Investigation of the Delamination of (RE)BaCuO High-Temperature Superconductor Tapes Subjected to Mechanical Stress

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Thesis to obtain the Master Degree in Electrical and Computer Engineering

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Declaration

I declare that this document is an original work of my own authorship and that it fulfills all the requirements of the Code of Conduct and Good Practices of the Universidade de Lisboa.
The research hereby presented was developed in collaboration with Karlsruhe Institute of Technology.
Acknowledgements

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**Abstract**

Rare earth-based high-temperature superconductor (HTS) tapes are a very attractive solution for several applications working with high currents and high magnetic fields. When the tapes are subjected to external forces, especially in magnet applications, they tend to delaminate. Nowadays, the conditions under which and the location in the tape where delamination happens are not fully clear. The study of the delamination has an important role for the design of mechanically stable HTS coils. Understanding the limits of delamination and the reasons that caused the delamination will help the suppliers to improve the mechanical properties of the tape, and the performance of the future applications.

This thesis is focused on the Investigation of the Delamination of (RE)BaCuO (REBCO) HTS tapes subjected to mechanical stress under the superconducting state. In this context, this investigation had two main goals: (i) a set-up had to be built to test the REBCO tapes, by upgrading an initial test tool developed at Karlsruhe Institute of Technology (KIT) and, (ii) application of mechanical stress gradually on the samples until the delamination stress. During this process the sample was in the superconducting state, and the tape’s critical current behavior was registered.

In the experiments was observed that in approximately 90 % of the samples, the critical current did not changed, throughout the gradual mechanical stress applied until the sample’s delamination. The variation of the load gradient of the mechanical stress applied did not provided any correlated impact on the delamination stress of each sample.

**Keywords:** Superconductor, REBCO, Delamination, Mechanical Stress, Critical Current.
Resumo

As fitas de supercondutores de altas temperaturas baseadas em terras raras são uma solução muito atraente para várias aplicações que funcionam com altas correntes e elevados campos magnéticos. Quando as fitas são sujeitas a forças externas, especialmente em aplicações de ímães, elas tendem a delaminar. Atualmente, as condições sob as quais, e a localização na fita, onde ocorre a delaminação não são totalmente claras. O estudo da delaminação tem um papel importante no projeto de bobinas HTS. Compreender os limites da delaminação e os motivos que a causaram, ajudará os fornecedores a melhorar as propriedades mecânicas da fita e o desempenho das aplicações futuras.

Esta tese está focada na investigação da delaminação de fitas (RE) BaCuO (REBCO) HTS sujeitas a força mecânica, sob o estado supercondução. Nesse contexto, esta investigação teve dois objetivos principais: (i) era necessário construir uma máquina para testar as fitas REBCO, que se baseou num melhoramento de uma ferramenta de teste inicial desenvolvida no Karlsruhe Institute of Tecnology (KIT) e (ii) aplicar a força mecânica gradual às amostras até que estas delaminassem. Durante esse processo, a amostra esteve no estado de supercondução e o comportamento da corrente crítica da fita foi registado.

Nas experiências, observou-se que em aproximadamente 90% das amostras, a corrente crítica não mudou ao longo da força mecânica aplicada gradualmente até a delaminação da amostra. A variação do gradiente de carga da força mecânica aplicada não proporcionou impacto correlacionado com a força mecânica máxima aplicada a cada amostra, força aplicada na qual a amostra delaminou.

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<th>Definition</th>
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<tbody>
<tr>
<td>( \Phi )</td>
<td>Magnetic Flux</td>
</tr>
<tr>
<td>( \Phi_0 )</td>
<td>Critical Magnetic Flux</td>
</tr>
<tr>
<td>CC</td>
<td>Coated Conductor</td>
</tr>
<tr>
<td>E</td>
<td>Electric Field</td>
</tr>
<tr>
<td>( E_c )</td>
<td>Critical Electric Field</td>
</tr>
<tr>
<td>H</td>
<td>Magnetic Field</td>
</tr>
<tr>
<td>( H_c )</td>
<td>Critical Magnetic Field</td>
</tr>
<tr>
<td>HTS</td>
<td>High-Temperature Superconductor</td>
</tr>
<tr>
<td>I</td>
<td>Current</td>
</tr>
<tr>
<td>( I_c )</td>
<td>Critical Current</td>
</tr>
<tr>
<td>IBAD</td>
<td>Ion-Beam-Assisted Deposition</td>
</tr>
<tr>
<td>J</td>
<td>Current Density</td>
</tr>
<tr>
<td>( J_0 )</td>
<td>Critical Current Density</td>
</tr>
<tr>
<td>LN_2</td>
<td>Liquid Nitrogen</td>
</tr>
<tr>
<td>LTS</td>
<td>Low-Temperature Superconductor</td>
</tr>
<tr>
<td>MOD</td>
<td>Metal Organic Deposition</td>
</tr>
<tr>
<td>MOCVD</td>
<td>Metal Organic Chemical Vapor Deposition</td>
</tr>
<tr>
<td>MPa</td>
<td>Megapascal</td>
</tr>
<tr>
<td>PIT</td>
<td>Powder in Tube</td>
</tr>
<tr>
<td>PLD</td>
<td>Pulsed Laser Deposition</td>
</tr>
<tr>
<td>RABITS</td>
<td>Rolling-Assisted Biaxially Textured Substrate</td>
</tr>
<tr>
<td>RCE</td>
<td>Reactive Co-Evaporation</td>
</tr>
<tr>
<td>SCS</td>
<td>Surrounded Copper Stabilizer</td>
</tr>
<tr>
<td>t</td>
<td>Time</td>
</tr>
<tr>
<td>T</td>
<td>Temperature</td>
</tr>
<tr>
<td>( T_c )</td>
<td>Critical Temperature</td>
</tr>
<tr>
<td>U</td>
<td>Electric Potential</td>
</tr>
<tr>
<td>YSZ</td>
<td>Yttrium-Stabilized Zirconia</td>
</tr>
</tbody>
</table>
1 Introduction

1.1 Motivation

Superconductivity is a phenomenon in which a superconductor material becomes zero electrical resistant at low temperatures. Since its discovery, by Heike Kamerlingh Onnes in 1911, scientists worldwide have increased the research in this field, due to its importance. The latest results lead to five Nobel prizes related to this subject [1].

Superconductors can be divided in two groups: Low-Temperature Superconductors (LTS) and High-Temperature Superconductors (HTS), depending on the critical temperature below whose materials become superconducting. HTS Coated Conductors became an interesting research topic due to their excellent parameters, i.e. their high critical temperatures, as well as high current densities in magnetic fields. These properties make HTS Coated Conductors a potential material for many applications, such as, Nuclear Magnetic Resonance (NMR) magnets [2], accelerator magnets (e.g. accelerator magnets developed at CERN [3]), fusion magnets [4], power cables [5], or energy storage systems [6].

Rare Earth-based High-temperature Superconductor tapes, an HTS tape is a very attractive solution for several applications working with high currents and high magnetic fields, namely solenoidal and toroidal coils. In this context, stands out REBCO, a rare Earth-based High-temperature Superconductor and commonly used due to their potential to sustain stronger magnetic fields than other superconductors materials. These tapes need to be synthesize as flat conductors, which become more sensitive to magnetic fields perpendicular to the broad face, compared to the narrow face. Minimizing exposure to this particular magnetic field vector is therefore a key part of the HTS coil design [7]. When those tapes are subjected to external repulsive and attractive forces, especially in magnet applications, they tend to delaminate.

Nowadays, there are several mechanical issues in HTS Coated Conductors that need to be faced, and the conditions that lead to the delamination are not yet fully clear. Factors as superconductor supplier and coated conductor batch can have impact on the results, however no clear patterns have emerged. Delamination is caused when certain levels of stress are applied to the superconductor material. The different ways of applying stress can be classified as axial, transverse tensile, transverse compressive, shear, cleavage, or peel stress.

In this thesis, to better understand the causes of delamination phenomenon, REBCO samples from SuperPower supplier will be tested with transverse stress applied on the tape. The samples will be tested mechanically until the delamination stress. Delamination stress was assumed to be the pressure applied to the sample that triggered the delamination. This study had the novelty that during this process, the measurement of the tape’s critical current was done. Delamination knowledge will help suppliers to...
improve the mechanical properties of the tape, and will allow users to choose the proper conductor for a given application.

1.2 Objectives

The main goal of this study is to evaluate the electro-mechanical effects of the REBCO tapes when subjected to mechanical stress until its delamination. In this context this thesis has two main objectives:

First, in order to become possible the study of delamination, a set-up had to be built to test the REBCO tapes, by upgrading an initial test tool developed at Karlsruhe Institute of Technology (KIT) by G. Jakubiec [8], previous master thesis student. Basically, the set-up supported only mechanical tests. In this work, as not only mechanical measurements are required, but also electrical ones, to determine the critical current values, this upgrade provided the possibility of performing critical current measurements for a given mechanical stress.

Secondly, to evaluate REBCO coated conductor tapes from SuperPower manufacturer [9] by submersing the sample in liquid nitrogen at 77 K, and subject the sample to mechanical stress. Stress will be applied gradually until the delamination stress. During this process the tape’s critical current behavior will be registered. This allows determining the precise conditions under which the degradation of the transport properties of the tape occurs. With this data, a statistical analysis will be performed.

1.3 Thesis Outline

The chapters of this document are organized as follows:

In Chapter 2, the rare earth-based coated conductor tapes, introducing the concepts of superconductivity, (RE)BaCuO coated conductors, problem of delamination are presented and, at last, the state of the art of the methods and results of delamination investigation.

In Chapter 3, the experimental technique for delamination tests, including experimental preparation and test procedure are presented.

In Chapter 4, the results and discussion are presented, where the delamination strength and critical current values obtained are shown, and the delamination strengths and critical current under strength of each sample are compared.

In Chapter 5, the conclusions of the dissertation are summarily discussed and recommendations are given to future developments concerning the concepts approached in this work.
2 Rare Earth-Based Coated Conductor Tapes

2.1 Introduction to Superconductivity

Superconductivity is a state of the matter characterized by a state of a weak attractive interaction between electrons, resulting in a zero electrical resistance [10]. This state can occur for many elements of the periodic table, in certain materials called superconductors. This weak interaction between electrons causes a long-range phase motion of electrons which triggers the perfect conductivity. Beyond the zero-resistance hallmark, ideal superconductors are characterized by a perfect and reversible diamagnetism [11]. This behaviour is called Meissner state, which consists on the expulsion of any magnetic flux from a superconductor during its transition to the superconducting state when it is cooled below the critical temperature.

These special features of the superconducting state are possible until an external input of energy (thermal, magnetic or kinetic) overcome to break down the phase equilibrium. Once the critical surface defined, by temperature, magnetic field and current density, is broken, the material loses the superconductivity. The critical surface mentioned is represented in the following figure.

Figure 2-1 Representation of Meissner effect [12].
There are two main groups of superconductors: The type-I superconductors, which lose their superconducting properties in relatively weak magnetic fields, and the type-II superconductors, usually alloys, that withstand very strong magnetic fields before losing their superconducting properties [14].

Type-I superconductors are generally pure metals and reach the superconducting state only under pressure. The Figure 2-3 shows Mendeleev table with marked elements which are superconductors or not.

