergo plastic deformation. When a metallic material is strained beyond its yield point, the material takes advantage of the available slip systems and can plastically deform without ultimate failure. Ceramics are primarily composed of ionic and covalent bonds, these bonds will tend to
fracture before any plastic deformation can take place because slip systems are unavailable. In addition, because ceramic materials tend to be porous, the imperfections will act as stress concentrators and cause the material to have a decreased tensile strength.[26][27]

Figure 16: Stress/strain plots for various materials[28]

**Fatigue of Composite Materials**

Fatigue in composite materials is a complicated process and is influenced by many variables such as the geometric and material types at the region of damage. Interfacial effects also play an important role in how fatigue damage effects composites. Fatigue damage in composite materials is randomly multiplied throughout the material and not limited to a single defect or crack. The damage that occurs depends heavily on the layering, material properties, and loading type. Main modes of fatigue failure in composites are debonding,
fiber breakage, and cracking of the matrix. There are three main stages of crack manifestation in composite materials, matrix cracking, delamination and interfacial debonding, followed by fiber breakage. Matrix cracking has been attributed 80% of the damage, but approximately 20% of the fatigue life. [29][30]

![Diagram](image)

Figure 17: Fatigue damage as a function of fatigue life for composite materials[2].

This is an important time to recall that we must distinguish between mechanical and electrical damage. Although the composite material may have only been subject to 20% of the mechanical fatigue life, the ceramic layer may have already been damaged enough that electrical transport has been reduced appreciably.

**Electromechanical Properties of REBCO**

Bending strain was investigated by Sutoh et al. on IBAD YBCO tapes.[31] It was found that $I_c$ did not reduce until 0.4% tensile strain and 0.5% compressive strain. Cracking was seen
on the YBCO film at strain values higher than tensile strain of 0.5%. It was reported that the crack generation was the primary cause of the reduction in $I_c$. Hazelton et al. reported on commercial SuperPower tapes response to bending strains, and found that the conductor retained more than 95% of the as-received $I_c$ when bent on a 11 mm diameter mandrel. [12] Single cycle tensile strain effects on REBCO conductors have been widely studied in recent years. Barth et al. reported on the electromechanical properties of various manufacturers’ REBCO conductors.[32] The irreversible strain limit, the strain at which the conductor will become permanently damaged from a single loading, varied from 0.45% to 0.7% between manufacturers.

Figure 18: Transport vs. strain for commercial REBCO conductors[32]

This data is particularly useful if you are concerned about exceeding a certain strain value for a single loading, but it does not tell you how the material will respond to multiple loadings.
Figure 19 represents a single cycle strain-$I_c$ measurement on YBCO coated conductor performed by Mbaruku et al. and shows the strain does not have an effect on critical current till strain values of 0.75%. Note how at 0.3% the critical current actually increases, this is due to the thermal contraction strain that is placed on the superconducting layer during cooldown. At this strain value the superconducting layer is near a zero net strain and will carry more current. [2]

![Figure 19: Transport vs strain for a YBCO conductor, note the sharp drop in transport at 0.8% strain][2]

Work by Cheggour et al. was done on YBCO coated conductor with Ni-alloy and Ni substrates.[33][34] Irreversible strain values of 0.38% were reported, and it was reported that the Ni-alloy substrate had improved mechanical properties. Further work by Cheggour et al. studied the transverse compressive strain effects in REBCO conductors. It was shown that
transverse compressive stress has a significant effect on $J_c$, however by using a Ni-5at%W alloy, the conductor had improved resistance to damage from transverse loadings.

Fatigue was studied by Mbaruku et al. in detail for 4 mm wide SuperPower conductors in 2006.[2] It was shown that at strain values much lower than the irreversibility limit, damage was possible when conductors were subject to multiple loadings. This work was paramount to understanding how important fatigue considerations were when designing superconducting applications. Figure 20 summarizes the results seen at different strain values for a fatigue ratio of 0.5.

![Figure 20](image)

**Figure 20:** Critical current as a function of number of cycles for a YBCO conductor at varying strain values with a fatigue ratio of 0.5.[2]

Figure 21 shows fatigue life for YBCO conductors at varying strain values with a fatigue ratio of 0.2.
What is noticeable is that fatigue on a sample at 0.4% strain with R=0.2 doesn’t degrade until 100,000 cycles, however at the same strain and R=0.5, electromechanical failure begins at 1000 cycles. Failure in composite materials is highly variable and dependent on many factors. Slight sample differences can have a profound effect at high cycle fatigue, and must be taken into consideration. It was concluded that cracking formed on the ceramic layer on all areas of the specimen perpendicular to the applied load. The cracking was the root cause of the I_c degradation in the fatigued samples, with cracks starting at the edge of the tapes and propagating inward. [35][36][37]
Objectives

The introduction of this thesis was intended to introduce the concept of superconductivity and why it is a valuable technology that is of interest to companies. It also highlighted what challenges must be overcome in order to use HTS in applications. Moreover, an introduction to fatigue failure in composite materials was presented, and the importance of understanding fatigue was described in how it relates to HTS.

The work performed by Mbaruku et al. was instrumental in highlighting the importance of fatigue in REBCO. However, since that work was completed the REBCO manufacturing processes and materials have significantly changed. Many manufacturers now offer conductors with varying substrates, in multiple widths, and with different protective layers. These changes have had a profound impact on the electromechanical properties as shown by Barth et al. The work performed in chapters two and three will focus on two manufacturers’ REBCO conductor that has different substrates and a different protective layer. The fourth chapter will look at an alternative packaging of multiple REBCO conductors for use in an energy storage application. Finally, chapter five gives a summary and conclusions that can be drawn from this work as well as the recommended future work.
References:


Chapter 2

Effects of Room-Temperature Tensile Fatigue on Critical Current and n-value of IBAD-MOCVD YBa$_2$Cu$_3$O$_{7-x}$/Hastelloy Coated Conductor

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Abstract
REBa$_2$Cu$_3$O$_{7-x}$ (REBCO) coated conductors potentially enable a multitude of superconducting applications, over a wide range of operating temperatures and magnetic fields, including high-field magnets, energy storage devices, motors, generators, and power transmission systems [1]. Many of these are AC applications and thus the fatigue properties may be limiting [2]. Previous electromechanical studies have determined the performance of REBCO conductors under single cycle loads [3], but an understanding of the fatigue properties is lacking. Here the fatigue behavior of commercial Ion Beam Assisted Deposition-Metal Organic Chemical Vapor Deposition (IBAD-MOCVD) REBCO conductors on Hastelloy substrates is reported for axial tensile strains up to 0.5% and up to 100,000 cycles. Failure mechanisms are investigated via microstructural studies. Results show that REBCO conductors retain electrical performance for 10,000 cycles at $\varepsilon$=0.35% and $\varepsilon$=0.45% strain, and $\varepsilon$=0.5% for 100 cycles. The main cause of fatigue degradation in REBCO conductors is crack propagation that initiates at the slitting defects that result from the manufacturing process.

Introduction
Significant progress has been made towards the commercial viability of high temperature superconductors for devices. A number of manufacturers now offer REBa$_2$Cu$_3$O$_{7-x}$ (REBCO) conductor in varying dimensions and electrical and mechanical behavior [4]. REBCO conductors have high critical current density ($J_c$) in the presence of a high background magnetic field and favorable mechanical properties due to the REBCO layer being near the neutral axis and the mechanical properties of the Ni-alloy substrate that comprises a large percentage of the cross-sectional area. As a result, REBCO conductors are of significant interest for a range of superconducting magnet applications, including colliders, nuclear magnetic resonance, motors, generators, and energy storage [5, 6]. Conductors in magnet applications are subjected to a variety of mechanical and thermal loads, including those due to the manufacturing process, differential thermal contraction, Lorentz forces during operation, and in some applications fatigue [2, 6-14]. Thus, fully understanding the electromechanical properties of REBCO conductors is key to successful systems development and operation.

