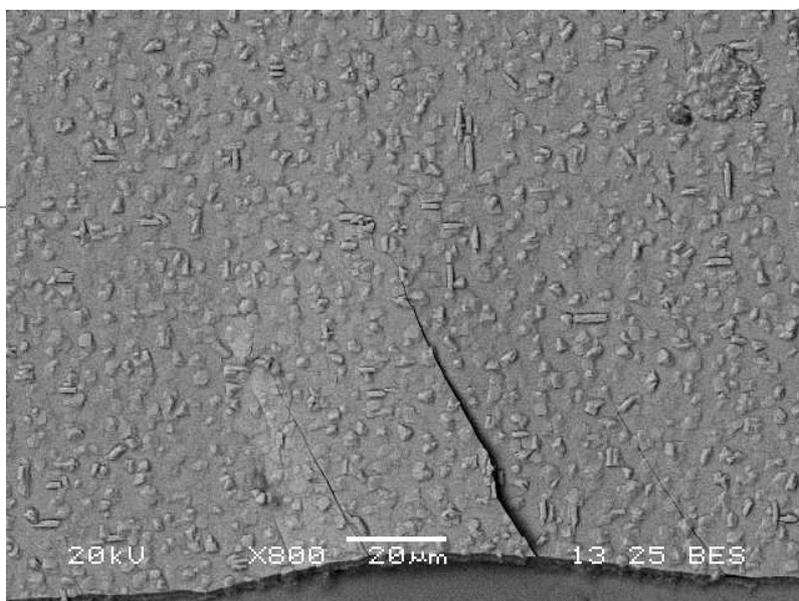


Bachelor assignment

Critical current degradation and damage assessment of YBCO coated superconductive tapes under transverse compressive stress.



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1 INTRODUCTION

Since the invention of electricity and the many discoveries made since then we came a long way. Today superconductivity is one of the big research topics. Superconductors offer some great potential. Just like a regular resistive conductor, like copper, a superconductor can be used to make components such as solenoids, magnets and power lines. A superconductor will do the same job but without the power consumption. There is however a catch, superconductivity only works below certain “critical” temperatures which means additional refrigeration costs. Even with these costs it can be more cost-efficient to use superconductors instead of resistive conductors in high current applications.

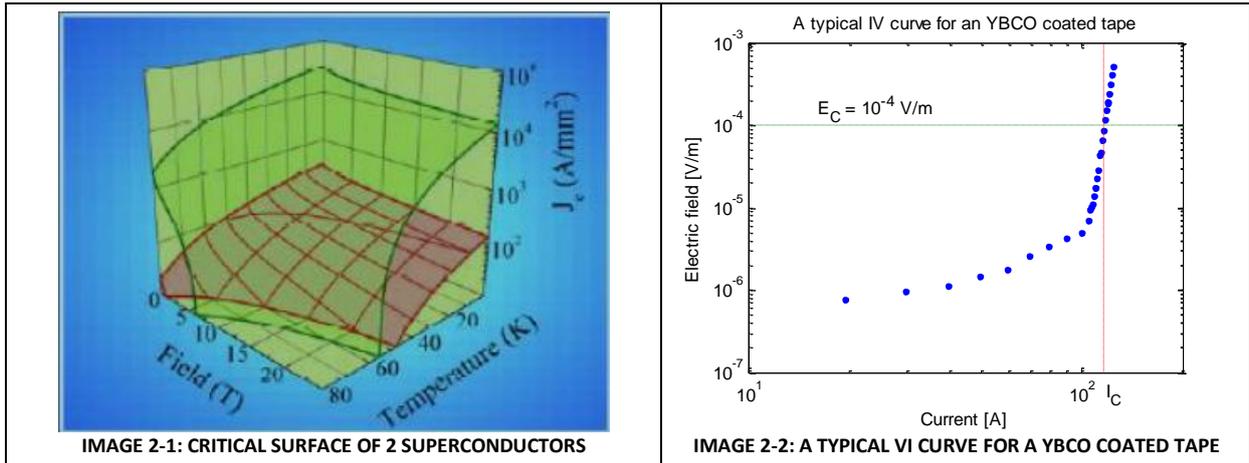
Some examples of superconductors applied; superconducting magnets guide protons in the Large Hadron Collider at CERN and solenoids are used to manipulate plasma in the ITER nuclear fusion reactor. The low temperature superconductors (LTS) used in these solenoids requires cooling with liquid helium. Liquid helium cooling is very expensive. The discovery of high temperature superconductors (HTS) means a lot of money can be saved on cooling. However before they can be applied a lot of knowledge is needed. In application the wires will have to withstand years of use. To design cables that are able to withstand these forces it is necessary to know how different materials will react to mechanical forces. YBCO is a HTS conductor with a critical temperature of 93 K, well above the boiling point of Nitrogen at 77 K. If all goes well YBCO wires may be used in DEMO, the proposed successor of ITER. Lorentz forces acting on wires can push them into their supports or other wires creating transverse loads.

Previous results from this group show variation in the degradation behavior and inconsistencies with the results in literature. This bachelors assignment will focus on degradation caused by transverse compressive loads on YBCO coated tapes. The results will be used to verify the previous results. The damage to layer of YBCO from the experiment will be examined by use of etching and SEM microscopy. By examining the damage a theory of how the damage is induced can be made. This theory will be verified by using Finite Element Methods calculation software. Together with other research a model can be constructed. This model can be used to design cables and predict their performance and durability.

2 THEORY

2.1 SUPER CONDUCTIVITY

Superconductivity is a phenomenon that occurs when a material exhibits zero electric resistivity. Superconductivity was discovered by the Dutch scientist Heike Kamerlingh Omnes in 1911 [1]. There are three constraints to superconductivity: current density, temperature and magnetic Field. These factors combined form a critical surface, as seen in Image 2-1.



The most important constraint is temperature. The conductor must be cooled to at least the critical temperature to exhibit superconductive properties. The lower the temperature becomes, the higher the other two factors can be. The current density will decrease when temperature and/or the external magnetic field increase. Each point that is enclosed by the critical surface describes a superconducting state.

When current is conducted through a superconductor there is no electric field. Until the critical current density is reached the behavior of the conductor will become resistive again resulting in an electric field. In-between fully super and resistive conductivity is a transition area in which the field will rise exponentially. When the conductor is conducting all the added current in the resistive state the field will raise linearly as one would expect from Ohm's law. The field plotted versus the current, or a VI curve, will show this behavior as can be seen in Image 2-2.

2.2 YBCO

Yttrium Barium Copper Oxide (YBCO) is a crystalline superconductor. Its chemical formula is $YBa_2Cu_3O_{7-x}$. It is the first material found to be superconductive above the temperature of boiling nitrogen (77 K). Materials above 77 K are High Temperature Superconductors (HTS). The critical temperature of YBCO is 93 K, this makes YBCO a HTS. These, compared to LTS, relatively high temperatures mean less cooling costs. The structure is of a crystalline nature with a Perovskite structure. YBCO and all the other HTS's are Type-II superconductors. The structure is a highly complex oxide ceramic. Ceramic properties include high Young's modulus (strong) but also brittle and prone to cracking. Yielding of the material could result in very sudden degradation due to crack propagation along the crystal structure[2]. Examples can be seen in Image 3-27.