Figure 2-3 Mendeleev table with marked elements which are superconducting [15].
In Type-I superconductors the breakdown from Meissner state, the superconducting state to Normal state is abrupt, and occurs when the applied magnetic field is higher than the critical magnetic field, $H_c$ [16]. This critical magnetic field depends on the Temperature, which has always to stay lower than the critical Temperature, $T_c$, as shown in the figure below.

![Diagram](image)

**Figure 2-4** Applied Magnetic field vs Temperature diagram for Type-I Superconductor [17].

As it was mentioned, this type of superconductors has very low critical magnetic fields, so they are not very good for applications.

Type-II superconductors are usually compounds and metals, such as, elemental vanadium, technetium, and niobium, for which Meissner state exists only below the critical magnetic field $H_{c1}$. Above this value and until the critical magnetic field $H_{c2}$, a flux pinning phenomenon occurs in which the magnetic flux starts to penetrate the material. This state is called Mixed state or, also called, Vortex state, which is characterized by the nucleation of normal metallic filaments in the superconductor, each one carrying a quantized magnetic flux $\Phi_0$.

$$\Phi_0 = \frac{h}{2e} \approx 2.07 \times 10^{-15} \text{Wb}$$  \hspace{1cm} (2.1)

When the applied magnetic field reaches values higher than the critical magnetic field $H_{c2}$, the superconductor returns to the Normal state, which is no longer superconducting. $H_{c2}$ is reached when the whole material is covered in vortices, i.e. when the material is completely transparent to the external field.
The following figure shows a sketch of the flux quantization phenomenon. The areas penetrated by the magnetic flux are not superconducting.

In the Mixed state, the superconductor is penetrated by magnetic field in form of super-current vortices. The number of vortices and their distribution in the sample are determined by the mutual repulsion among the vortices and the magnitude of the applied field, and an Abrikosov lattice is formed as shown in the following figure.
Figure 2-7 Surface of a superconducting alloy that had a magnetic field applied perpendicular to the surface [19].

Figure 2-7 shows small ferromagnetic particles applied to the surface of a superconductor, which is in the vortex state with magnetic field applied perpendicular to the surface. When applied current, the dark regions are normal, and the light regions superconducting. Pinning centers of vortices do not move and they are pushed in the perpendicular direction to electrical current and magnetic field, due to Lorentz force [17]. Pinning forces are acting on these vortices, which are fixed in the different kinds of the defects. Current density in copper conductor is typically 1 – 5 A/mm² with losses (DC), and in superconductors it can be 100 to even 10,000 A/mm² [20] without losses (DC). The high current density allows superconductors to have many applications, such as power cables or high magnetic field coils.

LTS refer to alloys based on Nb, such as, NbTi and Nb₃Sn. NbTi can be manufactured with low cost, around 1 €/kAm [21], and has become the dominant commercial superconductor. These superconductors have ductile form. NbTi is used for field applications below 10 T. Multifilamentary composites of Nb₃Sn are used to produce superconducting magnets with field strength above 10 T, up to 21 T. However, they are very brittle and sensitive to bending and tensile stresses. All these conductors have to be cooled down by liquid He-I, that reaches temperatures about 4 K, or even superfluid He-II, that reaches 1.8 K, temperatures below the superconductors critical temperature [22].

HTS are oxide superconductors with perovskite crystal structure. The first high-temperature superconductor was discovered by Bednorz and Muller, in 1986, composed by La_{2-x}Ba_xCuO_4 with a transition temperature of about 35 K [23]. A substitution of Yttrium for Lanthanum led to the discovery of YBa_2Cu_3O_{6+d} (d ≤ 1) often referred to YBCO or Y-123 because of the ratio of Y: Ba: Cu in this material. This was the first superconductor discovered with a Tc of 90 K, well above the boiling point of liquid nitrogen, 77 K. In 1988 Bismuth based superconductors (Bi_2Sr_2Ca_2Cu_3O_{10+d}) were discovered with an even higher Tc.
The BSCCO family of superconductors has also a perovskite type structure with copper oxide planes and chains and orthogonal unit cells. These are known as 1st generation tapes. Two common members are Bi-2212 or 85 K phase and mostly Bi-2223 or 110 K phase [24]. Long lengths of Bi-2212 wires are possible to be drawn with a surface coating method whereas the powder–in–tube (PIT) method is applicable for Bi-2223 tapes [25]. Despite the good performance PIT BSCCO requires high amount of silver and manufacturing process makes this HTS expensive for industrial applications. Although, the BSCCO based tapes and conductors are used to demonstrate a variety of HTS power devices including power cables, fault current limiters, motors and generators. More recently, large progress in Yttrium-based surface coated HTS conductors (YBCO) has been achieved. This conductors are known as 2nd generation tapes (2G HTS) offering operation at high magnetic fields close to 77 K and in a way for manufacturing next generation HTS electric power devices and components which makes them one of the most promising superconductors for many applications [26]. However, YBCO (2G HTS) production is much more complex than BSCCO (first generation conductors). The Table 2-1 shows the summary of the LTS and HTS mentioned with the respective characteristics values.

<table>
<thead>
<tr>
<th>Material</th>
<th>( T_c ) (in K) at ( B = 0 )</th>
<th>( B_{c2} ) (in T)</th>
</tr>
</thead>
<tbody>
<tr>
<td>LTS</td>
<td></td>
<td></td>
</tr>
<tr>
<td>NbTi</td>
<td>9.6</td>
<td>18 (0 K) 10 (4.2 K) [27]</td>
</tr>
<tr>
<td>Nb(_3)Sn</td>
<td>18</td>
<td>28 - 30 (0 K) 22 (4.2 K) [27]</td>
</tr>
<tr>
<td>HTS</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Bi-2212 85 ((\text{BiSr}_{2}\text{CaCu}_2\text{O}_y)) ((y = 8 - 10))</td>
<td>85</td>
<td>&gt; 100 (4.2 K)</td>
</tr>
<tr>
<td>Bi-2223 ((\text{BiPb}_{2}\text{SrCa}_2\text{Cu}_x\text{O}_y)) ((y = 8 - 10))</td>
<td>90</td>
<td>&gt; 100 (4.2 K) &gt; 0.5 (77 K)</td>
</tr>
<tr>
<td>YBCO ((\text{YBa}_2\text{Cu}<em>3\text{O}</em>{7-x}))</td>
<td>110</td>
<td>8 – 9 ( 77 K)</td>
</tr>
</tbody>
</table>

Table 2-1 Comparison of characteristics between some LTS and HTS [28]
Figure 2.8 Engineering Critical Current Density vs. Applied Field [29]
Since different rare earths can be used instead of Yttrium, the coated conductors are also known as REBCO coated conductors, where RE stands for Rare Earth. As shown in Figure 2-8, REBCO tapes have the highest critical current density what, besides very high critical temperature, makes them very attractive. This thesis will be focused on REBCO Coated Conductors, and this type of High Temperature Superconductors will be discussed with details in the subsection 2.2 (RE)BaCuO Coated Conductors.

2.2 (RE)BaCuO Coated Conductors

The discovery of HTS superconductors in 1986 was determinant for power applications. Powder in Tube (PIT) was the first successful technique to produce HTS wires and represents the first generation of HTS tapes [30]. This technique was used to produce conductors from brittle superconducting materials barium, copper, oxygen and compound ceramic cuprate superconductors such as BSCCO and after the process have a plate morphology. Nevertheless, critical current of PIT-wires drops considerably at liquid nitrogen temperatures when exposed to weak external magnetic fields [31]. In order to be used in high-field applications, these wires must be cooled down to temperatures within 20 to 30 K, and the fact of large amount of silver is needed to produce these wires, make these conductors very expensive.

In January of 1987 a research team at the University of Alabama-Huntsville substituted yttrium for lanthanum and achieved an incredible critical temperature of 92 K and overcame the variations on the critical current in presence of external fields [32]. Coated conductors has been thought to use (RE)BCO compounds which (RE) refers to rare-earth elements such as yttrium (Y). Additionally, Nd, Gd, Eu, Sm, Er, Yb, Tb, Dy, Ho, Tm and the like may be used as the rare-earth materials. The most common is YBa$_2$Cu$_3$O$_{7-x}$ (YBCO) [33]. In addition, YBCO is able to carry high currents in strong magnetic fields when cooled by liquid nitrogen [34].

From Figure 2-8 it can be noted that YBCO have better intrinsic flux pinning and irreversibility field than BSCCO. However, YBCO is brittle and highly anisotropic, which implies that electric current does not flow well if the crystals are not perfectly aligned, consequently an epitaxially growth YBCO coating is expected on metallic strips [35].

2G HTS Coated conductors are formed by an assembly of layers and each one has its own role resulting in a less than 100 µm thick conductor. These tapes need to be synthesize as flat conductors, which become more sensitive to magnetic fields perpendicular to the broad face becoming more resistant. A typical coated conductor is constituted by a thick and electrically-resistive substrate, usually made from Hastelloy or Stainless steel, a stack of oxide buffer-layers, a very thin superconducting film (REBCO), approximately 1 µm, and an electrically-conductive metallic stabilizer as shown in Figure 2-9.
For instance, the substrate gives a flexible and smooth mechanical support to the brittle superconductor and also helps thermal stabilization. The buffer layers align the anisotropic HTS and isolate the superconductor from chemically active compounds. The role of the stabilizer is to reduce the heat generation in the superconductor by diverting current once the superconductor becomes normal as well as to reduce the voltage overshoot during quenches. The stabilizer is in contact with the HTS and is usually made of noble metals such as silver or gold in order to avoid chemical reaction with the superconductor. Finally, manufacturers often encapsulate the wires in a copper cladding to reduce the tape overall resistance, while the wire is in the normal state.

There are two leading technologies to align grains in (RE)BCO. The Ion-Beam Assisted Deposition (IBAD) [35, 37] and the Rolling-Assisted Bi-axially Textured Substrate (RABiTS) [38].

Ion-Beam-Assisted Deposition (IBAD) is a technique that generates a buffer layer with preferred texture. The substrate layer used in this technique is made of stainless steel or Hastelloy whose typical thickness are 50 to 100 µm [39]. The substrate buffer stack layer is deposited which the first buffer’s layer is Al2O3, a diffusion-barrier of 100 to 200 nm and an Y2O3 layer of 10 to 20 nm thick [40], deposited by sputtering technique [41]. The successive layer, grown on the Y2O3 layer using RF plasma source, is MgO that assists ion beam. And the last layer is CeO2 or LaMnO3, depending on the manufacturer.

Rolling-Assisted Biaxially Textured Substrate (RABiTS) is the method that produces near-perfect biaxial texture in a range of materials, including metals, and certain oxides deposited epitaxially on a textured metal strip. These textured substrates serve as structural templates for the final superconductor layer, which has substantially fewer weak links. The RABiTS process consists in achieving lamination and recrystallization by texturing nickel or nickel alloys, replacing silver, which makes the HTS wires less expensive [42]. NiW is used instead of pure Ni because of the reduced magnetization of this alloy. RABiTS represents one potential solution to the difficulties associated with the fabrication of practical
long length YBCO wires suitable for practical electrical power applications. NiW substrate are manufactured industrially in the form of flexible strip, typically 75 µm and more than 1 km of length [39]. The first step is to reduce roughness on surface using plasma cleaning. Then the buffer layer is deposited on the substrate, and depending on the manufacturer, it can be Y₂O₃ or CeO₂. Next layer is typically Yttrium-Stabilized Zirconia (YSZ) used as an inter-diffusion of substrate atoms blocker. The top layer of buffer layer, which is usually CeO₂, is used to compensate the lattice mismatch between superconductor and YSZ layer. Buffer layer thickness is typically about 100 nm.