Many studies of the electromechanical behavior of superconductors have been performed on NbTi and Nb$_3$Sn wires and cables, and there have been a few studies on the emerging materials, Bi$_2$Sr$_2$CaCu$_2$O$_{8+x}/$AgX, MgB$_2$ and REBCO [9, 15]. The emerging conductors are still evolving, however, and it is likely that the electromechanical behavior will evolve as well.

Previous electromechanical studies focused mostly on the effects of single cycle of tensile strain. Mbaruku et al. [16] performed a Weibull analysis of REBCO conductor on NiW substrate and determined that the substrate was responsible for a universal strain behavior.
below yield, and dominated the mechanical properties of the composite REBCO conductor. Ilin et al. [17] investigated REBCO/Hastelloy conductors under single-cycle tensile loads and showed 100% reversible behavior for tensile strain ($\varepsilon$) up to $\sim 0.63\%$ at 77 K. Barth et al. [3] showed single-cycle tensile electromechanical results from a wide range of conductor manufacturers and found that the irreversibility limits range from $\varepsilon = 0.45\% - 0.7\%$. Many variables distinguish REBCO conductors including deposition method, e.g. ion beam assisted deposition (IBAD) and rolling-assisted biaxially textured substrates (RABiTS), substrate (NiW, Hastelloy, and stainless steel), and cap layer (electroplated Cu, Cu lamination, brass lamination, etc.) and each variable affects the electromechanical behavior. During magnet manufacturing and operation, strain values above the irreversibility limit are avoided due to the risk of conductor damage.

Cheggour et al. showed that the REBCO conductor manufacturing process influences the electromechanical behavior. REBCO conductors are typically manufactured as a 12 mm wide conductor that is slit to the desired width before the cap layer is added. [18] They showed that cracks due to the slitting process do not grow after being subjected to a transverse (edge to edge) loading of 150 MPa for 20,000 cycles. They recognized, however, that other forms of loading and higher stress could degrade the conductor critical current ($I_c$).

High-cycle fatigue was studied by Mbaruku et al. using 4 mm wide Superpower IBAD/MOCVD Hastelloy $\text{Yb}_2\text{Cu}_3\text{O}_{7-x}$ (YBCO) coated conductors [19]. They showed irreversible $I_c$ degradation for $\varepsilon > 0.4\%$ after 10,000 cycles with fatigue ratios $R = \varepsilon_{\text{min}}/\varepsilon_{\text{max}} = 0.2$ and 0.5 and a frequency of 0.4 Hz. Samples subjected to 0.35% and 0.367% maximum strain showed no change in $n$-value, whereas $n$-values for $\varepsilon = 0.38\%$ and $\varepsilon = 0.4\%$ decreased with $I_c$. 
Furthermore, the sample with $\varepsilon=0.4\%$ and 100 cycles showed no reduction in $I_c$, but a $\sim20\%$ reduction in $n$-value. Microstructural studies showed that cracks originating at the edge of the YBCO layer from the slitting process propagate and are the primary failure mode. Reductions in $n$-value correlated with $I_c$ reductions. Detailed analysis showed that the main source of fatigue strength came from the Hastelloy substrate.

Song et al. studied the degradation of YBCO conductors due to quenching [20]. They found that pre-existing defects within the conductor, which result from the manufacturing process, lead to very high local temperatures during quenching. After quenching, $\sim300\,\mu\text{m}$ diameter crater-like defects that expose the Hastelloy substrate were observed. Since the study by Mbaruku et al.[19], YBCO conductor manufacturing and single cycle electromechanical performance has improved significantly with irreversibility limits increasing from $\varepsilon=0.38\%$ to $0.67\%$. Here we report on the fatigue behavior of 4 mm wide YBCO conductors manufactured by Superpower for up to $10^5$ cycles with $R=0$.

**Experimental Approach**

All samples were cut from a single batch of SCS4050 IBAD/MOCVD YBCO coated conductor manufactured by SuperPower Inc. The conductor architecture is comprised of a 1 $\mu$m thick YBCO layer deposited on a bi-axially textured buffer stack atop of a 50 $\mu$m thick Hastelloy substrate. The buffer stack includes MgO, Al$_2$O$_3$, and LaMnO$_3$. The 12 mm wide YBCO layer is protected by a 1 $\mu$m Ag cap layer and then slit into 4 mm wide conductors by the manufacturer. Lastly, the 4 mm wide conductor is encapsulated by a 20 $\mu$m Cu stabilizer via electroplating for a total thickness of 100 $\mu$m. 100 mm long samples were cut from a
continuous roll of 4 mm wide conductor; the sample length was limited by the tensile loading machine.

Room temperature fatigue was performed for ε=0.35%, 0.45% and 0.50%, and for 1, 10, 10^2, 10^3, 10^4 and 10^5 fatigue cycles (N). A new sample was used for each unique combination of ε and N to reduce errors from the repeated thermal cycling required for electrical transport measurements and stresses associated with gripping the samples. For N<10^5, fatigue loadings were completed with a fatigue ratio R= 0 and a frequency of 0.25 Hz. For N=10^5, the frequency was increased to 0.5 Hz to reduce the time required. Thus, for each cycle, the sample was at rest (ε=0), strained to the desired value, and relaxed back to ε=0 in 4 s (2 s for N=10^5). Because a fixed frequency was used, the strain rate varied. For ε=0.35%, strain rate was 0.175 s^{-1} under 10^5 cycles and 0.35 s^{-1} for 10^5 cycles. For ε=0.45%, strain rate was 0.225 s^{-1} under 10^5 cycles and 0.45 s^{-1} for 10^5 cycles. For ε=0.50%, strain rate was 0.25 s^{-1} under 10^5 cycles and 0.50 s^{-1} for 10^5 cycles. Samples were gripped by MTS friction grips and fatigued using an Instron electromechanical system. After fatiguing for the desired number of cycles, the sample was removed from the Instron for characterization.

The manufacturer-reported critical current I_c(77 K, self-field) = 100 A, corresponding to J_c = 25,000 A/mm^2. In this work, I_c(77 K, self-field) was measured for all samples before and after fatiguing; any sample with a pre-fatigue I_c(77 K, sf) <100 A was discarded. All I_c measurements were performed in liquid N_2 using the four-point method, a 1 µV/cm electric field criterion and a voltage tap spacing of 5 cm. The transition index (n-value) was determined by the power-law model [21]. Effective fatigue experiments require gripping the
sample without slipping. Thus the gripping forces on the top and bottom edge of the conductors must be large and are likely greater than the c-axis irreversibility limit of 30 MPa [22]. As a result, the sections of conductor that are gripped are likely damaged and not viable attachment points for current leads. Instead, current leads were attached 10 mm from the grips to ensure good electrical contact.

YBCO microstructures of as-received conductors and after post-fatigue $I_c$ measurements were investigated. Samples were etched to expose the YBCO layer using a two stage process [20]. The first stage removed the Cu with APS Cu etchant at 40°C until the Cu was visibly removed, typically 15 minutes. The conductor was then washed with methanol and placed in a 1:1:4 mixture of H$_2$O$_2$, NH$_4$OH, and methanol to etch the Ag cap layer. This process required 1-2 minutes after which conductor was washed with methanol and air dried.

Samples were imaged in two scanning electron microscopes (SEMs), a FEI Verios 460L scan and a JEOL JSM-6010LA. Energy dispersive x-ray spectroscopy (EDS) in the Verios was used to image the distribution of elements within the samples.

**Results**

Figure 22 plots $I_c(\varepsilon)/I_{c0}$ at 77 K versus $N$ for the three strain values studied, where $I_c(\varepsilon)$ is the critical current after fatigue and $I_{c0}$ is the critical current before fatigue, $I_{c0}$ for these tapes ranged from 100 A to 115 A, with an average of 107 A and a standard deviation of 4.2 A. For $\varepsilon=0.35\%$ and $\varepsilon=0.45\%$, $I_c$ degradation is first observed for $10^4$ cycles, whereas for $\varepsilon=0.50\%$, $I_c$ is reduced after only $10^2$ cycles. The corresponding n-value versus $N$ are seen in Figure 23. Unlike $I_c$, n-value decreases for all combinations of $\varepsilon$ and $N$ studied.
Two types of defects are observed on the YBCO surfaces of as-received samples. Figure 24 shows the presence of ~3 µm diameter Cu rich particles, identified by EDS and consistent with [23]. Furthermore, cracks are observed along the conductor edge in the YBCO layer. The cracks are roughly parallel to each other and at an angle ~38° relative to the conductor edge. Their spacing varies from 5 to 30 µm. Note that the cracks penetrate through the Cu rich particles and in no case does it appear that a Cu rich particle causes crack arrest or blunting. The crack lengths range from 20 µm - 65 µm.