These material properties of YBCO are needed to do simulations in COMSOL, they are summarized in Table 2-1.

TABLE 2-1: MATERIAL PROPERTIES OF YBCO

Material	Poisson's ratio	Young's Modulus [GPa]	Density [kgm^{-3}]	Yield stress [MPa]
YBCO	0.3 [3]	157 [4]	6300 [5]	684-748 [6]

2.3 Scs4050

The wire that is being used for this experiment is produced by SuperPower INC. The wire is designed as a tape which consists of multiple layers. Each layer is responsible for a different task. The design is made to mechanically and electrically support the superconducting layer in the best way. Image 2-3 shows the composition of the tape.

The scs4050 tape has a thickness of 0.1mm and is produced from the inside out. The substrate is made of Hastelloy®C-276 which is very strong and meant to strengthen the tape. On top of it are a few buffer layers. These layers help in the growing of the YBCO (or (RE)BCO) layer which is grown epitaxial*. The actual superconducting layer of YBCO is only 1µm thick which is just 1% of the total height of the full tape. Cross-sections made by Kosse [7] in image show that the actual YBCO layer is a lot thicker at about 2-3µm. For the simulations a thickness of 2 µm will be used. The tape used for the cross-section is from the same batch as the samples used in this research. After the YBCO layer has been produced, the silver layer is created on both sides of the tape. The copper layer is made last. The silver layer is for protecting the YBCO layer; together with the copper layer it provides mechanical stabilization. The silver and copper also carry any current which the YBCO cannot conduct in case of damage, overcurrent or high temperature. The silver and copper layers are also useful because of their high heat transfer capabilities, the heat generated by a quench* can be carried away quickly to prevent burning of the sample.

TABLE 2-2: YIELD STRESSES FOR COMPONENTS OF SCS4050 TAPE

Material	Yield Stress @ 77k [MPa]
Copper	38.5 [8]
Silver	45-76 [9]
YBCO	684-748 [6]
Hastelloy	440-470 [6]

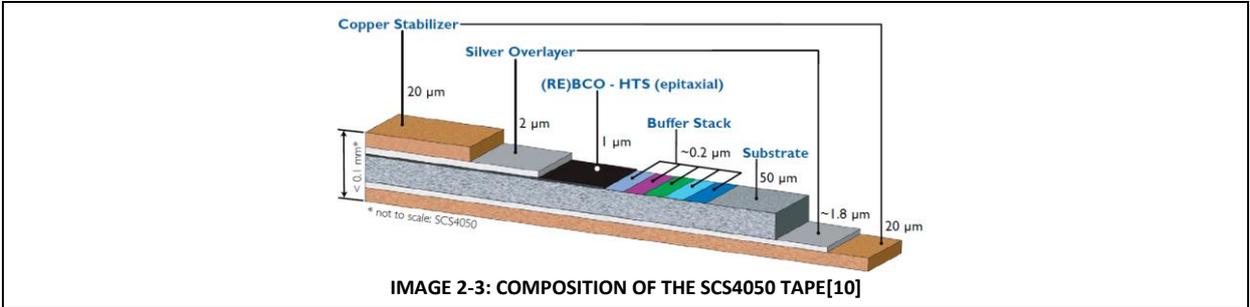


IMAGE 2-3: COMPOSITION OF THE SCS4050 TAPE[10]