REBCO layer is deposited after buffer layer. There is a plenty of methods used to deposit this layer used by different manufacturers such as Metal Organic Deposition (MOD), Metal Organic Chemical Vapor Deposition (MOCVD), Reactive Co-Evaporation (RCE), or Pulsed Laser Deposition (PLD).

Metal Organic Deposition (MOD) is the method where organic precursors are deposited on the substrate by dip coating [43, 22]. In this method the solution concentration and viscosity is fine-tuned in order to control the coating thickness. After deposition of precursors, carbon-containing materials are being removed by drying and decomposition. To optimize REBCO performance and wrap around from the surroundings, a passive silver layer is deposited on REBCO layer. In this method REBCO thickness achieves 0.8 µm with only a single coating step.

Metal Organic Chemical Vapor Deposition (MOCVD) is also a deposition method where organic precursors are used for rare Earth materials such as Yttrium, or other materials such as Barium or Copper. In this multistep method, the first step is to vaporize the liquid precursors at a constant temperature. Then precursors are carried by gas (argon and oxygen) and injected onto a hot substrate. At the end, REBCO films are deposited within temperature 700 to 800 ºC. With this process the thickness of the REBCO layer is typically around 1 µm [39].

Reactive co-evaporation (RCE) deposition method rare Earth materials such as Yttrium, or other materials such as Barium or Copper are heated to evaporate and be deposited on the substrate. Depending on the manufacturer this method can be used with a single step deposition film (SuNAM), or in a cyclic process of deposition and reaction (STI) [44]. With this process the thickness of the REBCO layer is around 1 µm.

At last, Pulsed Laser Deposition (PLD) is a process with a pulsed ultraviolet laser, typically KrF or XeCl, which irradiates a target with the composition that is desired to form layer of the superconductor. The target is vaporized and deposited on the substrate. Multiple layers are needed to be deposited to provide high critical current [39]. Using this process, the typical thickness of the REBCO film obtained is 0.3 to 0.5 µm.

Each manufacturer uses different techniques, REBCO layer depositions and buffer layers. The following table shows each technique, substrate, buffer layers and HTS type used by each manufacturer.
Table 2-2 Fabrication process and template of industrial coated conductors [39].

Table above shows that the technique most used by the manufacturers to produce the superconductors is IBAD, the substrate is Hastelloy and Gadolinium is the HTS type most used.

### 2.3 Problem of Delamination

REBCO Coated Conductors are characterized by their high critical current, high critical temperature and high critical magnetic field. However HTS Coated Conductors face several mechanical issues, of which the most relevant is the stress that can be caused by radial forces or the forces related to the presence of current and field. Those stresses can provoke delamination in REBCO tapes causing a drop in the critical current. As it was mentioned, REBCO Coated Conductors are multilayer tapes and every layer is able to delaminate, depending on the type of stress applied on the tape. There are three types of delamination in a tape: A cohesive delamination, which means the delamination in only one layer; The adhesive delamination, corresponds to a delamination at the interface between two layers; a mixed mode delamination that refers to a delamination crossing more than one layer [45].

![Diagram of REBCO Coated Conductor](image-url)

Figure 2-10 Transverse cross-section of REBCO Coated Conductor [45].
Depending on the action over the conductor results in different types of stress. Several types of stresses can be distinguished: transverse tensile, transverse compressive, axial, cleavage, shear and peel stress. In the following picture are schematized with the tape side view the different pressures mentioned and the correspondent common stress values.

<table>
<thead>
<tr>
<th>Type of stress</th>
<th>Stress (MPa)</th>
<th>Scheme</th>
</tr>
</thead>
<tbody>
<tr>
<td>Axial</td>
<td>&gt;700</td>
<td><img src="image" alt="Axial Scheme" /></td>
</tr>
<tr>
<td>Transverse</td>
<td>~10 - 100</td>
<td><img src="image" alt="Transverse Scheme" /></td>
</tr>
<tr>
<td>Transverse compressive</td>
<td>&gt;100</td>
<td><img src="image" alt="Transverse Compressive Scheme" /></td>
</tr>
<tr>
<td>Shear</td>
<td>&gt;19</td>
<td><img src="image" alt="Shear Scheme" /></td>
</tr>
<tr>
<td>Cleavage</td>
<td>&lt;1</td>
<td><img src="image" alt="Cleavage Scheme" /></td>
</tr>
<tr>
<td>Peel</td>
<td>&lt;1</td>
<td><img src="image" alt="Peel Scheme" /></td>
</tr>
</tbody>
</table>

Figure 2-11 Types of stress acting on a coated conductor and its values [46]

The figure above shows the types of stress that coated conductors are subjected to and the typical stress values. Axial stress refers to forces acting on coated conductor in axial direction. The limit of this type of stress is more than 700 MPa in case of a 50 µm thick Hastelloy substrate tape. Transverse stress is the type of stress investigated in this thesis and occurs when the force starts to pull the conductor up to the two opposed sides. The limit for transverse tensile stress is from around 10 to 100 MPa [46]. Transverse compressive stress is similar to the transverse stress but the force press on the conductor is in opposite directions and its limit is much higher, more than 100 MPa. Another type of stress verified
in superconducting tapes is the shear stress that occurs when forces pull the top side of the tape in opposite way from the applied force on the bottom side. This type of stress normally happens during the process of cooling down the coated conductor and its common values are above 19 MPa. Cleavage stress happens when opposite external forces act on the coated conductor to open one edge of the adhesive assembly. At last, Peel stress is very similar to the cleavage stress, but here adhered part is flexible. For both types of stresses, cleavage and peel stress, the limit is less than 1 MPa due to the small surface of the adhesive bond that forces are applied. Those type of stresses mentioned can cause delamination in coated conductors.

The type of stress investigated in this thesis was the transverse stress that occurs in solenoidal and toroidal coils. The transverse stress corresponds to the hoop stress as shown in Figure 2-12.

Figure 2-12 Forces and mechanical stress in solenoid (a) and in toroidal coil (b) [46].
2.4 Methods and results of delamination investigation

In order to investigate those different types of stress in Coated Conductors, several methods of delamination investigation have been developed: anvil method, compressive method, solder-pin method, peel method and cleavage method and shear method.

Anvil method was used by D.C. van der Laan et al. [47] Two anvils are soldered to the coated conductor where the stress is applied along the c-axis of the conductor. In this method at least one anvil is connected with a hydraulic actuator which is biaxial causing a proper alignment between the sample and the hydraulic as shown in Figure 2-13.

Figure 2-13 Anvils soldered to the sample and the biaxial top anvil [47].
Gorospe et al. used the same method building it in with possibility of current measurements [37]. This set is connected with lever which pulls up the top anvil and transverse stress is applied to the sample. This is a similar method compared to the method used in this this, however it was used to measure the effects of low cyclic loading on critical current and bending deformation.

Figure 2-14 Set of anvil method with critical current measurement [37].
Tomoaki Takao et al. used a transverse compressive method to characterize YBCO Coated Conductors [48]. The set is similar to the anvils method presented previously to test the tape to transverse stress. In this method, the forces acting on the anvils takes opposite directions, the tape is compressed. The set is shown in the Figure 2-15.

![Figure 2-15 Set of the compression method [48].](image)

Solder-pin method has the same principle of the anvil method, however solder-pin method, copper rod is soldered directly to the sample with a cross-section of 1 mm x 1 mm instead of being soldered to the anvil. The following figure shows the method set by Y. Xie et al. which lets investigate delamination in small areas of the coated conductors due to the small area soldered [49].

![Figure 2-16 Solder-pin method set-up [49].](image)
Shear method is shown in the Figure 2-17 which represents the method set made by Liyuan Liu et al.. In shear method the superconducting sample is soldered to a right and a left anvil and the mechanical load drags the tape's upper surface causing the shear stress.

Figure 2-17 Shear method set-up [50].
Cleavage method is shown by Y. Yanagisawa et al. [51]. This method is similar to the Anvil method namely two anvils are soldered on the top and bottom surface of the sample. However, the force applied does not act on the anvil’s central point but to the right edge of the conductor causing a cleavage stress. Figure 2-18 shows the sample with the anvils soldered and the action of force as well.

![Cleavage method set-up](image)

Figure 2-18 Cleavage method set-up [51]

The cleavage force can be controlled by a pneumatic system installed on the top anvil soldered to the sample. Cleavage force can be measured by the load cell installed on the pulling lever shown in Figure 2-19.

![Cleavage method set-up](image)

Figure 2-19 Cleavage method set-up [51].
Peel method was used by S. Otten [52] and is shown in Figure 2-20. In this method the sample is fixed on both ends in rotatable parts. By turning a rod, the angle between the two parts can be changed from 0 to 180°. The sample is bent and simultaneously the critical current is measured.

Figure 2-20 Bending machine for single tapes at an intermediate angle [52].
The HTS coated conductors delamination using the anvil method has been investigated by researchers from all over the world. The following tables show the state of the art regarding those experiments. Each table is split in columns with the information relative to the Author of the research, the tape, the size of the anvils’ surface soldered to the sample and the average results for mechanical tests or electro-mechanical tests.

<table>
<thead>
<tr>
<th>Author</th>
<th>Production method</th>
<th>Characteristics</th>
<th>Manufacturer</th>
<th>Average mechanical delamination strength [MPa]</th>
<th>Average electro-mechanical delamination strength causing a drop on critical current [MPa]</th>
</tr>
</thead>
<tbody>
<tr>
<td>D.C van der Laan et al. [47]</td>
<td>MOD/RABITS</td>
<td>Ag\textsuperscript{Y}BCO (0.8 µm) / Buffer layer / NiW (75 µm), Stabilizer: No, Width: 10 mm, Slitting: No</td>
<td>Excluding edges: 14.3, Including edges: 13.7</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Ag\textsuperscript{Y}BCO (0.8 µm) / Buffer layer / NiW (75 µm), Stabilizer: No, Width: 10 mm, Slitting: Yes</td>
<td>Excluding edges: 26.5, Including edges: 17.3</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Ag\textsuperscript{Y}BCO (0.8 µm) / Buffer layer / NiW (75 µm), Stabilizer: No, Width: 4 mm, Slitting: Yes</td>
<td>AMSC 4 x 10</td>
<td>17.3</td>
<td>10.8</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Ag\textsuperscript{Y}BCO (0.8 µm) / Buffer layer / NiW (75 µm), Stabilizer: Solder laminated Cu (50 µm, Width: 4.4 mm, Slitting: Yes</td>
<td>AMSC 4 x 10</td>
<td>&gt;28</td>
<td>25</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Ag\textsuperscript{Y}BCO (0.8 µm) / Buffer layer / NiW (75 µm), Stabilizer: No, Width: 4 mm, Slitting: Yes (After reaction)</td>
<td></td>
<td>&gt;18</td>
<td>-</td>
</tr>
</tbody>
</table>