Figure 25 shows an SEM image and the corresponding EDS maps of Al and Ba distributions, and a composite EDS map of Al, Cu and O, of a typical “crater defect” on the exposed YBCO surface of a region away from the edge of the same as-received conductor imaged in Figure 24. Al is studied because it is present in the buffer stack, so the existence of a nearly pure-Al area indicates that the crater is a local area that lacks YBCO. The crater shown here is ~ 70 µm in diameter, but similar craters ranging from 20 µm to 500 µm are randomly dispersed on the YBCO surface.

Figure 26 shows a SEM image of the YBCO surface after $10^3$ cycles at $\varepsilon=0.45\%$. For this case, $I_c(\varepsilon)/I_{c0}=1.0$ and $n(\varepsilon)/n(N=0)=0.55$. No YBCO delamination is observed and the cracks at the edge of the conductor are similar to those seen in the as-received conductor. Figure 27 shows an SEM image of a crater defect on the YBCO surface from the same sample as Figure 26. The crater is similar to that seen in Figure 25, but in this case multiple radial cracks are observed as a result of the fatigue loading. The cracks propagated linearly in directions that appear random relative to the loading direction. Crack lengths range from 9 to
32 μm. Similar cracks are observed emanating from other crater-defects, regardless of the size of the crater.

Figure 28 shows a SEM image of the exposed YBCO surface of a conductor subjected to $\varepsilon = 0.45\%$ for $10^4$ cycles, resulting in $I_c(\varepsilon)/I_{c0} = 0.96$ and $n(\varepsilon)/n(N=0) = 0.57$. EDS analysis confirms YBCO delamination and the exposure of the buffer layers. Delamination initiated at the conductor edge and propagated inward. Crack propagation from the conductor edge inwards at a $\sim41^\circ$ angle is also observed. Crater defects are visible and have cracks ranging in length from 10-45 μm. No delamination is seen near the crater defects or their cracks.

Figure 29 shows the exposed YBCO surface of a sample subjected to $\varepsilon = 0.5\%$ for $10^4$ cycles, resulting in $I_c(\varepsilon)/I_{c0} = 0.7$ and $n(\varepsilon)/n(N=0) = 0.37$. This image was taken 200 μm from the conductor edge. A network of cracks, mostly parallel and orthogonal to the applied load direction but not exclusively so, is observed. Although damage is severe in this area, large areas of undamaged YBCO are also observed, particularly in the center of the conductor.

**Discussion**

Mbaruku et al. [19] showed that the mechanical properties of REBCO conductor are driven primarily by those of the Hastelloy substrate. It is important to note that the REBCO conductor is a composite system, and failure within any of the conductor layers can trigger electrical degradation in the functional YBCO layer. Thus, for example, a crack in the Hastelloy or buffer layer likely results in crack nucleation in the YBCO. Tensile fatigue of Hastelloy has been studied at room temperature, with failure occurring at greater than $10^5$ cycles [24, 25] at the strain values used in this study. Furthermore, the conductor ultimate
tensile strength cannot be used as a failure criterion for magnets, as electrical transport is the primary conductor function; a physically in-tact conductor with significantly reduced $I_c$ has failed. Thus, an operational fatigue criterion depends on the electrical behavior of the YBCO layer. Here, any conductor showing $I_c(\varepsilon)/I_{c0} < 0.9$ is said to have failed electromechanically. The SEM images of the as-received conductor show microcracks along the conductor edge, which are likely due to the slitting process used to convert the 12 mm wide as-grown conductor into three 4 mm wide conductors. During tensile loading, the microcracks are stress concentrators with peak stresses at the crack tips. The most prevalent physical damage observed in conductors after fatigue are longer cracks that penetrate towards the tape center as well crack networks like that seen in Figure 29. These cracks are the result of fatigue-induced propagation of the pre-existing microcracks; no evidence of the nucleation of new cracks is observed. Due to the slow loss of $I_c$ with increasing $N$, we are observing slow crack growth which is common with strain controlled fatigue.

Another source of crack growth during tensile fatigue is the surface defects seen in the YBCO layer. Figure 26 shows a crater like defect in the YBCO layer with cracks that have grown radially. Although these defects appear circular, the jagged edges with small radii of curvature act as stress concentration sites that can facilitate crack growth.

Comparing these results to those from Mbaruku et al. [19], here we find that for $\varepsilon=0.35\%$ degradation occurs between $10^4$ and $10^5$ cycles as where Mbaruku saw no degradation at $10^5$ cycles. This may be due to differences in the fatigue ratio. Here we present data using a fully relaxed fatigue loading, $R=0$, where Mbaruku et al. used loading paths with $R=0.2$ and $R=0.5$. Fully relaxing the sample during each cycle increases the total strain energy imparted
per cycle, increasing the likelihood of crack propagation and damage at a lower number of cycles. Considering that the strain energy per cycle is \( U = \frac{1}{2} \int \frac{N_x^2}{EA} dx \), a REBCO sample that undergoes a loading path with R=0 is subject to 4.1% more strain energy per cycle than a path with R=0.2.

For \( \varepsilon=0.45\% \), Mbaruku et al. [19] showed that conductor damage occurs at 1,000 cycles whereas in this work \( I_c \) reduction did not occur until 10,000 cycles. Improvements in the manufacturing processes may explain the increased strain tolerance at elevated \( \varepsilon \) and low \( N \). These improvements are overshadowed at high \( N \) by the increased strain energy using a loading path with R=0. Thus at high \( \varepsilon \) and low \( N \), we find greater fatigue life than Mbaruku et al. [19], whereas at low \( \varepsilon \) and high \( N \) the fatigue life is shortened.

It is important to note that here we report on fatigue performed at room temperature, whereas Mbaruku et al. [19] fatigued the conductor at 77 K. For neither the 77 K nor room temperature tensile fatigue loadings does the REBCO begin in a zero-strain state due to thermal contraction mismatch from cool-down after processing. Because of this mismatch, at 77K the compressive strain on the REBCO (i.e., the “starting point” for the fatigue measurements) is greater than that at room temperature. Thus, for the same applied tensile strain during fatigue, the net strain on the REBCO is higher at room temperature than at 77 K. In addition, cracking in the Hastelloy substrate damages the REBCO layer [26], and the fatigue life of Hastelloy decreases with increasing temperature. Thus, room temperature fatigue measurements provide more conservative results than lower temperature measurements.
Mbaruku et al. [19] concluded that the main source of crack propagation came from the conductor edge, whereas here the crater defects also play a role in crack nucleation. Song et al. [20] reported seeing similar defects in quenched REBCO conductors, however Song et al. [20] showed the craters penetrating through to the Hastelloy substrate. This study only finds craters that penetrate into the buffer stack. Song et al. [20] studied quenched conductors that may have started with similar defects as seen in Figure 26, however the > 10 quench events and greater localized heating of the conductor caused more intensive local damage (delamination) and thus exposed the substrate.

Figure 23 shows n-value decreasing with increasing $\varepsilon$ and N for all cases. It is of interest to see the difference in shape from the $I_c(N, \text{strain})$ and the $n(N, \text{strain})$ curves. While the $I_c$ curve in Figure 22 decreases exponentially, the n-value curve in Figure 23 decreases linearly with N. Mbaruku et al. shows n-value results that are jagged with sharp decreases corresponding to the onset of $I_c$ reductions. These differences are explained as being due to delamination strength differences in the tapes. Delamination strength has been shown to be one of the primary challenges facing SuperPower REBCO conductors, so SuperPower focused its efforts on improving the delamination strength. The tapes studied by Mbaruku et al. likely had lower delamination strength and failed quickly over the course of a few cycles, whereas the tapes used in this study benefited from SuperPower’s more recent advances and thus likely had a slower failure, resulting in the broader curves observed [27].