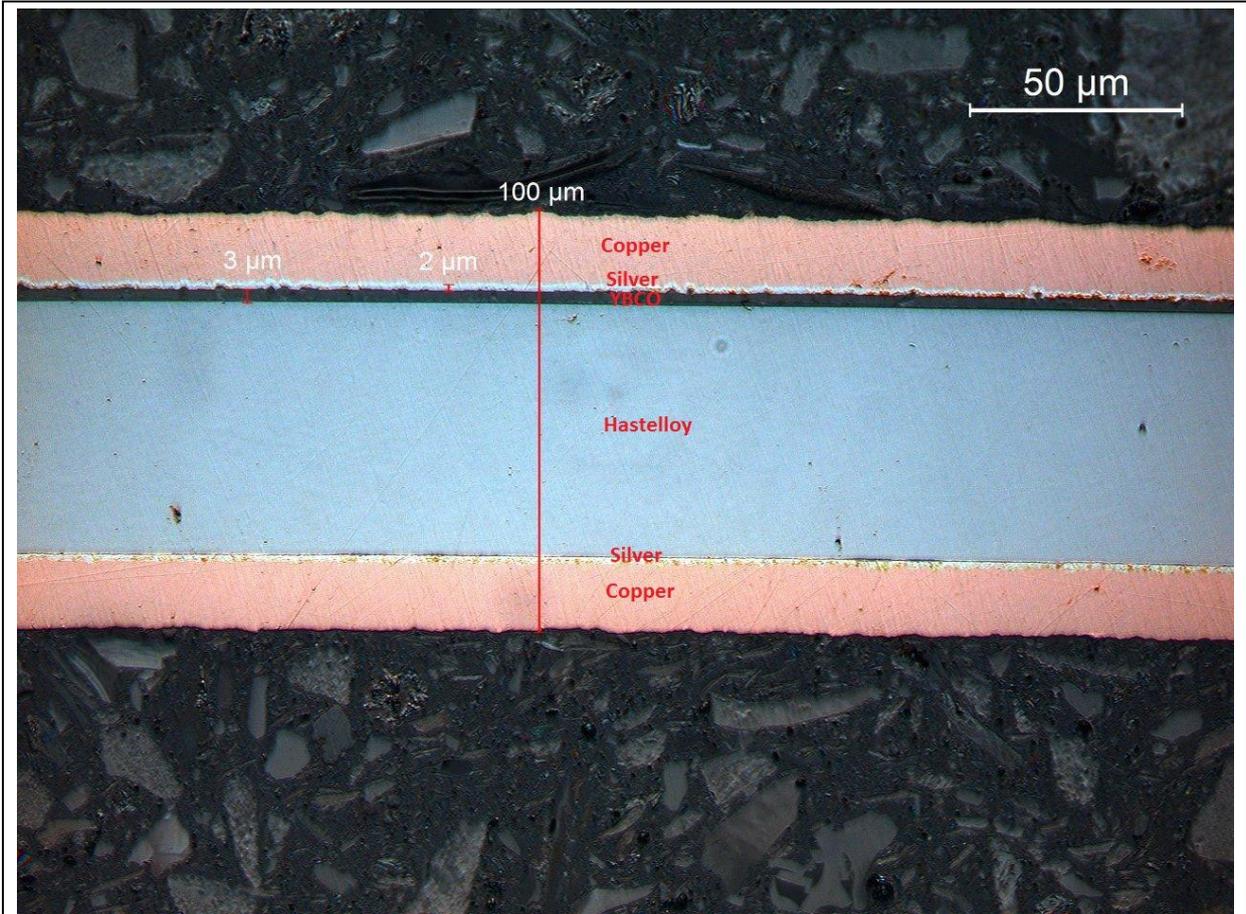
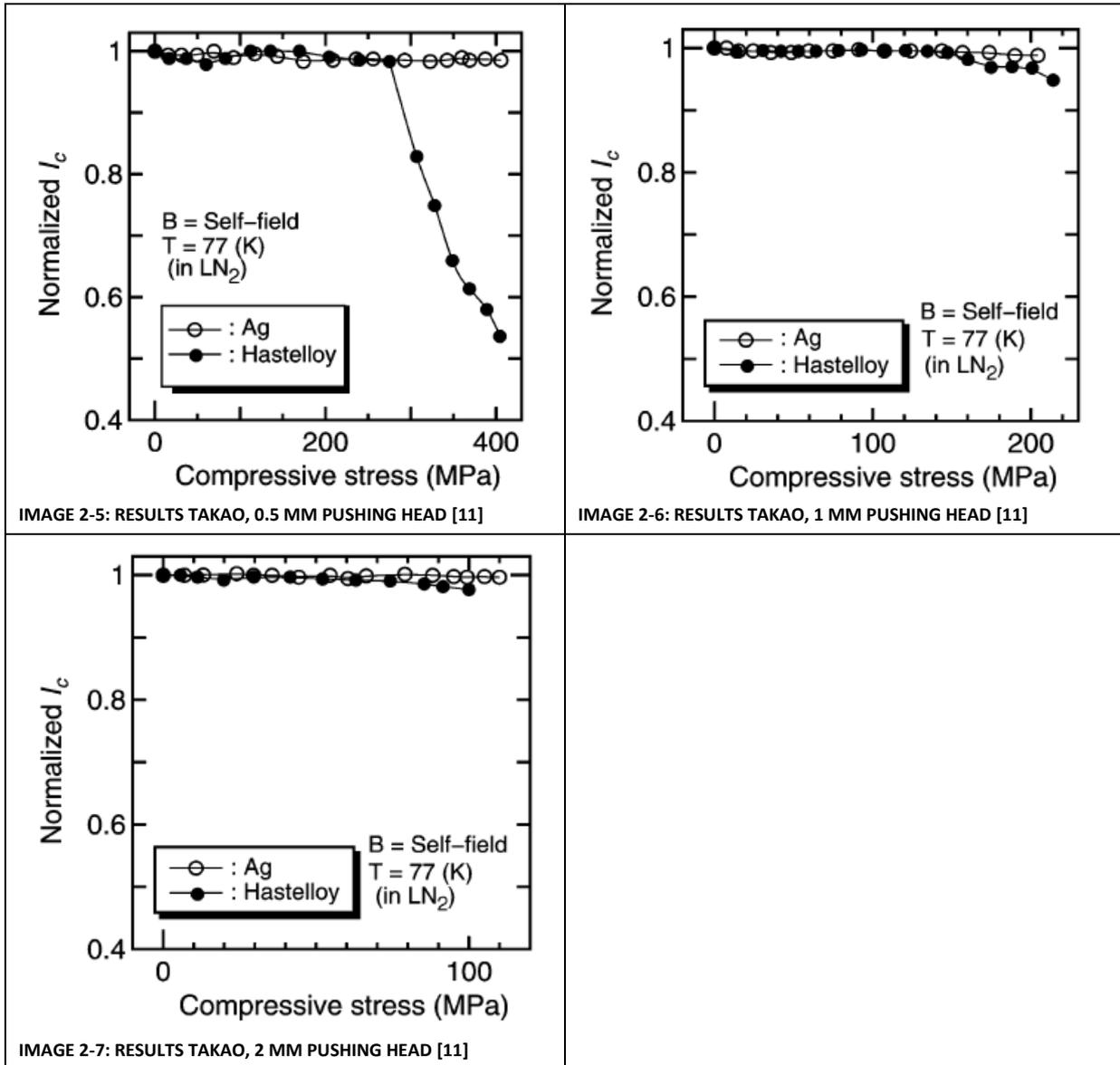
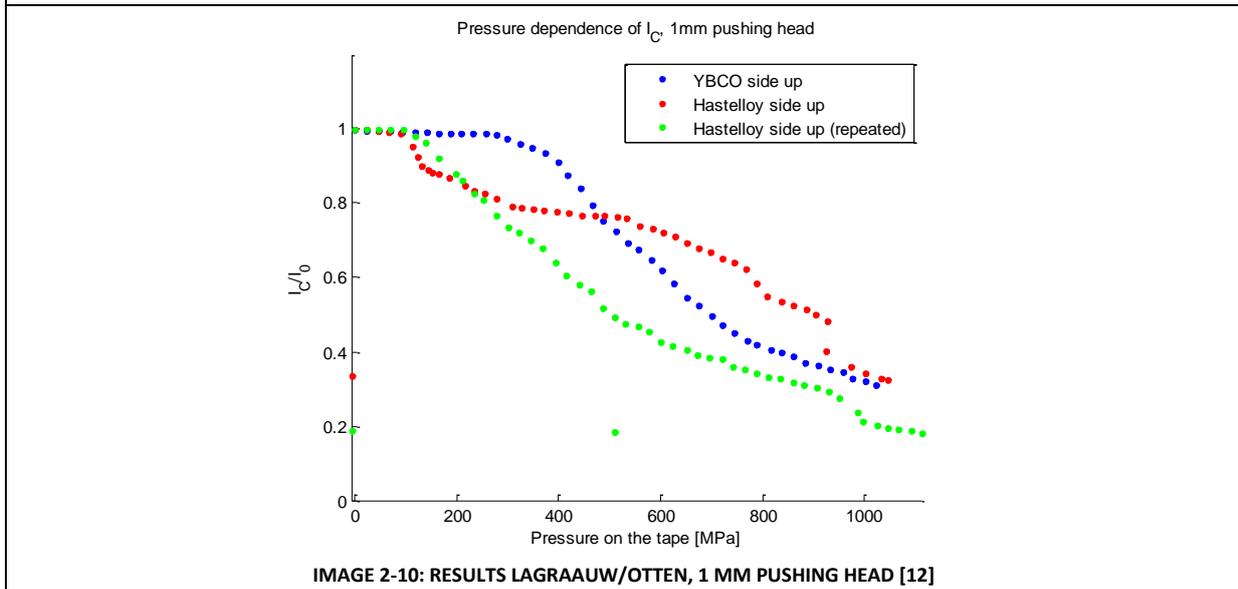
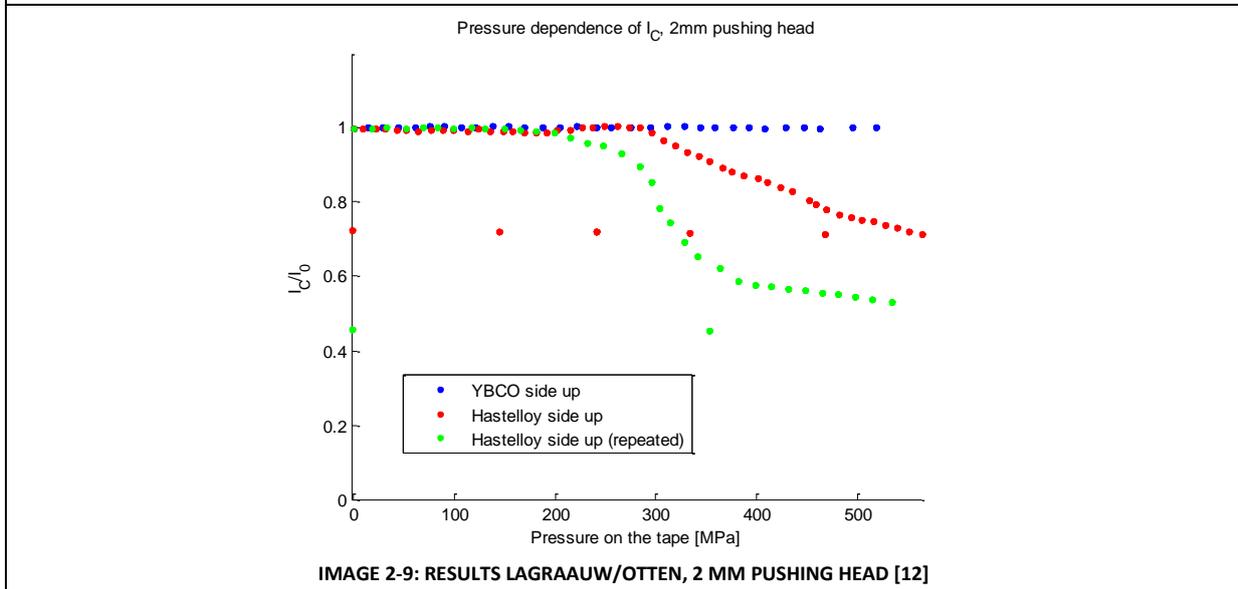
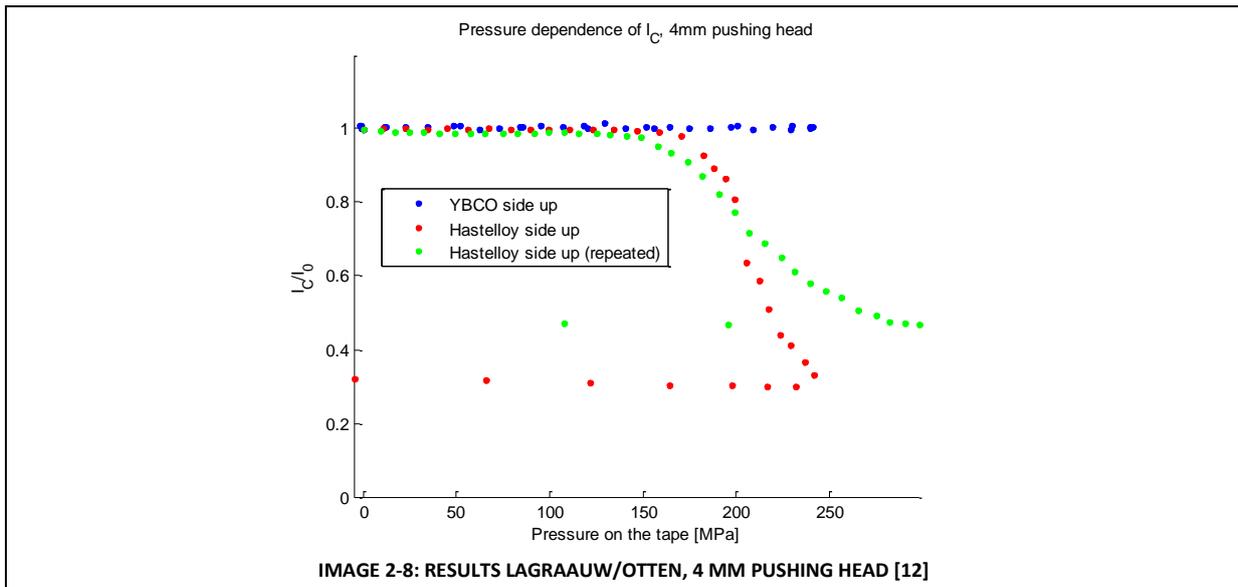