Table 2-3 Delamination results from DC van der Laan et al.
<table>
<thead>
<tr>
<th>Author</th>
<th>Production method</th>
<th>Tape Characteristics</th>
<th>Manufacturer</th>
<th>Anvil Dimensions [mm x mm]</th>
<th>Average mechanical delamination strength [MPa]</th>
<th>Average electro-mechanical delamination strength causing a drop on critical current [MPa]</th>
</tr>
</thead>
<tbody>
<tr>
<td>H. Jeong et al. [53]</td>
<td>MOCVD/IBAD</td>
<td>Ag/REBCO/ Buffer layer / Substrate Stabilizer: Plated Cu (2 x 20 µm), Width: 4 mm, Thickness: 0.1 mm</td>
<td>-</td>
<td>35.5</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td></td>
<td>EDDC/IBAD</td>
<td>Ag/REBCO/ Buffer layer / Substrate Stabilizer: Plated Cu (2 x 20 µm), Width: 4 mm, Thickness: 0.1 mm</td>
<td>4 x 4</td>
<td>36.3</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td></td>
<td>RCE/IBAD</td>
<td>Ag/REBCO/ Buffer layer / Substrate Stabilizer: Plated Cu (2 x 20 µm), Width: 4 mm, Thickness: 0.1 mm</td>
<td>-</td>
<td>31.3</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Ag/REBCO/ Buffer layer / Substrate Stabilizer: Plated Cu + Bass laminated, Width: 4 mm, Thickness: 0.16 mm</td>
<td>-</td>
<td>27.3</td>
<td>-</td>
<td></td>
</tr>
</tbody>
</table>

Table 2-4 Delamination results from H Jeong et al.
<table>
<thead>
<tr>
<th>Author</th>
<th>Production method</th>
<th>Tape Characteristics</th>
<th>Manufacturer</th>
<th>Dimensions (mm x mm)</th>
<th>Average mechanical delamination strength [MPa]</th>
<th>Average electro-mechanical delamination strength causing a drop on critical current [MPa]</th>
</tr>
</thead>
<tbody>
<tr>
<td>A. Gorospe et al. [37]</td>
<td>MOCVD/IBAD</td>
<td>Ag/GdBCO/Buffer layer / Hastelloy Stabilizer: Electroplated Cu, Dimensions (t x w): 0.089 x 4.04 mm</td>
<td>Superpower</td>
<td>3 x 8</td>
<td>35.5</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>4.5 x 8</td>
<td>36.3</td>
<td>-</td>
</tr>
<tr>
<td>RCE-DR/IBAD</td>
<td></td>
<td>Ag/GdBCO/Buffer layer / Stainless steel Stabilizer: Electroplated Cu, Dimensions (t x w): 0.137 x 4.08 mm</td>
<td>SuNAM</td>
<td>3 x 8</td>
<td>31.3</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>4.5 x 8</td>
<td>27.3</td>
<td>-</td>
</tr>
<tr>
<td>A. Gorospe et al. [54]</td>
<td>MOCVD/IBAD</td>
<td>Ag/GdBCO/IBAD/Hastelloy Stabilizer: Electroplated Cu (20 µm), Dimensions (t x w): 0.091 x 3.95 mm Ic: 116 A (77 K, under self-field)</td>
<td>Superpower</td>
<td>4 x 8</td>
<td>33.2</td>
<td>20</td>
</tr>
<tr>
<td></td>
<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td>RCE-DR/IBAD</td>
<td></td>
<td>Ag/GdBCO/IBAD/Hastelloy Stabilizer: Electroplated Cu (20 µm), Dimensions (t x w): 0.092 x 4.01 mm Ic: 170 A (77 K, under self-field)</td>
<td>SuNAM</td>
<td>4 x 8</td>
<td>17.2</td>
<td>13</td>
</tr>
<tr>
<td>Author</td>
<td>Production method</td>
<td>Tape</td>
<td>Anvil Dimensions [mm x mm]</td>
<td>Average mechanical delamination strength [MPa]</td>
<td>Average electro-mechanical delamination strength causing a drop on critical current [MPa]</td>
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<tr>
<td>A. Gorospe et al. [55]</td>
<td>RCE-DR/IBAD</td>
<td>Ag/ GdBBCO/ Buffer layer / Stainless steel Stabilizer: Electroplated Cu, Dimensions (t x w): 0.135 x 10.03 mm Ic: 600 A (77 K under self-field)</td>
<td>4 x 8</td>
<td>95.6</td>
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<td></td>
<td></td>
<td>Ag/ GdBBCO/ Buffer layer / Stainless steel Stabilizer: Electroplated Cu + bass, Dimensions (t x w): 0.235 x 12.49 mm Ic: 600 A (77 K under self-field)</td>
<td>4 x 8</td>
<td>67.8</td>
<td>-</td>
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<tr>
<td>HS Shin et al. [56]</td>
<td>RCE-DR/IBAD</td>
<td>Ag/GdBBCO/ Buffer layer / Stainless steel, Stabilizer: Cu, Dimensions (t x w): 0.132 x 4.08 mm</td>
<td>2 x 8</td>
<td>71.6</td>
<td>-</td>
<td></td>
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<tr>
<td></td>
<td></td>
<td></td>
<td>3 x 8</td>
<td>69</td>
<td>105</td>
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<td></td>
<td></td>
<td></td>
<td>3.5 x 8</td>
<td>74</td>
<td>-</td>
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<td>4.5 x 8</td>
<td>25</td>
<td>15.5</td>
<td></td>
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<tr>
<td>HS Shin et al. [57]</td>
<td>RCE-DR/IBAD</td>
<td>Ag/GdBBCO/ Buffer layer / Stainless steel, Stabilizer: removed, Dimensions (t x w): 0.132 x 4.08 mm</td>
<td>2 x 8</td>
<td>86.5</td>
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<td></td>
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<td></td>
<td>3.5 x 8</td>
<td>72</td>
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<td></td>
<td>4 x 8</td>
<td>14</td>
<td>-</td>
<td></td>
</tr>
</tbody>
</table>

Table 2-5 Delamination results from A. Gorospe et al.

Table 2-6 Delamination results from HS Shin et al.
<table>
<thead>
<tr>
<th>Author</th>
<th>Tape Characteristics</th>
<th>Manufacturer</th>
<th>Anvil Dimensions [mm x mm]</th>
<th>Average mechanical delamination strength [MPa]</th>
<th>Average electro-mechanical delamination strength causing a drop on critical current [MPa]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Jakubiek et al. [8]</td>
<td>Ag/REBCO/ Buffer layer / Substrate Stabilizer: Plated Cu (2 x 50 µm), Width: 12 mm, Thickness: 0.1 mm</td>
<td>Superpower</td>
<td>4 x 4</td>
<td>22.67</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>6 x 4</td>
<td>19.57</td>
<td>-</td>
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<td></td>
<td></td>
<td>13 x 2</td>
<td>13.4</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>Ag/GdBCO/ Buffer layer / Hastelloy Width: 12 mm</td>
<td>SuperOx</td>
<td>4 x 4</td>
<td>25.91</td>
<td>-</td>
</tr>
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<td></td>
<td></td>
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<td>13 x 2</td>
<td>31.9</td>
<td>-</td>
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<tr>
<td></td>
<td>Ag/REBCO/ Buffer layer / Hastelloy</td>
<td>SuNAM</td>
<td>4 x 4</td>
<td>53.4</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>6 x 4</td>
<td>20.06</td>
<td>-</td>
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<td></td>
<td></td>
<td>13 x 2</td>
<td>34.99</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>Ag/GdBCO/ Buffer layer / Hastelloy Width: 12.25 mm Thickness: 0.15 mm</td>
<td>Theva</td>
<td>4 x 4</td>
<td>26.14</td>
<td>-</td>
</tr>
<tr>
<td></td>
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<td>6 x 4</td>
<td>14.44</td>
<td>-</td>
</tr>
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<td>13 x 2</td>
<td>15.58</td>
<td>-</td>
</tr>
</tbody>
</table>

Table 2-7 Delamination results from Jakubiek et al.
3 Experimental Technique for Delamination Tests

In order to create and control delamination experiment investigating the layers’ separation of (RE)BaCuO HTS tapes subjected to mechanical stress, a special experimental set-up was required. In order to investigate the HTS tape’s critical current deterioration during delamination, the initial set-up was used from the previous KIT-ITEP master student [8]. This set-up allowed applying a perpendicular and progressive tensile stress on the sample and identifying the delamination point from the mechanical point of view, that is, when tape layers are separated. The work of G. Jakubiec showed a remarkable difference in delamination force between samples and within one sample type, but in his work delamination was not connected to critical current degradation. In order to understand the HTS tape’s delamination more deeply, the experimental set-up was upgraded by adding the possibility of critical current measurements.

The sample is placed inside a styrofoam box filled with liquid nitrogen at 77 K. A high precision mechanical stress machine was selected to apply the accurate force to the sample, as shown in Figure 3-2. A special sample holder was used to support and apply the pulling force on the sample during the experiment, shown in Figure 3-3.

In order to impose the current to the superconducting tape and at the same time measure the voltage, a power supply and a nanovoltmeter were used. The scheme of the experimental set-up is shown in the Figure 3-1.

![Figure 3-1 Scheme of the experiment set-up.](image)
In this chapter the preparation process of the experimental set-up and the experiment procedure will be described.

3.1 Experiment Preparation

As mentioned in the introduction, the experimental set-up from the previous master student work was adapted. This set-up contains two main parts – the mechanical test machine and the sample holder. The mechanical testing machine used for this experiment was the Galdabini Quasar 2.5. The Quasar 2.5 single column machine [58], shown Figure 3-2, is ideal for low-force testing, with ranges from 0.02 to 2500 N. This machine is designed with a high resolution for force and supports a full load testing speeds from 0.0005 to 1,000 mm/minute. For the machine control, the software Graphwork 6 is used.

![Galdabini Quasar 2.5](image)

**Figure 3-2 Galdabini Quasar 2.5 [58].**
The sample holder for the delamination test was designed and built in the framework of a previous master thesis student at KIT [8]. This sample holder consists of an upper and a lower part enabling the placement of the sample and the application of pulling force during the delamination.

![Sample holder](image)

**Figure 3-3 Sample holder (Based on [8]).**

The Figure 3-3 shows the sample holder. The upper part is attached to the Galdabini’s vertical arm which is pulled up applying mechanical stress on the sample. The upper part has a biaxial movement provided by the upper ball with two axes that ensures an adequate degree of freedom to a proper alignment between the sample and the holder avoiding possible tension on the sample.

The lower part is attached to the base of the testing machine. In the Figure 3-3, the parts shown in green colour are made of G10 fiberglass epoxy laminate which has an high strength, and the parts in grey are made of stainless steel due to low thermal expansion in low temperatures. The G10 fiberglass is lighter than stainless steel but in the previous master thesis work [8] revealed not to be enough resistant when the force is applied to the sample, which caused a change of the G10 parts in contact with the sample for stainless steel.
In this thesis, an upgrade of the measurement system by adding current-voltage measurements was done. In order to measure the critical current of the HTS tape within delamination experiment, the sample holder was modified. This is shown in Figure 3-3 - a copper block was mounted to each side of the holder’s lower part. These current leads ensure the connection between the power supply and the sample, one to the positive and the other to the negative pole. The sample holder’s stainless steel parts are electrically isolated from the sample and current leads (shown in yellow in Figure 3-4). As shown in the Figure 3-4, the end-to-end distance of the copper leads is 9.2 cm, which is equal to the sample length.