It is theorized that small microcracks have evolved in the YBCO layer, resulting in micro and nano-voltages, and reducing the sharpness of the V/I curve. Knowing that cracks and delamination in the YBCO layer can decrease the n-value, it is possible to understand at what
ε and N damage to the conductor begins. For the conductor subjected to 1,000 cycles at 
ε=0.45%, \( I_c(\varepsilon)/I_{c0} = 1.0 \) and Figure 25 shows no clear microstructural changes. The n-value
 for this conductor, however, has decreased by ~50%, indicating that the YBCO layer is
 affected significantly. As no changes are seen in the SEM, the damage may be occurring
 below the conductor surface, or there may be small areas of delamination between the
 YBCO/buffer layer/Hastelloy. Regardless of the mechanism, it is important to note, that we
detect damage to the conductor before it manifests as a reduction in \( I_c \). To further illustrate
this, by 100 cycles all conductors experience a reduction in n-value even though
\( I_c(\varepsilon)/I_{c0}>0.94\% \) for all conductors. Microcracks in the YBCO layer have given rise to local
voltages, implying the onset of fatigue damage. Mbaruku et al. [19] saw a similar
phenomenon in some samples. At \( \varepsilon=0.4\% \) and 100 cycles, a 20% reduction in n-value was
reported with no reduction in \( I_c \), and at \( \varepsilon=0.45\% \) and 11,000 cycles, a 31% reduction in n-
value was reported with no reduction in \( I_c \). These decreases in n-value were observed just
before a sharp drop in \( I_c \) occurred.

Although REBCO retains \( I_c(\varepsilon)/I_{c0} = 1.0 \) at 0.45% strain and 10,000 cycles, emerging
 technologies such as SMES subject REBCO conductors to higher cyclic loads. Increased
 fatigue life would result from reducing conductor defects and an improved slitting process.
 Furthermore, magnet design must consider the implication of reduced n-value on quench
detection and overall magnet performance.

Conclusion

REBCO conductors retain their as-received \( I_c \) after \( \varepsilon=0.35\% \) and \( \varepsilon=0.45\% \) at 10,000
cycles, and \( \varepsilon=0.50\% \) at 100 cycles. N-value decreases at very low values of N, however, and
continues to decrease linearly with N to 10,000 cycles. The main sources of fatigue failure are the defects from the manufacturing process leading to crack propagation. The slitting process used to reduce 12 mm conductors to 4 mm conductors introduces micro-cracks at the outer surface of the conductor edge, and these act as stress concentrators and grow during fatigue. R=0 fatigue experiments were performed and determined that the conductor shows both less and greater fatigue life when compared to Mbaruku et al. [19]. The difference in R value, and therefore strain energy explains the differences seen at higher N, while improvements in conductor manufacturing are realized at low N and high ε.

**Acknowledgements**

The authors thank ARPA-E (project # AR0000337) for providing funding for this research, Dr. Chris Rey and Trever Carnes for providing the conductor, Toby Tung for assistance with the SEM, and Weston Straka for providing useful discussions. The authors acknowledge the use of the Analytical Instrumentation Facility (AIF) at North Carolina State University, which is supported by the State of North Carolina and the National Science Foundation.
Figure 22: $I_c(\varepsilon)/I_{c0}$ vs number of cycles for $\varepsilon = 0.35\%, 0.45\%$ and 0.50\%. $I_c$ measurements are performed at 77 K, self-field.
Figure 23: $n$-value vs number of cycles for $\varepsilon = 0.35\%, 0.45\%$ and $0.50\%$ (77 K, self-field)
Figure 24: SEM image of as received conductor. Note the cracking on the edge, thought to be from slitting process.
Figure 25: EDS image (Al and Ba) of a crater defect seen on as received conductor. Note the center of the defect is showing the alumina layer from the buffer stack. These defects from manufacturing act as stress concentrators for crack nucleation and propagation.
Figure 26: SEM of the YBCO surface of a conductor subjected to 0.45% strain for 100 cycles. No cracking is seen propagating inward from conductor edge. Arrows indicate the load direction.
Figure 27: SEM image of the YBCO surface of a conductor subjected to 1,000 cycles at 0.45% strain. The area is in the center of the conductor. The craters have small microcracks radially propagating from the edges. Edge cracking from the slitting process was similar.
Figure 28: SEM image of a crack on the surface of a conductor subject to 0.45% strain for 10,000 cycles. Cracking here is limited to the conductor edge, note that the conductor Ic is reduced 4%. Arrows indicate the load direction.
Figure 29: SEM image of the surface of a conductor subjected to 0.50% strain for 10,000 cycles. The YBCO layer appears “shattered” and the damage resulted in a 30% reduction in $I_c$. Arrows indicate the load direction.
References


Chapter 3

Tensile Fatigue Behavior and Crack Growth in GdBa$_2$Cu$_3$O$_{7-x}$/Stainless-Steel Coated Conductor Grown via Reactive Co-evaporation

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Abstract

(RE)Ba$_2$Cu$_3$O$_{7-x}$ (REBCO) conductors have the potential to enable a wide range of superconducting applications over a range of temperatures and magnetic fields [1], yet AC applications and devices with a charge/discharge cycle may be limited by the conductor fatigue properties. Here the fatigue behavior of GdBa$_2$Cu$_3$O$_{7-x}$ (GdBCO) conductors grown by reactive co-evaporation on stainless-steel substrates is reported for axial tensile strains, $\varepsilon$, up to 0.5 % and 100,000 cycles. Failure mechanisms are investigated via microstructural studies and compared with other commercially available REBCO conductors. Results show that GdBCO/stainless-steel conductors retain their transport critical current for 10,000 cycles at $\varepsilon=0.35$ % and $\varepsilon = 0.45$%, and for 1,000 cycles at $\varepsilon = 0.50$ %. The main cause of fatigue degradation in GdBCO conductors is crack propagation and delamination that initiates at the edge of the conductor due to manufacturing defects.

Introduction

REBa$_2$Cu$_3$O$_{7-x}$ (REBCO) coated conductors are important materials for the generation of high magnetic fields for research and for industrial uses [2]. REBCO conductors are available
from many manufacturers using different deposition techniques, with varying electrical properties and mechanical behaviors. REBCO conductors have high critical current density, $J_c$, in the presence of a high background magnetic field, and favorable mechanical properties due to their high strength substrates and the REBCO layer being near the neutral axis [3]. As a result, REBCO conductors are of interest for a multitude of superconducting magnet applications, including nuclear magnetic resonance, motors, generators, and energy storage. Further development in quench detection has also made them a prime choice for use in fusion reactors and high energy physics [4]. Many of these superconducting applications require AC operation and will thus be subjected to fatigue loads in addition to the stresses from fabrication and differential thermal contraction. A thorough understanding of the electromechanical properties of REBCO conductors is thus needed to produce devices that perform over their lifetime [5].

Many factors differentiate the commercial REBCO conductors, including deposition method, e.g. ion beam assisted deposition (IBAD) and rolling-assisted biaxially textured substrates (RABiTS), substrate (NiW, Hastelloy, and stainless steel), and conductor outer layer (electroplated Cu, Cu lamination, brass lamination, etc.). Each of these variables affects the electromechanical behavior. Operation above the irreversibility limit must be avoided to prevent permanent damage to the conductor.

Several studies have reported REBCO conductors are easily damaged due to delamination, transverse compression, as well as quench events [6][7][8][9]. Burrs and micro-cracks at the conductor edges due to the slitting process reduce the mechanical and electromechanical
strength as measured via the anvil test method. In addition to delamination arising from transverse loading, high-cycle tensile fatigue damages conductors at strain values much lower than the irreversibility limit [10][11]. Recently, the need for a 32 T, 34 mm bore REBCO magnet that operates for 50,000 cycles over 20 years has been identified [12], yet data for high cycle electromechanical behavior is not currently available for the relevant REBCO conductors.