IMAGE 2-4: CROSS SECTION OF SCS4050 TAPE [7]

2.4 DEGRADATION CURVES TAKAO



2.5 DEGRADATION CURVES OTTEN/LAGRAAUW



For 1mm pushing head and Hastelloy side up, degradation starts at 160MPa in Takao's results and 130MPa (average) for the Otten/Lagraauw results. The rate of degradation is less with Takao.

For 2mm pushing head, degradation starts at 90MPa for Takao but 285Mpa (average) for Otten/Lagraauw.

The results from Otten/Lagraauw differ after measurements are repeated. Analysis of the setup and procedures are needed to verify results and decrease variation in results.

The samples used by Takao are 10mm in width and are without a copper stabilizer. The Otten/Lagraauw samples are of the same kind used in this experiment. So the results from Takao cannot be compared accurately with the results from Otten/Lagraauw and this experiment. Degradation starts at lower pressure for substrate side up loading in both experiments. Contradicting results are seen, the critical stress decreases with a different pushing head in Takao's results while Otten/Lagraauw have the reverse behavior.

2.6 DAMAGE MECHANISMS

As can be seen from both researches; the degradation occurs at different pressures when the orientation of the tape is changed. Degradation starts at a higher pressure for samples that have been loaded from the YBCO side of the tape. Takao [11] has a possible explanation:

When we push the tape from the silver face, the YBCO layer is above the Hastelloy in the tape structure; the Hastelloy is bent in convex shape underneath and hence the YBCO layer is compressed. On the contrary, when the tape is compressed from the Hastelloy face, the YBCO layer is tensile along the tape direction, because the YBCO layer is below the Hastelloy and the Hastelloy is bent in convex shape underneath. According to the bending test to the YBCO tape, degradation due to the compression to the YBCO layer hardly occurred. Hence in case of the compression from the silver surface, I_c did not decrease

Results from Cheggour [2] are explained in a similar way:

In the monotonic-loading mode, the sample has a strong frictional support from the pressing anvils. We believe this support prevents the sample from expanding laterally. In contrast, the frictional support is significantly reduced when operating in the load-unload mode. In-plane expansion becomes possible, which may lead to cracking of the buffer and YBCO layers. Another possible source for the degradation of could be delamination of the ceramic layers due to application of stress. More comprehensive data are still required to draw definitive conclusions.

The damage to YBCO under transverse compressive stress are cracks both in the longitudinal and transverse directions, mostly on the edges[2]. Tensile strain results in cracks transverse to the strain direction [13].

3 EXPERIMENT

3.1 SETUP

The press is attached to the far end of a tube; this tube is the housing of the current leads and other wires. The tube also houses the rod through which a force on the sample can be applied. The combination of the tube and press is called the insert. As its name reveals, the insert is inserted into the cryostat. The cryostat is filled with liquid nitrogen, cooling the insert down to a temperature of 77 Kelvin. The sample is soldered to the current leads (not pictured).

3.1.1 THE PRESS

The press used for this experiment was developed by Bennie ten Hake about 20 years ago for research in "Strain effects on the critical properties of high-field superconductors". The press was used to measure the influence of compressive stress on low temperature superconductors. It can exert forces up to 6kN.