![Figure 3-4 Top view from sample holder’s lower part.](image)

In order to mount the sample into the sample holder, copper anvils had first to be soldered to the sample enabling the force transmission from the tensile machine to the tape surface. Hence, the first step was to pre-solder the upper and lower sample’s surface where the copper anvils will be soldered. The used solder wire was indium tin S-In52Sn48 with melting temperature of 118 ºC. The soldering wire’s diameter was 1.5 mm. This type of solder was selected because it has a low melting temperature, and this should avoid possible cracks in a such sensitive tape. The soldering temperature used was 125 ºC, value below the maximum recommended temperature for SuperPower wire types with surround copper stabilizer (SCS). The maximum temperature recommended is 200ºC for less than five minutes [59]. Based on the previous master thesis, the pre-solder density had to be defined for each of the sample’s sides. For the sample’s upper surface, it was used a 1.5 cm in length, 5 mm wide soldering wire along the sample’s whole width. The pre-solder for the sample’s bottom side was a 3 cm in length, 10 mm wide soldering wire, also along the sample’s whole width, making both sample’s surfaces covered with the solder.
In order to remove any oxidized metal from the sample’s surface that annihilates its conductivity properties, and improve the wetting characteristics of the liquid solder and consequently an easier adhesion to the sample, a flux was added. The flux chosen for the copper sample’s surface was zinc chloride (ZnCl₂), which reacts with metal oxides [60]. The pre-solder sample is shown in Figure 3-5.

![Figure 3-5 a) Tape’s top surface. b) Tape’s bottom surface.](image)

To pre-solder the sample, a precise area’s measurement was done before, in order to pre-solder the defined amount of solder in each side of the sample. Then the pre-soldered copper anvils are soldered to the tape. The anvils chosen for the experiments have a contact surface with the sample of 6x4 mm² in the upper anvil that is soldered to the tape’s top surface, and a 13x13 mm² soldered to tape’s bottom surface with a larger pre-soldered surface. This lower anvil’s surface soldered to the sample must be larger than the upper anvil’s soldered surface to the sample in order to the lower surface be fixed on the bottom and the pulling force be applied on the top surface. Typical shape tape with both anvils soldered to it is shown in the Figure 3-6 c) e d).
After soldering the copper anvils to the sample, the voltage taps are soldered to the sample, as shown in Figure 3-6 c) The voltage taps distance of 1.9 cm was chosen in order to be the minimum distance to avoid possible noise. When the anvils and voltage taps are soldered to the sample, the sample is inserted into the space between upper and the lower part of the sample holder, and then the copper leads plates screwed.

To supply the tape electric current, an Agilent 6680A [see Append B.1] current source with a capacity of 0-5V/0-875 A was chosen. For the measurement of the voltage a Keysight 34420A [see Append B.2 and B.3] nanovoltmeter was selected to measure the voltage of the sample. Both devices are connected to the computer by the General Purpose Interface Bus (GPIB) of National Instruments, a high-speed interface for communication between instruments and controllers.

To connect the power supply to the copper blocks, copper cables were used. The connection from the power supply to the sample was done by two paired copper cables with 120 mm$^2$ cross-section each to support the high currents transported. Each pair of copper cables is connected to a shorter and copper cable of 120 mm$^2$ inserted in a liquid nitrogen bath reducing its temperature. The shorter cable is more malleable which avoids pressure on the sample.

The whole experiment is placed into a styrofoam box filled with liquid nitrogen at the temperature of 77 K. This styrofoam box has a ceiled bore in the bottom where the sample holder lower part is attached to the testing machine. The box’s dimensions are 30 cm length, 25 cm width and 16 cm height with a 2.5 cm of thickness.
In the Figure 3-7 is shown the final set-up already prepared to accomplish the electro-mechanical experiments proposed in this thesis.

Figure 3-7 a) Front view of the experiment. b) Top view.
3.2 Test Procedure/Technique

The experimental procedure of delamination versus critical current measurement is a result of three methodologies done one by one in order to get precise and reliable results.

In every experiment, the same cooling time before measurement had to be provided. After inserting the superconducting tape into the sample holder and filling the styrofoam box with LN\textsubscript{2}, 10 minutes of cooling down are counted before starting the test, time to the sample reaches 77 K and consequently become superconducting. Due to the cooled down materials from the sample holder having different thermal expansions at low temperature [61] [62] [63] [64], the tape might be subjected to an additional stress. To avoid any measurement uncertainty due to the materials thermal expansion, a pre-force load step of 0.1 N was added before filling the styrofoam box with LN\textsubscript{2} and kept during the sample’s cooling down period. After 10 minutes, a critical current’s pre-measurement is done at the pre-force load step resulting in a typical current versus voltage measurement curve as shown in Figure 3-8. Then the sample is subjected to pulling force and critical current is measured. During the experiment there are two programs running in parallel, Graphwork 6 and LabVIEW. The Graphwork 6, software that came with the mechanical machine, controls to pulling force; LabVIEW is used to measure the taps voltage and control the input current in order to obtain the sample’s critical current.

For technical applications, it is important to introduce a criterion for the definition of the critical current. The voltage-current (U-I) characteristics of superconductors reveal how dissipation occurs as a result of current. To be able to compare different conductors and measurements, the voltage U is translated to the electric field E. It is necessary to define a criterion for the critical current I\textsubscript{c} as the current measured at a certain electrical field, E\textsubscript{c}. The critical current, I\textsubscript{c}, is defined as the current at which the developed electric field - E, reaches a certain critical electrical field E\textsubscript{c} [65]. For example, in case of low temperature superconductor (LTS) materials like e.g. NbTi or Nb\textsubscript{3}Sn, E\textsubscript{c} = 0.1 \mu V/cm is used and for high temperature superconductor (HTS) materials like e.g. BSCCO or YBCO, E\textsubscript{c} = 1.0 \mu V/cm is widely used [66]. The electric field strength criterion is based on the formula (2.1):

\[ E = E_c \left( \frac{I}{I_c} \right)^n \]  \hspace{1cm} (2.1)

where the unit \( n \) is a finite number that represents the power-law exponent for the “take off” of the E-I characteristic at the superconductor-normal transition. It characterizes the steepness of the transition into normal conductivity and represents thereby some quality factor of the wire.
In Figure 3-8 is an example of a critical current measurement obtained in this research:

![Figure 3-8 Current versus voltage measurement of SuperOx superconducting tape.](image)

In the experiment of Figure 3-8 the distance between voltage taps was 1.9 cm. Therefore, according to the electric field strength criterion \((E_c = 1 \, \mu\text{V/cm})\), the sample’s critical current is the value achieved when the tap voltage reaches 1.9 \(\mu\text{V}\). In the graph, it was indicated the tap voltage of 1.9 \(\mu\text{V}\) and the critical current of 408.9 A. For this thesis, the critical current values considered as valid are only those whose voltage response are within a range between \(\pm 5\% \) of \(1\mu\text{V/cm}\) from the tap voltage, which means that obeys to the following formula:

\[
U = \left(\text{voltage taps distance [cm]}\right)E - 6 \, \text{V} \pm 5\% \cdot 1E - 6 \, \text{V} \quad (2.2)
\]

This formula let the experiments done in this thesis reveal more accurate results, excluding critical current values less precise.
In the first methodology, after the critical current pre-measurement, the pulling force starts increasing. This load increment is divided in steps and the increasing load between those force load steps was denominated as phase that finishes when the load step is reached. The load steps were pre-selected in Graphwork 6 software and for each experiment were set to the following values: 0.1 N; 10 N; 20 N; 30 N; 50 N and 50 N by 50 N until the 1,900 N, in which the maximum number of pre-set phases was reached.

![Graphwork 6 software phase setting.](image)

Figure 3-9 Graphwork 6 software phase setting.

The phase setting was done in three parameters as shown in the Figure 3-9:

1- Number of phases is set. The software allows from 1 to 42 phases.

2- In the second parameter is defined the increasing speed of each phase until the next force load step is reached.

3- In the third part the target force load step in which the phase ends is defined.

After reaching every pulling force load step, a critical current measurement was done and then a new phase was actioned manually in Graphwork 6. Between each step, the load speed increment was 0.5 N/s or 0.25 N/s enabling fast and reliable experiments. It would take a couple of hours to collect the same detail during a full 500 N delamination sample’s test for example and this is a process that depends directly on human hand to trigger the next load step.
A typical behaviour of the pulling force load versus time is represented in Figure 3-10.

In the experiment represented in Figure 3-10 the force load steps pre-set were 0.1 N; 10 N; 20 N; 30 N; 50 N and 50 N by 50 N until the 1,900 N and the pre-set speed in each phase was 0.25 N/s. It is possible to verify in Figure 3-10 a constant load during the force load steps of 10 N and 20 N, time interval that a critical current's measurement was done in each force load step.

In this approach, the critical current's measurement was done with a LabVIEW program available at KIT-ITEP which includes multiplexer for multichannel voltage measurements Agilent 34970A. This program controls the power supply and measures the voltage with nanovoltmeter. A typical result of an experiment performed during this research with a REBaCuO sample in a graph composed with the critical current's measurements in every force load step until the sample's delamination using this approach is shown in Figure 3-11.
Figure 3-11 Critical current measured in the pre-set force load steps in a SuperOx sample.

The vertical axis represents the critical current measured in each force load step and the horizontal axis represents the force load recalculated to pressure according anvil surface. The last point on the right of the graph corresponds to the last critical current’s measurement before the tape’s delamination.

The problem of this procedure was the low number of critical current measurements along the force load which avoided a precise understanding of the critical current’s behaviour along the load progression as well as during the delamination. Therefore, this approach was abandoned and replaced by a second approach.
In the **second methodology** it was defined only a 0.1 N pre-measurement force load step and 2500 N because a continuously critical current measurement during the entire experiment was implemented instead of a critical current’s measurement at each load step. To be possible a continuous critical current’s measurement, the input current has to follow the changing sample’s critical current attending to every taps voltage measurement. *LabVIEW* program provided a control function to increase or decrease the amount of current set manually in each iteration of tap voltage measurement, shown in Figure 3-12.

Each time that desires to increase the input current the user should type the amount of current in the *Strom increment* space and then click in the *Strom hoch* button twice. To decrease the input current, the method was the same but the user should type a negative amount of input current.

![Figure 3-12 Second methodology Labview interface.](image)

In this approach the test time and the delamination load were collected. A linear load speed was assumed, therefore the test time and test delamination load were divided in as many number of measurements done during the experiment and then matched up. The implemented methodology could in this way fill the lack of critical current measurements during the entire test going from around a dozen to hundreds of measurements depending on the sample’s maximum stress. The results of an experiment with the first methodology and an experiment with the second methodology are compared with the same set-up for the same pressure interval. Comparing critical current’s measurements from the experiments using first and second methodologies until 10 MPa, With the first methodology only 6 measurements were done while with the second methodology 139 measurements were done in the same interval of pressure.
Despite the better results obtained, this methodology could still be improved in some aspects. The number of measurements had greatly increased but it could increase even more with more reliable measurements. The process for which the input current follows the critical current along the test should become automatic. This should increase the critical current's measurement precision. This automatic system could also be more effective during the delamination providing a faster input current, getting a faster critical current measurement, a difficult task for the manual system used in this methodology.