Recent studies by Barth et al. investigated the single cycle behavior of commercially available REBCO conductors. They showed that the irreversibility limit, the strain ($\varepsilon$) at which transport is permanently reduced, ranges from $\varepsilon = 0.45$-$0.70$ % [13]. High-cycle fatigue was first studied by Mbaruku et al. [10] using 4 mm wide SuperPower IBAD/MOCVD Hastelloy YBa$_2$Cu$_3$O$_{7-x}$ (YBCO) coated conductors. They showed irreversible degradation to the critical current ($I_c$) for $\varepsilon >0.4$% after 10,000 cycles with fatigue ratios $R = \varepsilon_{\text{min}}/\varepsilon_{\text{max}} = 0.2$ and 0.5 and a frequency of 0.4 Hz. Samples fatigued with 0.35 % and 0.367 % maximum strain showed no change in n-value, however n-values for $\varepsilon_{\text{max}} = 0.38$ % and $\varepsilon_{\text{max}} = 0.40$ % decreased with $I_c$. Microstructural investigation showed that cracks at the edge of the YBCO layer from the slitting process propagate and are the primary cause of failure.

Osamura et al. [14] reported on low cycle (N<100) fatigue damage. They showed that critical current decreases by 1-5% at ~20 cycles when the strain equals the recovery strain. From 20-100 cycles the critical current remains constant or decreases slowly.

More recently, Rogers et al. [15] investigated the high cycle fatigue behavior of SuperPower
conductor similar to but more recently manufactured than used by Mbaruku. They showed that $I_c$ was unchanged after $\varepsilon = 0.35\%$ and $\varepsilon = 0.45\%$ for 10,000 cycles, and $\varepsilon = 0.50\%$ for 100 cycles, with $R=0$. The n-value decreased for a low number of cycles (N) and continued to decrease linearly to $N = 10,000$. Fatigue failure was caused by the manufacturing processes, leading to crack propagation. In particular, the slitting process used to reduce the conductor width from 12 mm to 4 mm introduced micro-cracks at the outer surface of the conductor edge, and these microcracks act as stress concentrators that result in crack growth during fatigue.

Here we report on fatigue results for industrially-manufactured GdBa$_2$Cu$_3$O$_{7-x}$/stainless-steel conductor under identical conditions as reported previously [15]. Results indicate how differences in conductor manufacturing impact electromechanical damage under high cycle tensile loadings.

**Experimental Approach**

All samples were cut from a single batch of GdBCO coated conductor produced by SuNAM using reactive co-evaporation (RCE) [16]. The conductor architecture includes a 2 $\mu$m thick GdBCO layer deposited on a bi-axially textured buffer stack consisting of MgO, Al$_2$O$_3$, and LaMnO$_3$ atop a ~105 $\mu$m thick stainless steel substrate. The 12 mm wide GdBCO layer is protected by a Ag cap layer and then slit into 4 mm wide conductors by the manufacturer. Finally, the 4 mm wide conductor is encapsulated by brass lamination for a final width of 4.2 mm and thickness of 0.25 mm. For fatigue experiments, 10 cm long samples were cut from
the continuous roll of conductor; the sample length was limited by the tensile testing machine.

Room temperature tensile fatigue was performed for $\varepsilon = 0.35 \, \%$, $0.45 \, \%$ and $0.50 \, \%$, and $N = 1, 10, 10^2, 10^3, 10^4$ and $10^5$. A new sample was used for each unique combination of $\varepsilon$ and $N$ to reduce errors from the thermal cycling required for electrical transport measurements and stresses associated with handling and gripping the samples. Fatigue strain rate and loading conditions were as follows. For $\varepsilon=0.35\%$, strain rate was $0.175 \, s^{-1}$ under $10^5$ cycles and $0.35 \, s^{-1}$ for $10^5$ cycles. For $\varepsilon=0.45\%$, strain rate was $0.225 \, s^{-1}$ under $10^5$ cycles and $0.45 \, s^{-1}$ for $10^5$ cycles. For $\varepsilon=0.50\%$, strain rate was $0.25 \, s^{-1}$ under $10^5$ cycles and $0.50 \, s^{-1}$ for $10^5$ cycles. These are identical conditions to those used previously [15]. Samples were gripped by MTS friction grips and fatigued using an Instron mechanical system. After fatiguing for the desired number of cycles, the sample was removed from the Instron for characterization.

The manufacturer reported a critical current $I_c (77 \, K, \text{self-field})$ of 250 A, corresponding to $J_c$ of 31,250 A/mm$^2$. In this work, $I_c (77 \, K, \text{self-field})$ was measured for all samples before and after fatiguing; any sample with a pre-fatigue $I_c (77 \, K, \text{self-field}) < 250 \, A$ was discarded. All $I_c$ measurements were performed in liquid N$_2$ using the four-point method with a 1 $\mu$V/cm electric field criterion and a voltage tap spacing of 5 cm. The transition index ($n$-value) was determined by the power-law model [17].

Microstructures of as-received conductors and after post-fatigue $I_c$ measurements were investigated. The GdBBCO layers were exposed using a two-step process. The first step was to
heat the conductor slowly until the solder holding the brass lamination melted, so the brass laminate was removed easily using tweezers. The conductor was then washed with methanol and placed in a 1:1:4 mixture of H$_2$O$_2$, NH$_4$OH, and methanol to etch the Ag cap layer. This process required ~2 minutes after which the conductor was washed with methanol and air dried. Samples were imaged in a JEOL JSM-6010LA scanning electron microscope. Energy dispersive x-ray spectroscopy (EDS) was used to image the distribution of elements within the samples.

**Results**

Figure 30 plots $I_c(\varepsilon)/I_{c0}$ verses N at 77 K for the three strain values studied, where $I_c(\varepsilon)$ is the critical current after fatigue and $I_{c0}$ is the critical current before fatigue. $I_{c0}$ for these conductors ranged from 251-258 A, with an average of 253 A and a standard deviation of 2.4 A. For $\varepsilon = 0.35$ % and $\varepsilon = 0.45$ %, $I_c$ degradation is first observed for $10^4$ cycles, whereas for $\varepsilon = 0.50$ %, $I_c$ decreases after $10^3$ cycles. The corresponding n-value versus N results are seen in Figure 31. Unlike $I_c$, n-value decreases for $N>10^2$ for all values of $\varepsilon$ and continues to decrease with increasing N.

Figure 32 shows an SEM micrograph of the exposed GdBCO surface of an as-received conductor. Two types of defects are observed, copper rich surface particles distributed throughout the GdBCO layer surface and micro-cracks originating at the conductor edge that propagate inward at an angle of $\sim 30^\circ$. The average length of the micro-cracks is 50 $\mu$m and they are $\sim 20$ $\mu$m apart on average.
Figure 33 shows an SEM micrograph and corresponding EDS images of the GdBCO surface after $10^4$ cycles at $\varepsilon = 0.50 \%$. For this case, $I_c(\varepsilon)/I_{c0} = 0.62$ and $n(\varepsilon)/n(N=0) = 0.48$. Both delamination and cracking are observed in the GdBCO layer. An area $\sim 70\text{-}100 \, \mu \text{m}$ from the edge has delaminated, and cracks penetrate $150 \, \mu \text{m}$ into the conductor from the edge. The corresponding EDS images show that the delamination has exposed the stainless steel substrate, as no trace of the buffer stack elements are detected. Cracking is also seen in the stainless steel near the edge of the conductor where delamination has occurred.

Figure 34 shows a SEM micrograph of the GdBCO surface after $10^5$ cycles at $\varepsilon = 0.50 \%$. In this case, $I_c(\varepsilon)/I_{c0} = 0.29$ and $n(\varepsilon)/n(N=0) = 0.18$. Significant cracking and delamination are seen across the conductor face, with cracks observed from the edge of the conductor that originated as slitting defects.