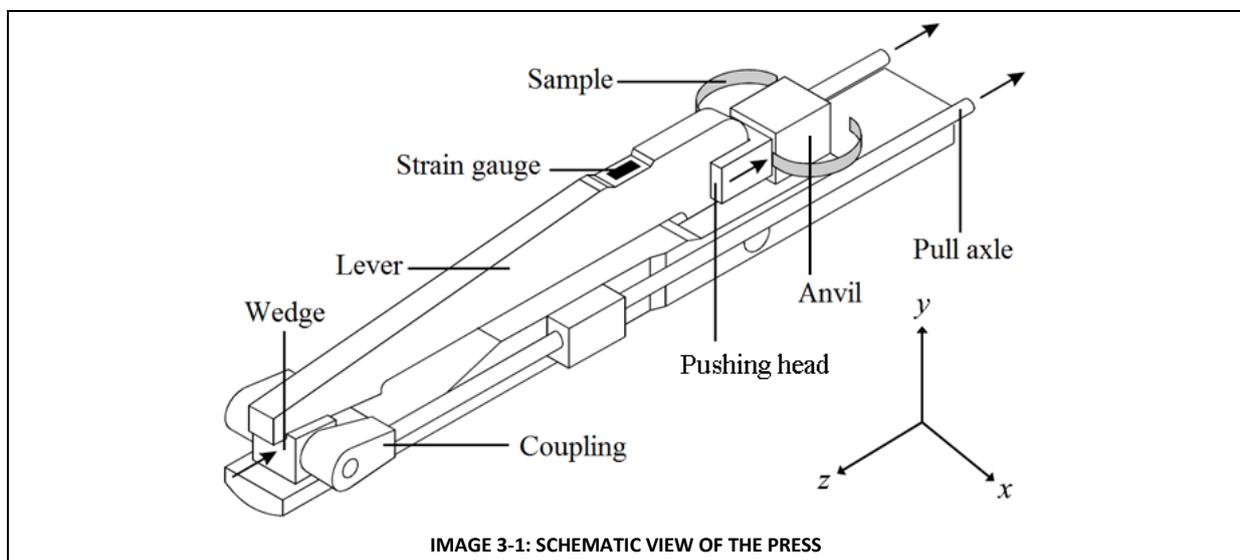


IMAGE 3-1: SCHEMATIC VIEW OF THE PRESS

A transverse compressive stress needs to be exerted on the tape while it is submerged in liquid nitrogen. Therefore, the press is designed to control the force on the tape externally.

Rotating a rod on the end of the insert causes the pull axle to move in the upward ($-z$) direction (arrow direction in figure 3.1), the coupling part has to move along and takes the wedge with it. The wedge is obliquely cut, the same way the lever is cut. Thus by moving upwards, the wedge will push the lever in the sideward (y) direction, which will push the pushing head towards the stainless steel anvil in the $-z$ direction. As can be seen in figure 3.1, the tape is positioned between the pushing head and the anvil. The strain gauge is used to measure the applied force.

It is important that a pushing head presses perpendicular to the tape to produce a homogeneous load. Each pushing head is circularly curved on the inside; so when under a small angle, the pushing head will rotate until the pressing surface is parallel to the sample.

3.1.2 EQUIPMENT

To obtain a VI curve the sample will need to conduct a current while the electric field is measured. When there is a force applied, this needs to be measured. Also the sample needs to be protected against quenching. If the nitrogen level inside the cryostat is too low the sample may warm up and lose its superconductive properties and quench. If a too high current is supplied the sample may quench too. The sample voltage, quench detector voltage, force and current are monitored by VI.

To prevent a quench from overcurrent the voltage over the outer two voltage tabs is constantly measured by a Nano Volt meter. The voltage from the Nano Volt meter is monitored by a quench detector, as the voltage exceeds a threshold of 15 times the critical field the current is inhibited.

The nitrogen level is monitored by a blue 4mm LED. The LED is put at a safe working height and supplied with 10mA from a current supply. The voltage over the LED is measured and can be checked on a volt meter. At room temperature the voltage over the LED is about 3.2V. When cooled down the mobility of the ions in the semiconductor the LED is made of will get less mobile and an increase in voltage will be observed. When the LED is submerged in the liquid Nitrogen the voltage will be about 7.8V. The LED is just in indication of the level and is not used to automatically inhibit the current like the quench detector does.

To supply the current a 200A current source is used. The current can be set through the VI software. Since the current control is not very accurate the actual supplied current is measured with a zero flux transducer which is controlled by an amplifier and measured by a multi meter.

The sample voltage is measured using a nano volt meter.

The force can be calculated from the strain on the lever that is applying the force to the sample. The strain is measured by a multi meter. The meter measures the resistance of the resistor bridge in the strain gauge through one pair while a second pair is used to supply a voltage. The used 4 point method prevents measuring errors caused by bad connections since the voltage measuring pair is not used to supply the current.

3.1.3 VI

The software used in this experiment was originally written by Bennie ten Haken and has since been developed by the EMS group. The software can control the hardware and read out the values from the meters. The measurements can be automated with the use of macros.

3.2 CHECKING THE PRESS

Because previous results from Lagraauw/Otten are inconsistent and the first measurements made with the 4mm pushing head did not agree with either the previous results or literature it was decided to check the press for any problems. During the checkup the anvil was polished with very fine sandpaper. The polished surface got very smooth and thus reflective. The lack of reflection revealed the dents that were present in the anvil as can be seen in Image 3-2. To reduce the influence of the dents, the anvil was carefully polished until all dents were gone, Image 3-3 shows the polished damage.

With the anvil being repaired the results for the 4mm pushing head yielded in a much higher critical pressure and faster degradation rate. These results are in better agreement with the literature so the press has been repaired. The dents can cause pressure concentrations which would explain the earlier degradation and low degradation rate.



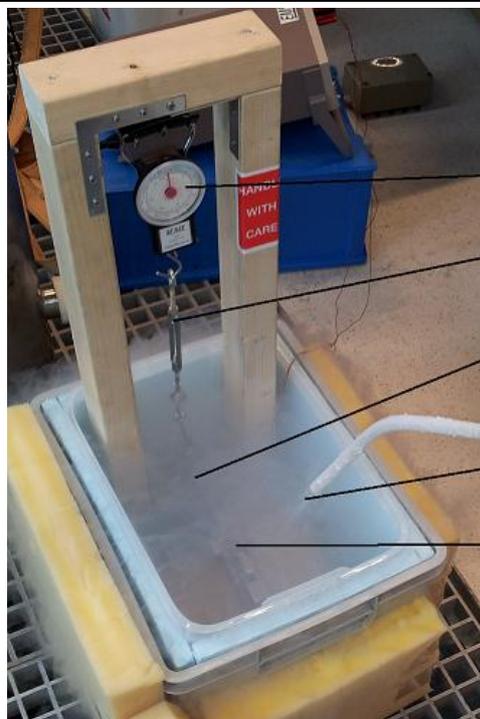
IMAGE 3-2: ANVIL AFTER SHORT POLISHING, DAMAGE IS CLEARLY VISABLE



IMAGE 3-3: ANVIL AFTER POLISHING, THE DAMAGE HAS BEEN REMOVED

3.3 CALIBRATION

The calibration has to be done under similar circumstances as for the measurement. The modulus of elasticity is temperature dependent so calibration is conducted at the same temperature as the experiment will be (77K). For the calibration the press is removed from the insert and put in a bin filled with liquid Nitrogen. Inside the bin is a wooden frame to which the press is fixed. On the lever of the setup there is a hole through which with steel wire and a cable tensioner a force is applied. The force applied to the lever is measured with a load cell. The force on the anvil/sample can be calculated: momentum exerted by the lever = momentum exerted by the anvil. At zero force the value of the strain gauge is measured. Then the force is increased stepwise and at each step the force and strain are measured. The results should obtain a linear relation between strain and force. Nonlinear strain is a sign of plastic deformation of the lever which would mean the press is being damaged. The calibration setup can be seen in Image 3-4, except the suitcase weigher is replaced by a load cell for more accurate results.