Therefore, a third methodology was developed. In order to increase the number of critical current measurements during the experiment, especially during the delamination since it was not possible to collect any data related to the delamination with the previous methodologies, the multiplexer was removed from the initial set-up and the LabVIEW was adapted. The multiplexer was implemented in the initial set-up from other different experiments which enables more than one channel of voltage measurement for a simultaneously critical current’s measurement of many tapes. In this methodology, two load steps were defined. The constant force load steps were unnecessary because the critical current measurement was done continuously. The steps are an initial pre-measurement step and a final force load step.

To achieve automatic and more critical current measurements during the experiment, a LabVIEW program was done by Roland Gyuráki (KIT-ITEP). This program had the same features as the initial one used but without multiplexer.

After the 10 minutes sample's cooling down time in the 0.1 N load pre-measurement step, the critical current is reached and the next force load phase starts as happened in the previews methodology. But in this one an adjustment on the input current is made in order to keep the taps voltage as much constant as possible because it tends to have variations due to the mechanical stress applied on the sample as well as the errors associated the power supply and the measurement of nanovoltmeter in each iteration. In order to do the adjustment, it was implemented in the LabVIEW program a system that allows automatically in each iteration to increase or decrease a deliberated amount of current to be injected in superconductive tape if in the previous iteration measurement, the resultant voltage of the current injection is below or above the critical current voltage respectively. After a couple of tests, it was possible to figure out that when the critical current was reached the best values to adjust the current were ± 1%, that are actually the minimum possible switching values. It permits a small correction in the current values providing a better error resistance because it avoids big voltage jumps.
In the Figure 3-13 is the interface of the LabVIEW programme developed is shown. This interface is split in 6 areas which the 1., 2. , 3. and 6., are inputs and 4. and 5. outputs. From all the squares only the 1. and 2. are defined before starting the test, the remaining can be changed during the test.

1- In the first marked field is the directory where the data is recorded. The second field is to select the power supply Agilent 6680A and on the third field is to select the Agilent 34420A nanovoltmeter.

2- In the second block it is possible to select the maximum current possible during the test for safety reasons. The maximum current should be around 50 A above the critical current. The next one is defined the interval the milliseconds to wait between each interaction of the test from the nanovoltmeter but it is defined as 0 in order to get as many points as the nanovoltmeter capacity because the delamination interval is extremely fast in the most of the cases and its start is unpredictable. The nanovoltmeter has a reading speed of 0.3 readings/sec for the resolution used (7-1/2 digits) [67] which is the highest resolution of this device. The third item of this square is the distance between voltage taps that will define the critical current when multiplied by 1E-6 and at last there is the starting current value that generally was 0.

3- In the third block there are the commands to start the test. In order to stop the test, there are two ways, the Back to 0 button for an emergency situation or in the Stop button which can take almost 1 sec than the previous in order to finish the program cycle before the test be interrupted.

4- In the block 4 the outcome is the input current and the voltage measured of the last iteration.

5- This square shows the current increment when the current is below the critical current and the current decrement when the current is above the critical current. These values are referred by the user and can be changed during the test in any test iteration.

6- The square 6 shows an error box which will alert the user for any error during the test.
After several tests and with the know-how acquired related to the behaviour between current applied and correspondent voltage it was implemented a system in the LabVIEW aiming to get the maximum number of critical current points during the sample delamination.

The system consists of a comparison with the previous iteration registered voltage value. Depending on how far it is from the critical current correspondent voltage value using the criterion, the current is decreased a certain value in order to match again with the critical current correspondent voltage.

It is possible to check in the Table 3-1 the decrement for each value of voltage.

<table>
<thead>
<tr>
<th>Previous voltage measurement (V)</th>
<th>Current quantity decrement (A)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\geq 1 \cdot 10^{-5}$</td>
<td>50</td>
</tr>
<tr>
<td>$\geq 5 \cdot 10^{-6}$</td>
<td>10</td>
</tr>
<tr>
<td>$\geq$ Distance between voltage taps * $1 \cdot 10^{-6} + 2 \cdot 10^{-7}$</td>
<td>1</td>
</tr>
</tbody>
</table>

Table 3-1 Decreasing values of current attending the voltage values.

Considering to the voltage taps distance is always 1.9 cm, according to the critical current criterion the voltage measured is $1.9 \cdot 10^{-6}$ V.

The first voltage value compared in the Table 3-1 is with the highest value. When the voltage measured is above $1 \cdot 10^{-5}$ V that corresponds to a decay of input current of 50 A. When the voltage measured is between $5 \cdot 10^{-6}$ V and $1 \cdot 10^{-5}$ V the current decrements 10 A. For these two decays, the precise distance between voltage taps is not relevant. At last when the voltage goes higher than $2 \cdot 10^{-7}$ from the sample’s critical current, the current decreases 1 A. For values under those represented in the table the current decreases 0.01 A as referred in this previously in this chapter.

In order to protect the sample against getting burned to be still useful for an optical microscope examination, the program has also implemented an automatic Stop function that resets immediately the input current if the voltage measured during the test reaches values above $1 \cdot 10^{-3}$ V, generally it happens immediately after the sample delaminates.
At this point, a comparison between two experiments with the same set-up and tape using the second methodology and the third methodology respectively was carried. The Figure 3-14 and Figure 3-15 presents the results of the experiments of two samples from the same SuperPower superconducting tape for the same range of time. The experiment of the Figure 3-14 was carried using the second methodology while the experiment presented in the Figure 3-15 was carried using the third methodology.

Comparing the results of the second with the third methodology, the conclusion is that the improvement in quantity is clear and the quality also improved, which means more accurate voltage response to the current injection and consequently precise critical current measurements. Critical current measurement implies that the response voltage measured to the current injected on the sample is within the range of values that obeys to the formula (2.2) as mentioned at the end of chapter 2. Comparing both graphs,
the standard deviation from the second to the third methodology decreased 31.67 %. For the same period of time, in the second methodology were done 355 critical current’s measurements while in the third methodology were done 689 measurements. Almost doubled down the number of critical current’s measurements.

After the delamination all the collected data from Graphwork 6 is exported to .txt file which contains the load (N), the pressure (MPa) a conversion from the load which depends on the anvil’s surface, and the test duration (s). From the LabVIEW was collected the inject current and the tap voltage measured in each iteration.
4 Results and Discussion

In this chapter will be presented and discussed the results obtained from the experiments executed with the third methodology developed and explained in the previous chapter will be discussed.

The tape tested in this thesis was SuperPower 2G HTS Wire type SCS12050-AP. Surround Copper Stabilizer (SCS) is applied to completely encase the wire and provide overcurrent capability depending on the application. SCS protects the conductor and produces rounded edges that are beneficial for high-voltage applications which reduces the probability of a failure caused by voltage breakdown. SuperPower’s SCS has been successfully implemented and tested on continuous lengths of hundreds of meters of wire. AP wire means Advanced Pinning and this exhibits superior performance at a range of temperatures from 77 K to as low as 4 K, range that covers the liquid nitrogen temperature, where the samples were submerged during the experiments.

The relevant characteristics from the datasheet produced from the manufacturers for this specific Superpower SCS12050-AP tape used in the tests are in the following table.

<table>
<thead>
<tr>
<th>Tape</th>
<th>Width [mm]</th>
<th>Thickness [mm]</th>
<th>$I_c$ Average [A]</th>
<th>STDEV [%]</th>
<th>$I_c$ Minimum [A]</th>
</tr>
</thead>
<tbody>
<tr>
<td>SCS12050-AP</td>
<td>12</td>
<td>0.98</td>
<td>346</td>
<td>3.33</td>
<td>316</td>
</tr>
</tbody>
</table>

Table 4-1 SCS12050-AP KIT sample characteristics.

![Table 4-2 Characteristics of SCS12050 SuperPower tape](68).
Besides specifications from Table 4-2, this tape has 2 µm of Silver layer at top and 1.8 µm at the bottom side, 1 µm of REBCO layer grown epitaxially, 0.2 µm of Buffer stack and 50 µm of Hastelloy C-276 substrate.

Testing with the experimental set-up described in the chapter 3, 9 samples of HTS tapes from SuperPower were subjected to a delamination stress, with a load gradient of 0.05, 0.1, 0.5 N/s. In each test the sample was submitted to mechanical force which was registered in a pace of 0.01 s while the critical current measurements had a pace of 0.5 s, so the pace of the following graphs critical current/ delamination stress is 0.5s, the mechanical force data was filtered in order to match the current data.

Along the experiments, the use of critical current measurements, was useful to determine if the sample had already delaminated or partially delaminated in the soldering process, during the preparation of the experiment. This fact helps to validate the delamination experiments because there were samples with an initial critical current lower than minimum acceptable value provided by the manufacturer. Then the results of each successful experiment are presented. An important detail in the results analysis, only those experiments for which the initial sample’s critical current was higher than 316 A, the minimum critical current acceptable presented in the Table 4-1.

The results will be divided in 3 categories: Delamination strength and critical current, comparison of delamination strength and comparison of critical current under strength. The samples’ number shown in this chapter was ordered by the load gradient applied in the experiment and sample’s maximum stress.

4.1 Delamination Strength and Critical Current

In this section is presented the graphical results of each of the 9 samples. The results are presented in form of two graphs for each sample tested. First graph represents Critical Current vs Puling Pressure applied on the sample and the second graph represents Critical Current vs Time. The time represented in those graphs starts when the transverse stress started to be applied on the sample. Those graphs allow to understanding the critical current’s behaviour along the increasing stress applied on the sample until the delamination stress. A table with the delamination stress achieved by the sample during the experiment and critical current statistics is also presented.
Sample 1

For this experiment, the load gradient imposed was 0.05 N/s.

![Critical Current vs Pressure](image)

**Figure 4-1** Critical current versus applied stress of the experiment with the SuperPower sample 1.

![Critical Current vs Time](image)

**Figure 4-2** Critical current versus Time of the experiment with the SuperPower sample 1.

<table>
<thead>
<tr>
<th>Sample 1</th>
<th>Load gradient [N/s]</th>
<th>Max stress [MPa]</th>
<th>Initial $I_c$ [A]</th>
<th>Final $I_c$ (before delamination) [A]</th>
<th>$I_{c\ max}$ [A]</th>
<th>$I_{c\ min}$ [A]</th>
<th>$I_{c\ avg}$ [A]</th>
<th>Standard Deviation of $I_c$ [A]</th>
<th>Variance of $I_c$ [A]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.05</td>
<td>3.18</td>
<td>329.11</td>
<td>329.16</td>
<td>329.74</td>
<td>328.75</td>
<td>329.33</td>
<td>0.266</td>
<td>0.071</td>
</tr>
</tbody>
</table>

**Table 4-3** Statistical information of the experiment with sample 1.
Sample 2

For this experiment, the load gradient imposed was 0.05 N/s.

Critical Current vs Pressure

![Critical Current vs Pressure graph](image)

Figure 4-3 Critical current applied stress of the experiment with the SuperPower sample 2.

Critical Current vs Time

![Critical Current vs Time graph](image)

Figure 4-4 Critical current versus Time of the experiment with the SuperPower sample 2.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Load gradient [N/s]</th>
<th>Max stress [MPa]</th>
<th>Initial (I_c) [A]</th>
<th>Final (I_c) (before delamination) [A]</th>
<th>(I_c) (_{\text{max}}) [A]</th>
<th>(I_c) (_{\text{min}}) [A]</th>
<th>(I_c) (_{\text{avg}}) [A]</th>
<th>Standard Deviation of (I_c) [A]</th>
<th>Variance of (I_c) [A]</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td>0.05</td>
<td>6.76</td>
<td>340.78</td>
<td>341.02</td>
<td>341.74</td>
<td>340.44</td>
<td>340.95</td>
<td>0.225</td>
<td>0.050</td>
</tr>
</tbody>
</table>

Table 4-4 Statistical information of the experiment with sample 2.
Sample 3

For this experiment, the load gradient imposed was 0.1 N/s.