Figure 35 plots average crack length vs. $N$ at $\varepsilon = 0.45\%$. Cracks were measured at 3 locations along the conductor, at both ends where the current leads were attached and at the conductor center. 10 cracks were measured at each location and their lengths averaged.

Figure 36 shows a SEM micrograph of a 12 mm wide SuNAM conductor as received from the manufacturer. This SEM highlights that there are no edge defects present in the un-slit tape; thus we infer that the slitting process is responsible for the edge damage seen in the 4 mm wide as-received conductor.
Discussion

Mbaruku et al. showed that the mechanical properties of REBCO conductor are driven primarily by the Hastelloy substrate [10], and that a failure in any of the materials in the composite can cause electrical degradation in the superconducting layer. Here, any conductor showing $\frac{I_c(\varepsilon)}{I_{c0}} < 0.9$ is said to have failed electromechanically. The conductors studied here have a stainless steel substrate with a fatigue life $N<10^5$ when subject to $\varepsilon = 0.50 \%$ [18]; note that the substrate does not fail under the parameters studied here. One characteristic difference between stainless steel and Hastelloy is the manner in which they yield. Luder’s bands, non-uniform bands of stress that occur when the material yields, form in Hastelloy but not in stainless steel. The continuous yield exhibited by stainless steel explains the higher irreversibility strain limits observed in REBCO conductors with a stainless steel substrate.

SEM images of the as-received conductor show microcracks on the conductor edge that result from the slitting process used to reduce the conductor width from 12 mm to 4 mm. As shown previously [15], these cracks act as stress concentrators which cause the cracks to penetrate towards the center of the conductor during fatigue. The resulting longer cracks reduce the transport critical current [10].

This work was performed using strain-based fatigue to compare results from previous similar work using Superpower conductors [15]. The SuNAM conductors have 30% higher as-received $n$-value compared to the SuperPower conductors. The as-received Superpower conductors had crater like defects ranging from 50-300 $\mu$m in diameter, whereas the SuNAM surface is free of this type of defect. Based upon SEM images, the SuNAM slitting process
produces damage similar to what is observed in SuperPower conductors; the resulting cracks are of similar length, whereas the SuNAM conductor crack-to-crack spacing is larger than the ~15 µm spacing seen in the SuperPower conductors.

SuNAM conductors show less I\textsubscript{c} degradation at strain values studied than the SuperPower conductors. One possible explanation is that the SuNAM deposition process results in a more uniform GdBCO layer. The surface of the SuNAM conductor contains less defects than the SuperPower conductor, and this increased homogeneity may account for reduced crack propagation during the slitting process and fatigue. Cracks in the SuperPower tapes are 3-5 % longer than in the corresponding SuNAM conductor. It is also important to consider if the REBCO layer and buffer stack are more strongly and homogeneously bonded to the substrate, then there will be less areas of stress concentration in the composite material. Crack propagation in the SuNAM tape requires more strain energy due to less stress concentration than in the SuperPower conductors.

A second possible explanation for higher I\textsubscript{c} retention in the SuNAM conductors is the substrate; stainless steel yields at a lower stress than Hastelloy [19] resulting in the ability for the stainless steel to work harden at a strain value for which the Hastelloy would not. Work hardening will increase the mechanical properties of the substrate and likely affect fatigue life.

Figure 33 shows the GdBCO surface of a conductor subjected to \( \varepsilon = 0.50 \) % for \( N=10^4 \). In addition to cracking, the conductor has delaminated continuously at the edge. This behavior occurs between the stainless steel substrate and the Al\textsubscript{2}O\textsubscript{3} diffusion barrier. In the
SuperPower conductors subjected to higher cycles, delamination occurred within the buffer stack and at the buffer/REBCO interface. The fundamental difference in interlayer adhesion likely plays an important role in the fatigue life in composite conductors.

Another significant difference between the conductors’ behaviors is their initial n-value and how it is affected by low cycle fatigue. The n-value quantifies how sharply the E-J curve transitions from the superconducting to normal state [17], and in general n-value increases with a more homogeneous and phase-pure superconducting layer. The SuperPower n-value was initially 30, and decreased linearly for N > 10. SuNAM conductors exhibited an initial n-value of 41 and showed no appreciable decrease until N = 100. The higher initial value is consistent with the relatively high homogeneity in the SuNAM conductor as compared to the SuperPower conductor. It is important to look at how the n-value changes as a function of N between the different manufacturers. SuperPower n-value shows a linear decrease in response to loading at N>10, representative of damage in the superconducting layer resulting in a less uniform transition. It is expected that as a superconductor is damaged the n-value will decrease, however for the SuperPower conductor at N<100 I_c remains constant. Thus despite some local damage to the REBCO layer, the conductor is still able to transport the same current density. It is inferred that there are local voltages being produced by nano-cracking or grain misalignment.

The SuNAM conductor n-value does not show a large decrease until N=100, corresponding to more rapid crack growth. n-value and crack length are inversely related and show a much
clearer correlation than that between crack length and $I_c$. It also appears that n-value may predict the onset of mechanical failure of a conductor before the critical current decreases.

Figure 35 shows increasing crack length with increasing $N$. As the ability of the conductor to transport current is directly related to the cross sectional area of a continuous superconducting layer, one would expect the crack length to be linearly proportional to the decrease in $I_c$; but it is also important to note that the results in Figure 35 are average crack lengths, and transport will be limited by the longest crack which results in the narrowest conductor width. For example, at $N=100,000$, the average crack penetrates ~23 % of the tape width, however $I_c$ is reduced by 39 %. This is likely due to a long crack that extends further into the conductor reducing transport, but not seen in the SEM image used for the crack length measurements.

For $N=100$-$10,000$, the percentage reduction in cross-sectional area is greater than the percentage reduction in $I_c$. This is hypothesized to be caused by multiple effects. First consider self-field effects. As the transport current is reduced, so is the self-field. Reduced self-field results in increased local $J_c$ and thus a lower reduction in $I_c$ than predicted solely by crack length. A second reason is the edge defects present in the as-received conductor that penetrate ~50 µm into the conductor. However, the superconducting layer may have been damaged by nano-cracking that was not visible under the SEM. This crack area would not transport current, but would appear to be mechanically sound when viewed under SEM.

Although the SuNAM conductor shows retained transport at $N<10^4$, magnet manufacturers are aiming for 50,000 cycles of operation with less than 5% decrease in transport. To
increase fatigue life, conductor manufacturers need to improve or eliminate the slitting process currently used to reduce the conductor width, thereby eliminating the edge cracks that propagate and limit fatigue life. Another area for improvement remains in the adhesion between layers of the composite conductor. Composite materials transfer stresses between the layers, and if localized delamination occurs, it will also act as a stress concentrator and reduce fatigue life.

It is important to note that these experiments were performed at room temperature and therefore do not have the pre-compression that occurs on the REBCO layer due to differential thermal contraction with the conductor is cooled to cryogenic temperature. The compressive stress pre-strain in the REBCO layer depends on the substrate thickness and the outer protective layers. The SuNAM conductor has a thicker substrate and thicker copper lamination than the SuperPower conductor. Although thicker substrates and protective layers reduce the engineering current density, $J_e$, they apply more compressive pre-strain to the REBCO layer which could improve the fatigue properties.

**Conclusion**

GdBCO conductor manufactured by SuNAM retained their as-received $I_c$ after $\varepsilon = 0.35 \%$ and $\varepsilon = 0.45 \%$ after 10,000 cycles, and $\varepsilon = 0.50 \%$ after 1000 cycles. $n$-value decreased for $N=100$ and continued to decrease up to $N = 100,000$. The main causes of fatigue failure are the slitting process used to reduce the conductor width, which introduces micro-cracks at the conductor edge. These cracks act as stress concentrators and grow longer with increasing $N$. In comparison to the SuperPower conductors studied previously, the SuNAM conductor
shows greater fatigue life and higher initial n-value due to less dense slitting cracks, a different conductor architecture, and a superconducting layer with less defects.