Suitcase weigher

Cable tensioner

Lever

Liquid nitrogen

Strain gauge

IMAGE 3-4: SETUP FOR CALIBRATION OF THE STRAIN GAUGE IN LIQUID NITROGEN [12]

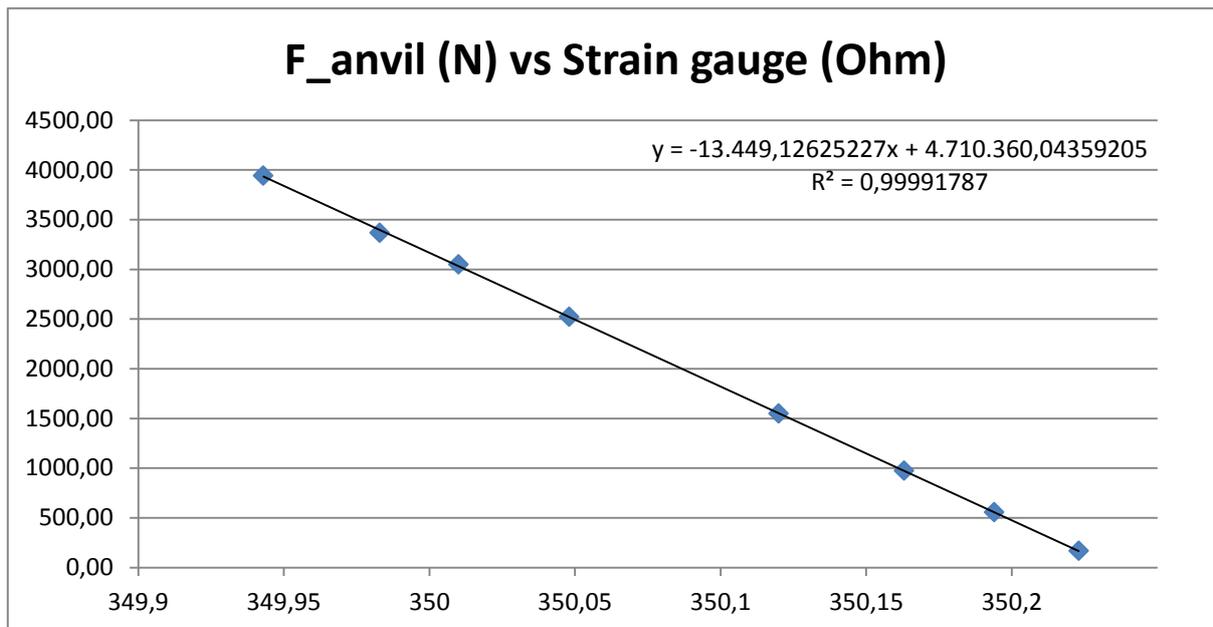


IMAGE 3-5 FORCE ON THE ANVIL PLOTTED AGAINST STRAIN GAUGE VALUE. THE FITTED LINE'S FORMULA AND ERROR ARE DISPLAYED IN THE TOP RIGHT CORNER.

3.4 PUSHING HEADS

This experiment uses 3 pushing heads which are similar in shape but differ in size. The pushing heads all cover the full width of the sample. The widths of the pushing heads are 1,2 and 4mm. In the image you can see the sample in orange with on top in blue the pushing area covering the full width of 4mm and 1mm along the length of the tape.

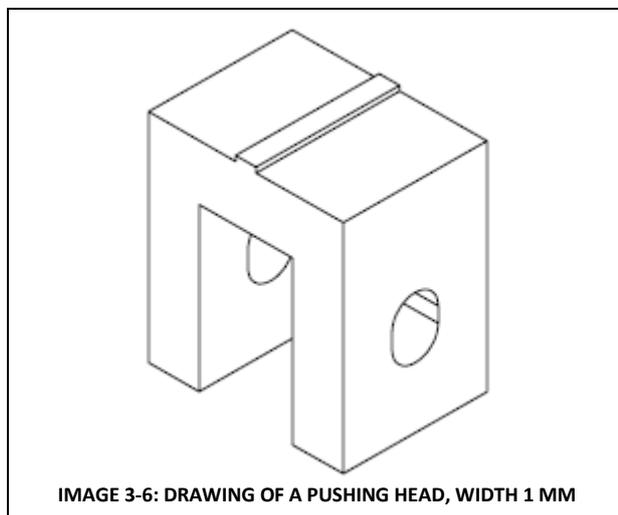


IMAGE 3-6: DRAWING OF A PUSHING HEAD, WIDTH 1 MM

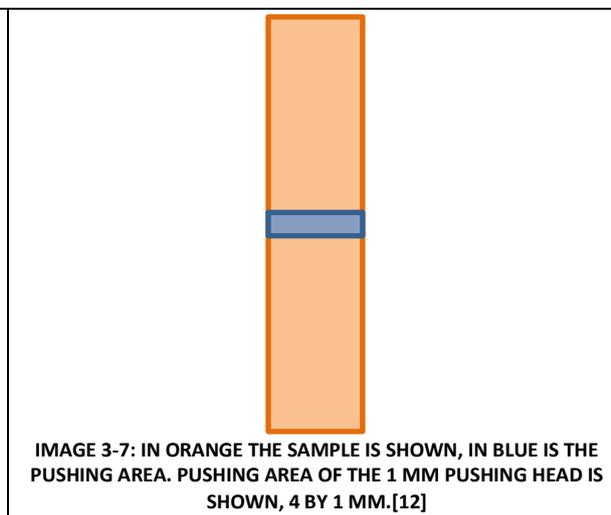


IMAGE 3-7: IN ORANGE THE SAMPLE IS SHOWN, IN BLUE IS THE PUSHING AREA. PUSHING AREA OF THE 1 MM PUSHING HEAD IS SHOWN, 4 BY 1 MM.[12]

3.5 SOLDERING

The sample is soldered to the current leads. This connection needs to be able to carry the current and should not affect the properties of the sample. *“The soldering temperature should be kept below ~ 250°C, especially for elongated processing time (e.g., a few hours), to avoid possible degradation in superconductivity.”* [14] If it is necessary to exceed 250°C the soldering time should be limited to 30 seconds and 350°C. Soldering of the sample to the current leads was analyzed and optimized to prevent sample damage. The resulting soldering procedure can be found on page 35.

3.5.1 DIFFICULTIES

Before the soldering procedure was produced there were a lot of samples that showed no superconductivity. Analyses showed damage to the superconducting layer at the edges of the current leads. The alignment of the current leads and the anvil was checked and a misalignment was found and improved. One current lead was above the anvil while the other current lead was below the anvil. This forced the sample to bend over the edge of the high current lead and the edge of the anvil to reach the lower current lead. To prevent strain from heating and cooling of the sample during soldering (250°C) and cooling down (-196°C) results in a very large temperature difference of $\Delta T = 250 + 196 = \sim 450^\circ\text{C}$. The different materials the setup is composed of are all subject to high temperature differences which can cause forces on the sample which would induce a force on the sample. To prevent forces on the sample one of the current leads is connected loosely to the rest of the setup.