![Critical Current vs Pressure](image)

Figure 4-5 Critical current versus applied stress of the experiment with the SuperPower sample 3.

![Critical Current vs Time](image)

Figure 4-6 Critical current versus Time of the experiment with the SuperPower sample 3.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Load gradient [N/s]</th>
<th>Max stress [MPa]</th>
<th>Initial $I_c$ [A]</th>
<th>Final $I_c$ (before delamination) [A]</th>
<th>$I_c_{max}$ [A]</th>
<th>$I_c_{min}$ [A]</th>
<th>$I_c_{avg}$ [A]</th>
<th>Standard Deviation of $I_c$ [A]</th>
<th>Variance of $I_c$ [A]</th>
</tr>
</thead>
<tbody>
<tr>
<td>3</td>
<td>0.1</td>
<td>6.51</td>
<td>328.08</td>
<td>328.65</td>
<td>328.65</td>
<td>328.65</td>
<td>328.02</td>
<td>0.164</td>
<td>0.027</td>
</tr>
</tbody>
</table>

Table 4-5 Statistical information of the experiment with sample 3.
Sample 4

For this experiment, the load gradient imposed was 0.1 N/s.

Figure 4-7 Critical current versus applied stress of the experiment with the SuperPower sample 4.

Figure 4-8 Critical current versus Time of the experiment with the SuperPower sample 4.

<table>
<thead>
<tr>
<th>Sample [#]</th>
<th>Load gradient [N/s]</th>
<th>Max stress [MPa]</th>
<th>Initial $I_c$ [A]</th>
<th>Final $I_c$ (before delamination) [A]</th>
<th>$I_{c\ max}$ [A]</th>
<th>$I_{c\ min}$ [A]</th>
<th>$I_{c\ avg}$ [A]</th>
<th>Standard Deviation of $I_c$ [A]</th>
<th>Variance of $I_c$ [A]</th>
</tr>
</thead>
<tbody>
<tr>
<td>4</td>
<td>0.1</td>
<td>7.41</td>
<td>329.68</td>
<td>330.43</td>
<td>330.98</td>
<td>329.67</td>
<td>330.64</td>
<td>0.242</td>
<td>0.058</td>
</tr>
</tbody>
</table>

Table 4-6 Statistical information of the experiment with sample 4.
Sample 5

For this experiment, the load gradient imposed was 0.1 N/s.

![Critical Current vs Pressure](image1)

**Figure 4-9** Critical current versus applied stress of the experiment with the SuperPower sample 5.

![Critical Current vs Time](image2)

**Figure 4-10** Critical current versus Time of the experiment with the SuperPower sample 5.

<table>
<thead>
<tr>
<th>Sample 5</th>
<th>Load gradient [N/s]</th>
<th>Max stress [MPa]</th>
<th>Initial $I_c$ [A]</th>
<th>Final $I_c$ (before delamination) [A]</th>
<th>$I_{c\text{ max}}$ [A]</th>
<th>$I_{c\text{ min}}$ [A]</th>
<th>$I_{c\text{ avg}}$ [A]</th>
<th>Standard Deviation of $I_c$ [A]</th>
<th>Variance of $I_c$ [A]</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>0.1</td>
<td>11.54</td>
<td>345.74</td>
<td>327.9</td>
<td>346.16</td>
<td>327.9</td>
<td>340.40</td>
<td>5.223</td>
<td>27.27</td>
</tr>
</tbody>
</table>

Table 4-7 Statistical information of the experiment with sample 5.
Sample 6

For this experiment, the load gradient imposed was 0.1 N/s.

Critical Current vs Pressure

Critical Current vs Time

Table 4-8 Statistical information of the experiment with sample 6.
Sample 7

For this experiment, the load gradient imposed was 0.1 N/s.

![Critical Current vs Pressure](image)

**Figure 4-13** Critical current versus applied stress of the experiment with the SuperPower sample 7.

![Critical Current vs Time](image)

**Figure 4-14** Critical current versus Time of the experiment with the SuperPower sample 7.

<table>
<thead>
<tr>
<th></th>
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<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>7</td>
<td>0.1</td>
<td>12.6</td>
<td>325.06</td>
<td>325.41</td>
<td>325.92</td>
<td>324.64</td>
<td>325.41</td>
<td>0.242</td>
<td>0.0587</td>
</tr>
</tbody>
</table>

Table 4-9 Statistical information of the experiment with sample 7.
Sample 8

For this experiment, the load gradient imposed was 0.5 N/s.

Figure 4-15 Critical current versus applied stress of the experiment with the SuperPower sample 8.

Figure 4-16 Critical current versus Time of the experiment with the SuperPower sample 8.

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
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<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>8</td>
<td>0.5</td>
<td>6.95</td>
<td>325.89</td>
<td>269.28</td>
<td>325.94</td>
<td>325.58</td>
<td>325.70</td>
<td>0.065</td>
<td>0.004</td>
</tr>
</tbody>
</table>

Table 4-10 Statistical information of the experiment with sample 8.
Sample 9

For this experiment, the load gradient imposed was 0.5 N/s.

Figure 4-17 Critical current versus applied stress of the experiment with the SuperPower sample 9.

Figure 4-18 Critical current versus Time of the experiment with the SuperPower sample 9.

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
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<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>9</td>
<td>0.5</td>
<td>11.04</td>
<td>338.18</td>
<td>336.51</td>
<td>338.69</td>
<td>336.43</td>
<td>337.76</td>
<td>0.546</td>
<td>0.298</td>
</tr>
</tbody>
</table>

Table 4-11 Statistical information of the experiment with sample 9.
4.2 Comparison of Delamination Strength

The following table shows the load gradient imposed in each experiment, maximum stress the sample reached before delaminate and the corresponding load.

<table>
<thead>
<tr>
<th>Sample [#]</th>
<th>Load Gradient [N/s]</th>
<th>Max Stress [MPa]</th>
<th>Load [N]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.05</td>
<td>3.19</td>
<td>76.59</td>
</tr>
<tr>
<td>2</td>
<td>0.05</td>
<td>6.79</td>
<td>163</td>
</tr>
<tr>
<td>3</td>
<td>0.1</td>
<td>6.60</td>
<td>158.3</td>
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<tr>
<td>4</td>
<td>0.1</td>
<td>7.45</td>
<td>178.8</td>
</tr>
<tr>
<td>5</td>
<td>0.1</td>
<td>11.87</td>
<td>284.8</td>
</tr>
<tr>
<td>6</td>
<td>0.1</td>
<td>12.08</td>
<td>289.8</td>
</tr>
<tr>
<td>7</td>
<td>0.1</td>
<td>12.62</td>
<td>302.8</td>
</tr>
<tr>
<td>8</td>
<td>0.5</td>
<td>7.1</td>
<td>170.4</td>
</tr>
<tr>
<td>9</td>
<td>0.5</td>
<td>11.05</td>
<td>265.2</td>
</tr>
<tr>
<td>AVG</td>
<td>0.17(8)</td>
<td>8.75</td>
<td>209.96</td>
</tr>
</tbody>
</table>

Table 4-12 SuperPower sample's max stress.

The values obtained reveal to be within the range of maximum stress expected values comparing with the max stress values exposed in the literature, with an average of 8.75 MPa.

To investigate the influence of the load gradient on the sample's maximum stress a box and whiskers plot was created with the data from the Table 4-12.

Figure 4-19 Box and Whiskers plot max stress versus load gradient.
Analysing the Figure 4-19, the correlation between the load gradient imposed during the experiment and REBCO sample’s max stress was not conclusive. There were too few samples to make deeper analysis.

4.3 Comparison of Critical Current under Strength

The previous graphs shown in chapter 4.1, are shown, this time, overlapped in only one graph. The following graph shows the critical current vs pressure applied on the sample until the delamination of every sample.

Figure 4-20 Critical current versus applied stress of the SuperPower samples.
The following table has the information of the critical current and the applied force for each sample:

<table>
<thead>
<tr>
<th>Sample [#]</th>
<th>Load gradient [N/s]</th>
<th>Delamination stress [MPa]</th>
<th>Initial $I_c$ [A]</th>
<th>Final $I_c$ (before delamination) [A]</th>
<th>$I_{c\text{ max}}$ [A]</th>
<th>$I_{c\text{ min}}$ [A]</th>
<th>$I_{c\text{ avg}}$ [A]</th>
<th>Standard Deviation of $I_c$ [A]</th>
<th>Variance of $I_c$ [A]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.05</td>
<td>3.18</td>
<td>329.11</td>
<td>329.16</td>
<td>329.74</td>
<td>328.75</td>
<td>329.33</td>
<td>0.266</td>
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<tr>
<td>2</td>
<td>0.05</td>
<td>6.76</td>
<td>340.78</td>
<td>341.02</td>
<td>341.74</td>
<td>340.44</td>
<td>340.95</td>
<td>0.225</td>
<td>0.050</td>
</tr>
<tr>
<td>3</td>
<td>0.1</td>
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<td>328.65</td>
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<td>328.02</td>
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<tr>
<td>4</td>
<td>0.1</td>
<td>7.41</td>
<td>329.68</td>
<td>330.43</td>
<td>330.98</td>
<td>329.67</td>
<td>330.64</td>
<td>0.242</td>
<td>0.058</td>
</tr>
<tr>
<td>5</td>
<td>0.1</td>
<td>11.54</td>
<td>345.74</td>
<td>327.9</td>
<td>346.16</td>
<td>327.9</td>
<td>340.40</td>
<td>5.223</td>
<td>27.27</td>
</tr>
<tr>
<td>6</td>
<td>0.1</td>
<td>12.6</td>
<td>329.13</td>
<td>327.92</td>
<td>329.48</td>
<td>327.18</td>
<td>328.49</td>
<td>0.384</td>
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<tr>
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<td>325.41</td>
<td>325.92</td>
<td>324.64</td>
<td>325.41</td>
<td>0.242</td>
<td>0.0587</td>
</tr>
<tr>
<td>8</td>
<td>0.5</td>
<td>6.95</td>
<td>325.89</td>
<td>269.28</td>
<td>325.94</td>
<td>325.58</td>
<td>325.70</td>
<td>0.065</td>
<td>0.004</td>
</tr>
<tr>
<td>9</td>
<td>0.5</td>
<td>11.04</td>
<td>338.18</td>
<td>336.51</td>
<td>338.69</td>
<td>336.43</td>
<td>337.76</td>
<td>0.546</td>
<td>0.298</td>
</tr>
<tr>
<td>AVG</td>
<td>0.18</td>
<td>8.75</td>
<td>332.41</td>
<td>324.03</td>
<td>329.92</td>
<td>331.86</td>
<td>0.81</td>
<td>3.12</td>
<td></td>
</tr>
</tbody>
</table>

Table 4-13 Statistical Analysis of the 9 samples.

Analysing the results from the Figure 4-20 and Table 4-13 is possible to get a pattern of the critical current behaviour along each experiment. The standard deviation and consequently the variance of the critical current measured along the experiments, until the sample delamination was approximately zero, except for the sample 5, according to the Table 4-3. In average, and excluding the experiment of the sample 5, the variance of critical current was 0.089.