**Acknowledgements:**

The authors would like to thank SuNAM for providing the conductor used in this study. This work was performed in part at the Analytical Instrumentation Facility (AIF) at North Carolina State University, which is supported by the State of North Carolina and the National Science Foundation (award number ECCS-1542015). The AIF is a member of the North Carolina Research Triangle Nanotechnology Network (RTNN), a site in the National Nanotechnology Coordinated Infrastructure (NNCI).
Figure 30: $I_c(\varepsilon)/I_{c0}$ vs number of cycles for $\varepsilon = 0.35\%$, 0.45\% and 0.50\%. $I_c$ measurements are performed at 77 K, self-field.
Figure 31: $n$-value vs N for $\varepsilon = 0.35\%, 0.45\%$ and $0.50\%$ (77 K, self-field)
Figure 32: SEM micrograph of the exposed GdBaCO surface of an as-received conductor. Note the cracks due to the slitting process which formed at ~30° angle to the edge of the tape and are 50 µm in length.
Figure 33: SEM micrograph and EDS images of the GdBCO surface after 104 cycles at $\varepsilon = 0.50\%$, $I_c(\varepsilon)/I_c0 = 0.62$. Delamination is seen through to the substrate, the GdBCO layer shows cracks that penetrate 150 µm from the tape edge.
Figure 34: SEM micrograph of the GdBCO surface subject to $10^5$ cycles at $\varepsilon = 0.50 \%$. $I_c(\varepsilon)/I_c0 = 0.29$ and $n(\varepsilon)/n(N=0) = 0.18$. Significant cracking and delamination can be seen throughout the surface of the conductor.
Figure 35: Crack length and transport as a function of number of cycles at 0.45% strain. Note the correlation between crack length and decreased transport.
Figure 36: SEM of an as-received 12 mm wide SuNAM conductor that shows no edge cracking as the slitting process was not performed.
References:


29, no. 9, p. 94003, 2016.


Chapter 4

Tensile Fatigue of IBAD-MOCVD YBa$_2$Cu$_3$O$_{7-x}$/Hastelloy Coated Conductor Packaged in an Aluminum Conduit

Introduction

As discussed in the introduction, HTS conductor is viable for many energy applications [1]. One of these applications that shows great promise is Superconducting Magnetic Energy Storage (SMES). SMES is the storage of energy in a magnetic field. This technology has been investigated and proposed for multiple decades as a way to efficiently regulate power to meet the needs of society’s diurnal power cycle [2]. Unlike batteries and traditional power storage techniques, SMES is a “green” technology with little environmental impact and allows greater than 95% round trip efficiency. Another benefit to SMES is the ability to charge and discharge the system extremely rapidly without damage that would occur to a battery. The time needed to charge and discharge a SMES is as fast as supercapacitors. [3][4]
SMES has been met with problems in the past when attempted with LTS, such as low energy density, limited storage capacity due to poor conductor performance in high magnetic fields, and costly external support structures to make up for the large size needed to achieve adequate storage capacity. [2] This lack of “scale up” ability is what lead SMES to be abandoned as a technology with LTS.

Now that improved HTS conductor is available that allows operation in excess of 30 T, HTS SMES devices would have over 35 times the energy density of a comparable LTS SMES and would allow for these systems to transported by truck or train. [5][6]

In order to wind and construct a SMES, multiple HTS conductors must be used and packaged together. Past cable in conduit conductor (CICC) has been used on many large scale
superconducting builds such as ITER. CICC offers many benefits such as increased thermal mass to prevent a quench event, and the ability to easily wind multiple conductors in a large coil. [7][8][9]

SMES systems will be naturally charged and discharged during use, potentially hundreds of thousands of times during the life of the system. This charge/discharge cycle will vary the Lorentz force developed due to the magnetic field. As mentioned in the introduction, hoop stress can be locally realized as a tensile strain on the conductor. Since we expect varying tensile loadings to be imparted onto the conductor, fatigue life must be well understood for this application. Millions of dollars in HTS conductor will be utilized for each large SMES system [2], and in order to prevent catastrophic failure we must understand the fatigue limits of the conductor as it will be installed into the magnet.

CICC using HTS raises concerns due to the poor compressive tolerance of REBCO conductors [10]. In order to maintain thermal contact with the conduit, the conductor must be physically touching the conduit. This can be achieved through the compaction of the conduit around the stack of HTS conductor. While performing the compaction, it is imperative that the compaction only proceed to the point of electrical contact. Any additional stress imparted to the edge or top of the conductor could cause cracking and delamination in the brittle REBCO layer or buffer stack.
Experimental Setup

The CICC was provided by Energy to Power Solutions (E2P) after work was performed to determine the optimum compaction factor. The cross section of the CICC is shown in the figure below. It consists of 42, 4 mm wide YBCO HTS conductors from SuperPower, in an aluminum conduit. The HTS tape is the same model number as used for the previous fatigue studies on SuperPower conductors as to provide accurate comparison.

The coupon samples provided for fatigue testing only had 4 HTS conductors in the center of the stack, and were surrounded by 19 copper “dummy” tapes on each side of the 4 HTS conductors. This was done due to current limitations for $I_c$ measurements. A stack of 42 tapes would require a power supply in excess of 5 KA.

The fatigue testing regimen was performed using the same methods as the previous work in this thesis, the only difference was the coupon samples used were a stack, and the conduit
only encapsulated the center of the coupon, so that the ends were exposed for gripping and electrical attachments.

Room temperature fatigue was performed $N \leq 10,000$ cycles at varying $\varepsilon_{\text{max}}$ and $\varepsilon_{\text{min}}$ in order to model different fatigue loadings. Critical current reduction was measured and graphed vs. number of cycles to understand at what $\varepsilon$ and $N$ the onset of electrical failure begun.

**Stacked Conductor Fatigue, R=0**

First the stacked conductor was subject to the same fatigue loading strain as performed in previous chapters, although only up to $N=1000$. The results below plot $I_c(\varepsilon)/I_{c0}$ at 77 K versus $N$ for the three strain values studied. At 100 cycles the onset of failure is seen for $\varepsilon = 0.45\%$ and $0.50\%$. After 1000 cycles the $\varepsilon = 0.35\%$ coupon has begun to show a reduction in $I_c$. 
It is important to compare the results seen here to the previous single tape fatigue results.

What we see is that fatigue damage has begun at a much earlier number of cycles and shows much higher transport losses at all N values over 100 as compared to the single tape study. Testing was concluded after 1000 cycles because those were the requirements set forth by the project.

**Stacked Conductor Fatigue, Altering Fatigue Ratio to Simulate Pre-Strain**

As discussed in the introduction, the conductor will see different types of strain throughout its lifetime. Some of the strain will come from the magnet manufacturing or winding process, and will be determined by the overall diameter of the magnet build, and in the case of the CICC the strain will also be affected by the location in the stack as it relates to the

![Figure 39: Stacked conductor transport vs. number of cycles at varying strains with a R value=0](image-url)
neutral axis of the CICC. In order to determine exactly what fabrication strains would be imparted on the conductor, simulations were performed to calculate the tensile strain on certain conductors within the stack. This data is presented below, and shows that for the smallest bend radius of 0.5 m the conductor will be under 0.4 % tensile strain. The 10\textsuperscript{th} tape from the outside of the stack will be subject to 0.2% tensile strain after fabrication is complete. These values were used to perform fatigue in order to simulate more realistic loads that the conductor would see during operation.

![Theoretically Computed Strain on specific tapes in HTS stack when subject to different bend radii](image)

Figure 40: Calculated strain for individual tapes inside the CICC when bent to different radii. This was the basis for the strains chosen in the fatigue study.

With these values we then outlined a fatigue test that would undergo the following loading condition. The minimum strain of 0.15% was chosen due to the fact that 0.65 m bend radius
was the radius of the proposed SMES, and the 10th turn from the outside would give a realistic approximation for the majority of the stack of HTS. This is simply an example of what 2 cycles of fatigue would look like using a $\varepsilon_{\text{min}} = 0.15\%$ and a $\varepsilon_{\text{max}} = 0.35\%$. The R value for this condition would be $R = 0.43$.