3.6 MEASURING

Measurements are made with the VI software. To make the degradation curves we need to determine the critical current. The software uses a macro to produce the VI curves from which it will calculate the critical current. When the critical current is found the pressure on the sample is increased and the software will measure the critical current again. These steps are repeated until the critical current has been reduced to about 60% of its original capabilities. The pressure on the sample is then released and the critical current is measured once more to see how much of the damage is reversible. The pressure is never released between measurements, this is called continuous loading mode.

3.7 QUALITATIVE APPROXIMATION OF MECHANICS IN PRESS

Hooke's law is used which assumes the material behaves linearly, which it doesn't for the critical stress (yielding occurs). Volume change between anvil and pushing head related to stress:

$$\Delta V_{tot} = (2d)^2 * (x_0 - x) = 4d^2 * x_0 * \frac{\sigma}{E}$$

$$\Delta x = \varepsilon * x_0$$

$$\varepsilon = \frac{\sigma}{E}$$

The displacement caused by this can be calculated by assuming the displacement is linearly dependent of the height in the sample. Because of friction the displacement is 0 at the anvil and pushing head. In the center the displacement is maximal.

$$q = q_{max} * \left(1 - 2 * \frac{|z|}{x_0}\right) = 2d * \frac{\sigma}{E} * \left(1 - 2 * \frac{|z|}{x_0}\right)$$

$$q_{max} = 2d * \frac{\sigma}{E}$$

The change in displacement is the longitudinal strain, so deriving q:

$$\varepsilon_{YBCO} = \frac{dq}{dd} = 2 * \frac{\sigma}{E} * \left(1 - 2 * \frac{|z|}{x_0}\right) = \text{constant}$$

With;

d the distance from the center of the pushing area

ε the transverse strain

E the modulus of elasticity

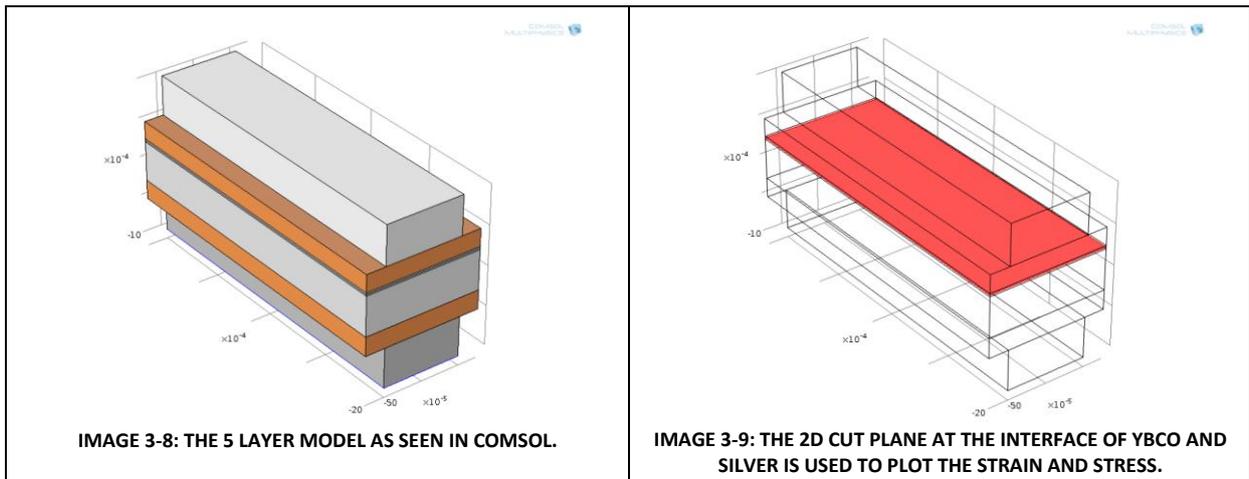
σ the strain
 q the displacement
 x the thickness of the sample because of the load
 x_0 the thickness of the sample before loading
 z the height in the sample, with $z=0$ the center of the sample

Conclusion, strain itself is not causing cracks until critical strain is reached. Strain is constant for whole pushing area, so damage would be everywhere in pushing area. The 3 layer simulations seem to agree.

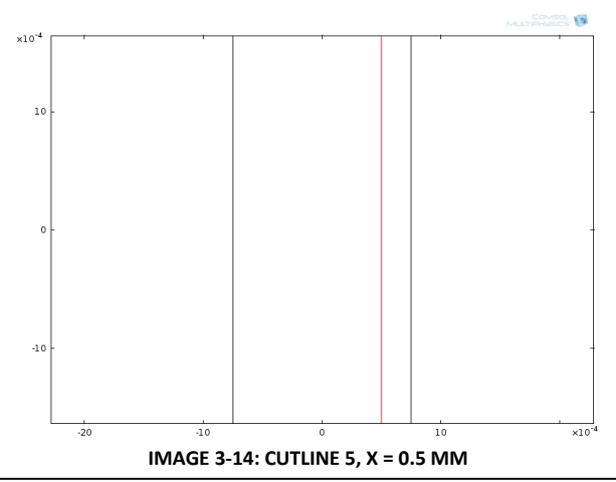
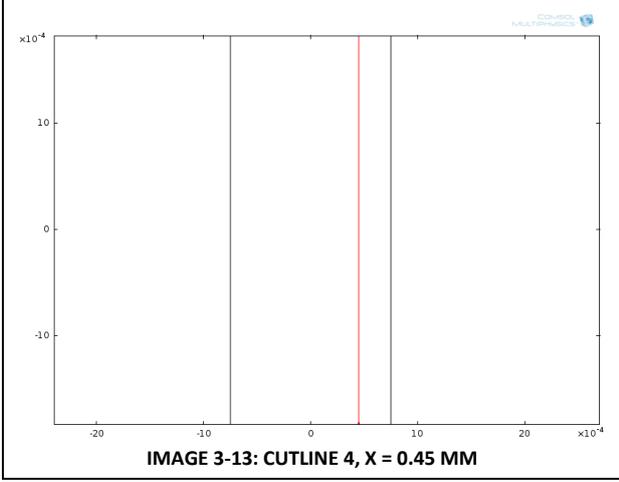
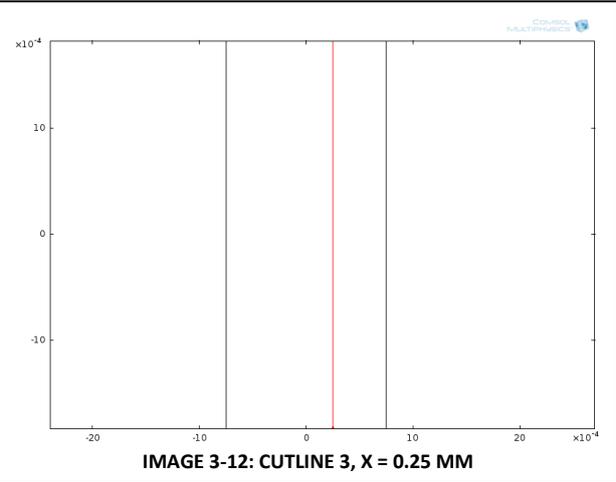
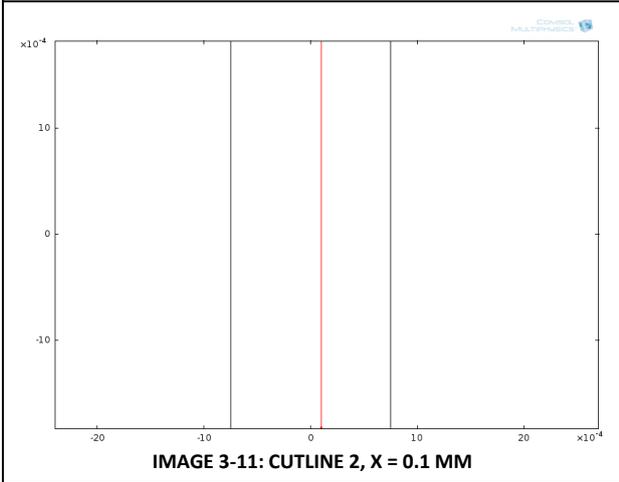
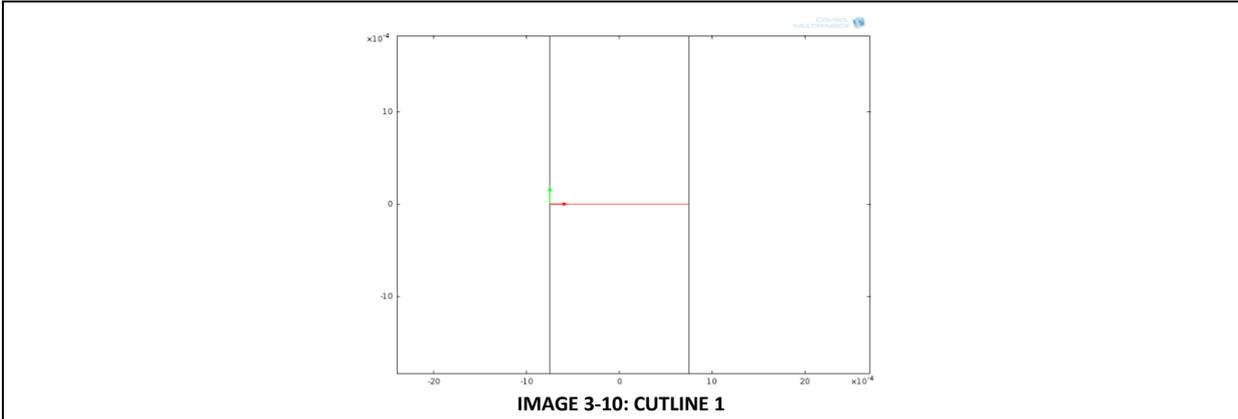
3.8 SIMULATION