All the samples tested followed the pattern described except the sample 5. The critical current of the sample 5 kept constant until a certain moment that dropped 7 A and after that point, the critical current started to decrease slowly every iteration of measurement. Probably in this case some layer of the tape delaminated and caused in the other layers instability. A good way to analyse the occurred phenomenon is to investigate the sample’s layers with a microscope for future work.

Similarly to what happened to the samples’ delamination stress, attending the load gradient imposed to each sample did not have any impact to the variance of critical current throughout the experiments.
5 Conclusion and Future Work

5.1 Conclusion

In this thesis, the delamination of SuperPower tapes, REBCO HTS coated conductors, was investigated. This investigation was performed by testing the REBCO electro-mechanical properties. The results of the investigation show that, during the increment of the transversal mechanical stress applied to the samples, in 88.9% of the experiments the samples’ critical current kept the variance almost zero until the delamination. There was one exception, where the critical current started dropping systematically before the delamination. At the delamination, the tape’s critical current of every single sample dropped abruptly, leaving instantly the superconducting state.

The maximum stress from the samples was on average 8.75 MPa, which fits the interval of values of reference, mentioned in the state of the art. The variation of load gradient imposed in each experiment did not have any correlated impact on the sample’s delamination stress. This variance did not have any correlated impact on the critical current variance throughout the experiments as well.

Although the results presented provided important insights in this field of research, there is still a lot of experiments to carry out to understand the HTS coated conductors delamination issue. Finding out the causes for the issues is a good way to make the next generation HTS more reliable and it will be possible to use them in wider range of applications.

5.2 Future Work

For future work, data obtained from this laboratory measurements could be used for evaluating and modelling the flow of current in the conductor, when the latter is subjected to mechanical stress. This can be done by using finite element (FE) analysis with commercial programs, such as, Matlab and Comsol Multiphysics.

The samples delaminated used in this thesis could be analysed by microscope, in order to figure out in which type of delamination occurred during the experiments, and which REBCO layers were affected. With this data, it could be possible to get a pattern of the samples’ delamination layers.

The same experiment could be also applied to the superconductors from others manufacturers, to understand their behavior. Consequently, a selection of the superconductor with better characteristics could be done according to each application, attending their behavior towards the delamination. It could also help to understand why superconductors from some manufacturers are stronger than the others, which could improve the robustness of future superconductor tapes.
6 References


[7] R. F. e. al, “Minimising exposure to this particular magnetic field vector is therefore a key part of our HTS coil design,” 9th European Conference on Applied Superconductivity (EUCAS 09), 2010.


7 Appendix

A.1

Material Safety Data Sheet

SuperPower Inc.
450 Duane Avenue
Schenectady, New York 12304 USA
Information: 518-346-1414

EMERGENCY ASSISTANCE
Call SuperPower Inc. at: 1 800-459-2519

IMPORTANT: Read this MSDS before handling and disposing of this product. Pass this information on to employees, customers and eventual end users.

Date MSDS Issued: June 12, 2007

SECTION I – PRODUCT IDENTIFICATION

Identity: SCS4050, SF12050, SF12100; High Temperature Superconductor Wire
Chemical Family: Metal Alloy
Formula: Each alloy contains nearly identical elements formulated in different concentrations.

SECTION II – HAZARDOUS INGREDIENTS

<table>
<thead>
<tr>
<th>CONSTITUENT</th>
<th>CAS Number</th>
<th>SCS4050</th>
<th>SF12050</th>
<th>SF12100</th>
<th>OSHA PEL (mg/m³)</th>
<th>ACGIH TLV (mg/m³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Co(II) (Co)</td>
<td>7440-48-4</td>
<td>1.3</td>
<td>2.3</td>
<td>2.4</td>
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<td>0.02</td>
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<tr>
<td>Chromium* (Cr)</td>
<td>7440-47-3</td>
<td>6.2</td>
<td>15</td>
<td>15</td>
<td>Metal 1</td>
<td>Metal and Cr III 0.5</td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Cr II 8, III, as Cr 0.5</td>
<td>Soluble Cr VI 0.05</td>
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<td>Copper* (Cu)</td>
<td>7440-50-8</td>
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<td>15</td>
<td>15</td>
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<td>Dust 1</td>
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<tr>
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<td></td>
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<td>Fume 0.1</td>
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<td>Iron (Fe)</td>
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<td>5</td>
<td>5</td>
<td>5 Ceiling</td>
<td>Fume 6</td>
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<td>Manganese (Mn)</td>
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<tr>
<td>Molybdenum (Mo)</td>
<td>7439-98-7</td>
<td>8.2</td>
<td>15</td>
<td>15</td>
<td>5</td>
<td>Insoluble 10 Soluble 5</td>
</tr>
<tr>
<td>Nickel* (Ni)</td>
<td>7440-02-0</td>
<td>29</td>
<td>52</td>
<td>55</td>
<td>1</td>
<td>Metal 1.5 Insoluble Compounds 0.2</td>
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<td>Silicon (Si)</td>
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<td>0.1</td>
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<td>Total 15 Respirable 5</td>
<td>Total 10 Respirable 3</td>
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<tr>
<td>Silver* (Ag)</td>
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<td>4.3</td>
<td>6.3</td>
<td>3.3</td>
<td>0.02</td>
<td>0.1</td>
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<td>Tungsten (W)</td>
<td>7440-33-7</td>
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<td>4</td>
<td>4</td>
<td>10 STEL</td>
<td>5</td>
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<tr>
<td>Vanadium (V)</td>
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<td>Ceiling 8, 5 Ceiling 8, 5 Ceiling</td>
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<td>REBCO(1)</td>
<td>None</td>
<td>1</td>
<td>2</td>
<td>1</td>
<td>None Determined</td>
<td>None Determined</td>
</tr>
</tbody>
</table>

*Identifies substances that are subject to the requirements of Section 313 of Title III of Superfund Amendments and Reauthorization Act of 1986 and 40 CFR Part 372.

(1) REBCO, “Rare Earth” elements with Barium Copper Oxide. RE elements include the following metals: Europium (CAS 15622-71-1), Dysprosium (CAS 15622-69-7), and Gadolinium (CAS 14768-16-1), Yttrium (7440-65-6), Samarium (7440-19-9)

Revised/Updated: 2012-0204  Effective: 2012-0204

Figure 7-1 SuperPower Datasheet.
### Specifications

| Specifications                                      | 6680A | 6681A | 6682A | 6683A | 6684A | 6680A-J04
<table>
<thead>
<tr>
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<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>(at 0 °C to 95 °C unless otherwise specified)</td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td>Special order option</td>
</tr>
<tr>
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</tr>
<tr>
<td>Output ratings</td>
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<td></td>
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<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Output voltage</td>
<td>0-5 V</td>
<td>0-8 V</td>
<td>0-21 V</td>
<td>0-32 V</td>
<td>0-40 V</td>
<td>0-3.3 V</td>
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<tr>
<td>Output current</td>
<td>0-875 A</td>
<td>0-580 A</td>
<td>0-249 A</td>
<td>0-160 A</td>
<td>0-128 A</td>
<td>0-1000 A</td>
</tr>
<tr>
<td>Programming accuracy (at 25 °C ± 5 °C)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Voltage</td>
<td>0.04% +</td>
<td>5 mV</td>
<td>8 mV</td>
<td>21 mV</td>
<td>32 mV</td>
<td>40 mV</td>
</tr>
<tr>
<td>Current</td>
<td>0.1% +</td>
<td>450 mA</td>
<td>330 mA</td>
<td>125 mA</td>
<td>85 mA</td>
<td>65 mA</td>
</tr>
<tr>
<td>Ripple and noise constant voltage mode from 20 kHz to 20 MHz</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Voltage</td>
<td>1.5 mV</td>
<td>1.5 mV</td>
<td>1.5 mV</td>
<td>1.0 mV</td>
<td>1.0 mV</td>
<td>3.4 mV</td>
</tr>
<tr>
<td>peak-to-peak</td>
<td>15 mV</td>
<td>10 mV</td>
<td>10 mV</td>
<td>10 mV</td>
<td>10 mV</td>
<td>15 mV</td>
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<tr>
<td>Readback accuracy at 25 °C ± 5 °C (percent of reading plus load)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Voltage</td>
<td>0.05% +</td>
<td>7.5 mV</td>
<td>12 mV</td>
<td>32 mV</td>
<td>48 mV</td>
<td>60 mV</td>
</tr>
<tr>
<td>Current</td>
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<td>600 mA</td>
<td>400 mA</td>
<td>165 mA</td>
<td>110 mA</td>
<td>90 mA</td>
</tr>
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<td>Load and line regulation</td>
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<td>1.5 mV</td>
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<td>65 mA</td>
<td>40 mA</td>
<td>17 mA</td>
<td>12 mA</td>
<td>9 mA</td>
</tr>
<tr>
<td>Transient response time</td>
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<tr>
<td>Less than 900 µs for the output voltage to recover to within 150 mV following a change in load from 100% to 50% or 50% to 100% of the output current rating of the supply</td>
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### Supplemental Characteristics

<table>
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<tr>
<th>Characteristics</th>
<th>6680A</th>
<th>6681A</th>
<th>6682A</th>
<th>6683A</th>
<th>6684A</th>
<th>6680A-J04</th>
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<tbody>
<tr>
<td>(Non-warranted characteristics determined by design and useful in applying the product)</td>
<td></td>
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<td>Special order option</td>
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<td>Ripple and noise constant voltage mode from 20 kHz to 20 MHz</td>
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<tr>
<td>Current</td>
<td>290 mA</td>
<td>190 mA</td>
<td>40 mA</td>
<td>28 mA</td>
<td>23 mA</td>
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<tr>
<td>Average programming resolution</td>
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<tr>
<td>Voltage</td>
<td>1.35 mV</td>
<td>2.15 mV</td>
<td>5.7 mV</td>
<td>8.6 mV</td>
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<td>Current</td>
<td>285 mA</td>
<td>195 mA</td>
<td>64 mA</td>
<td>48 mA</td>
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<td>260 mA</td>
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<td>Output voltage programming response time*</td>
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<td>(excluding command processing time)</td>
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<tr>
<td>Voltage</td>
<td>9 ms</td>
<td>12 ms</td>
<td>45 ms</td>
<td>60 ms</td>
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<td>9 ms</td>
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<tr>
<td>Current</td>
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<td>10 mA</td>
<td>20 mA</td>
<td>20 mA</td>
<td>20 mA</td>
<td>12.5 mA</td>
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<tr>
<td>Output common-mode noise current (to signal-ground binding post)</td>
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<td>Voltage</td>
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<td>3 mA</td>
<td>3 mA</td>
<td>2.0 mA</td>
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<td>10 mA</td>
<td>20 mA</td>
<td>20 mA</td>
<td>20 mA</td>
<td>12.5 mA</td>
</tr>
</tbody>
</table>

* Full load programming rise/fall time (10% to 90% or 90% to 10%) with full resistive load equal to rated output voltage/rated output current.

Figure 7-2 Agilent 6680A Power Supply Datasheet.
Figure 7-3 Keysight 34420A Nanovoltmeter datasheet pp03.
Figure 7-4 Keysight 34420A Nanovoltmeter datasheet pp.04.
Figure 7-5 Labview Program to control the electrical component of the experiment.