![2 Cycle Fatigue: 0.15% Pre Strain; .20% Cyclic Strain](image)

Figure 41: Representation of a loading path with a R value greater than 0. This was used to simulate the pre strain on the conductor during fabrication.

Another loading condition was also utilized with a $\varepsilon_{\text{min}} = 0.15\%$ and a $\varepsilon_{\text{max}} = 0.45\%$ and a R value of 0.33. The $\Delta \varepsilon$ values of 0.15% and 0.2% were chosen based on calculations performed by Wan Kan Chan which showed that was a realistic strain the HTS would be subject to when charging the SMES with the designed support structure. The figure below shows fatigue data for the pre-strain loading pathways.
Data above shows that the fatigue life of the “pre strained” samples are much higher than that of the static samples at corresponding $\varepsilon$ and $N$. Fatigue damage is not realized until $10^3$ cycles, and even then the damage is less than a 2% reduction in $I_c$. Damage is clearly taking place at $10^4$ cycles where it is clear the sample with the higher maximum strain has reduced transport of 7%. These values line up with expectations that as you increase the $\Delta\varepsilon$, you will see increased damage due to greater strain energy being input to the material.

It is also important to compare the results from the R=0 fatigue to the “pre-strained” fatigue tests as we see drastically different results. For the R=0 fatigue we saw electrical failure at $N>100$, a full order of magnitude earlier than with these “pre-strained” samples. Part of this can be attributed to the difference in R value, and that greater strain energy was input to the samples with R=0. However, another factor can also be influencing these results. Since
these stacked conductors are held together simply by the conduit and a light amount of solder, slippage between tapes during fatigue testing is possible. For the fatigue loading curves with $R=0$, the fatigue machine returns to a value of $\varepsilon=0.0\%$ at the end of each cycle. If the tapes had slipped during loading, when the machine returns to what it knows as $\varepsilon=0.0\%$, some of the tapes may now be under compression and this could cause buckling and additional damage to the REBCO ceramic layer.

**Comparing Single Tape to CICC Fatigue**

It is necessary to look at the single tape fatigue and stacked conductor fatigue and compare results to determine how HTS reacts to different conductor configurations. Comparing the single tape to the CICC fatigue with $R=0$, it is clear that the stacked fatigue fails at a much lower number of cycles then the single tape does at the same strain value. It has been thoroughly discussed in this thesis that existing defects and cracks will drastically affect the fatigue life of the ceramic superconducting layer. The compaction of the aluminum CICC
around the tapes is not uniform as shown in the figure below.

Figure 43: Optical image of the CICC post compaction using copper dummy tapes. This was the chosen compaction value that was tested during fatigue, and shows how the aluminum is biting into the tapes.

There are voids, and also areas along the edge of the conductor where the aluminum conduit has clearly caused deformation at the interface between the CICC and the conductor. The compaction process is performed with a 12 sided die, and is not a uniform compaction process, so the compaction process will input more strain on certain areas as opposed to others. Another factor to consider is the inside of the aluminum conduit is not a perfectly polished surface, it is rough and has burs on the interior walls. All of these factors increase the likelihood that the YBCO conductor, which already had edge defects from the slitting process, was further damaged from the compaction process and resulted in a conductor with a higher concentration of defects than the single tape. During the fatigue process, the tape stack is gripped and the aluminum conduit is at rest. This results in the tape stack sliding inside the rough aluminum conduit and causing more edge damage to the YBCO conductor.
Taking into account all of these factors, it is understood why the fatigue life of the CICC conductor stack would be lower than that of a single tape.
References


Chapter 5
Conclusions and Future Work

This chapter gives a summary of the work and results performed, as well as recommendations for future work.

Summary
Superconductors are both elements and compounds that have zero resistivity below a critical temperature. A type of high temperature superconductor, REBCO, shows great promise for many applications that are operating at increased temperature and magnetic field. Due to the high cost of these materials, implementation relies on understanding their mechanical properties and ensuring that they will not prematurely fail. During magnet construction, cooldown, and operation the superconducting material sees many stresses that must be accounted for. Single cycle stress/strain studies have been investigated, but literature has lacked an understanding for high cycle fatigue.

This work has investigated how REBCO behaves when subjected to tensile fatigue loading at different strain values of 0.35%, 0.45% and 0.5%. The samples were fatigued, and then their critical current was measured to check for reduction in transport. The results showed that for SuperPower YBCO tapes with a Hastelloy substrate, as-received $I_c$ was retained after $\varepsilon = 0.35\%$ and $\varepsilon = 0.45\%$ at 10,000 cycles, and $\varepsilon = 0.50\%$ at 100 cycles. SuNAM GDBCO conductors retained their as-received $I_c$ after $\varepsilon = 0.35\%$ and $\varepsilon = 0.45\%$ after 10,000 cycles,
and $\varepsilon = 0.50\%$ after 1000 cycles. In both conductors it was shown that n-value decreases with increasing cycles, and shows the onset of fatigue damage. The main cause of fatigue failure in the composite conductors is due to the slitting process used to reduce the conductor width from 12 mm down to the customers desired width. This process introduces microcracking and therefore stress concentrators that grow in length with increasing number of cycles. Increases in crack length correlate to a reduction in critical current. In order to improve fatigue life in these conductors, it is important to remove or reduce the edge defects due to manufacturing processes. Careful consideration to the substrate must also be given, understanding the mechanical properties of the substrate material will have a profound impact on the fatigue properties of the composite system. Support structures must also be considered in order to reduce strains placed on the superconducting material during operation. Remaining below 0.35% strain will give the greatest fatigue life.

Packaging the conductor is another important consideration, as it is known that edge defects have a large influence on mechanical properties of ceramics. In order to understand the effect of packaging a stack of conductors, fatigue loading was performed on stacked conductors at varying strain values, as well as fatigue ratios. The data shows that the conductor that is packaged shows less resistance to fatigue loadings than the single tape. Due to the compaction process stressing the REBCO tape edge, and the jagged nature of the inside of the conduit, it is understood why this packaging has a detrimental effect on the conductor’s fatigue life. By removing and reducing the roughness of the conduit, as well as
performing a uniform compaction around the conductor, conduit packaged REBCO conductors would see improved fatigue life.

**Recommended Future Work**

It has been shown that REBCO conductors have high mechanical strength under a tensile stress, which allows for various applications of these superconductors. In recent years, conduction cooled magnets utilizing REBCO have been manufactured. If quench occurs, and a thermal runaway occurs, the magnet will be burnt and rendered useless. To avoid this, the coil is impregnated with epoxy resin and helps to form cooling paths from the inner turns of the magnet to the outermost turns. It is imperative that the coil windings are not damaged, because this damaged area would generate local heat and in the worst case, cause a thermal runaway situation. Degradation of REBCO epoxy impregnated coils have been documented in literature.

An epoxy impregnated coil is typically constructed using a REBCO conductor, an insulating tape such as Kapton, and epoxy resin so that upon cooldown, the mismatch in thermal expansion generates a radial tensile stress acting transverse to the conductor length. This stress could possibly damage the coil due to the low delamination strength of the REBCO conductor. Therefore, it is important to understand the delamination strengths of various REBCO conductors to limit the transverse tensile stress during magnet construction and cooldown.
The delamination strength of REBCO is also of interest due to the role interlayer adhesion plays in fatigue failure. If delamination is seen during high cycle fatigue, it is possible that localized delamination of the REBCO layer could lead to crack growth at the site of delamination. Delamination experiments will be performed on both SuperPower and SuNAM 4 mm tapes, results compared and discussed as it pertains to their fatigue life.

By performing delamination experiments and understanding the differences in adhesion strength, more information will be understood about the mechanical properties of this material system. Delamination measurements have been attempted and are underway.

A second study that would be of interest is the investigation into fatigue of joint regions. Due to the manufacturing limits of the commercial suppliers, magnets currently have splices and joints that electrically and mechanically bond the superconducting conductors. While efforts to create a superconducting joint are underway, the mechanical properties of these splices must also be well understood. Investigating the different types of joints for fatigue failure, and determining the most robust joint architecture would be of great value to HTS magnet engineers.