To get a basic understanding of the mechanics in the press a simulation is performed. In the first simulation the tape is made of one layer of copper. The thickness is the same as for the sample used in the experiments. Then the model is made more complex. First a 3 layer model is used. In the most detailed simulation, 5 layers are used. The silver layer opposite to the YBCO layer and the buffer layers are omitted. These layers are very thin so their aspect ratio is very large. If these layers were incorporated they would require a lot of nodes. More nodes, equals more memory usage. The layers are omitted because they are of small relevance to the mechanics. The memory saved can be used to refine the mesh in the rest of the model.

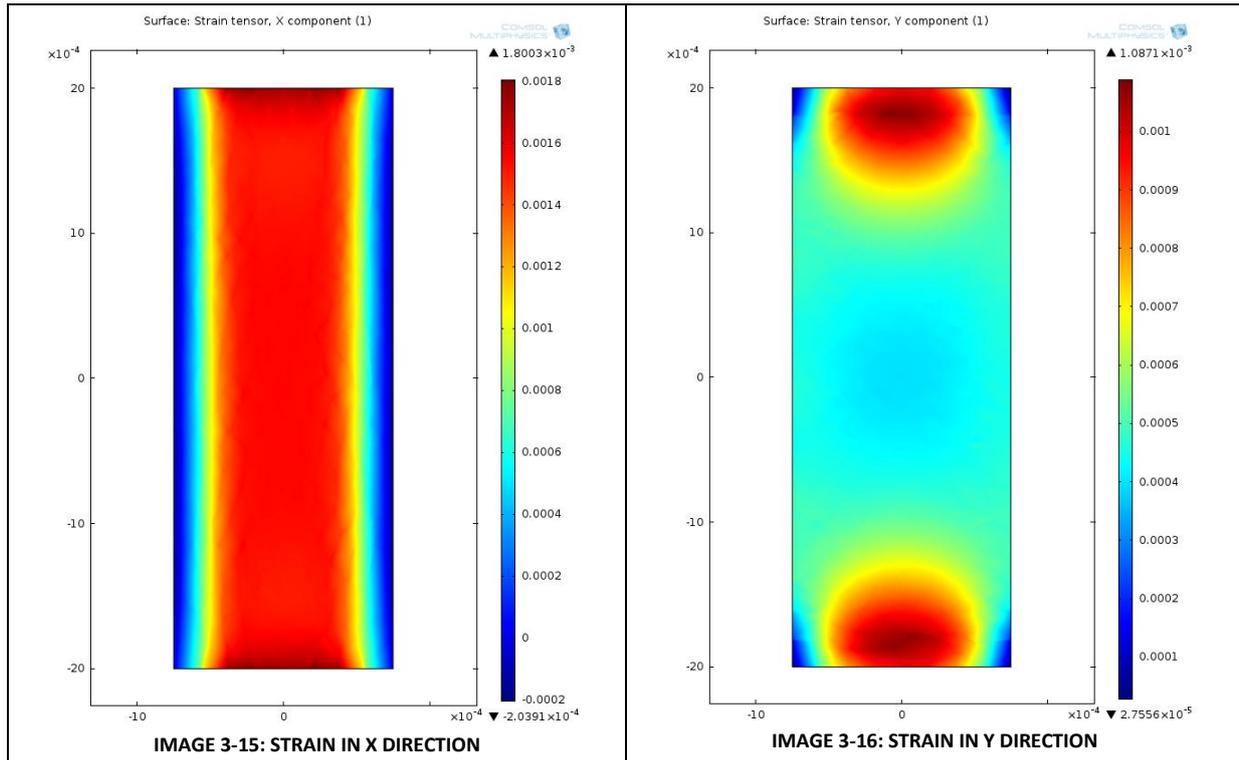
To determine the influence of non-symmetric pushing area the 1mm pushing head is used. To find the locations in which damage will occur the strain and von Misses stress are plotted in both 2D and 1D. The interface of the YBCO and silver is used to plot the stain and stress. This surface is the one we observe with SEM. Because all nodes are in the same plane the plots should be smooth.



To produce the 1D plots; cutlines are made in the 2D cut plane. Cutline 1 is parallel to the side of the sample, through the center. Then there are cutlines 2,3,4,5. They are along the side of the pushing head, perpendicular to the side of the tape. They are located at 0.1, 0.25, 0.45 and 0.5 mm from the center of the pushing area. Cutline 5, at 0.5 mm, is located at the edge of the pushing area.

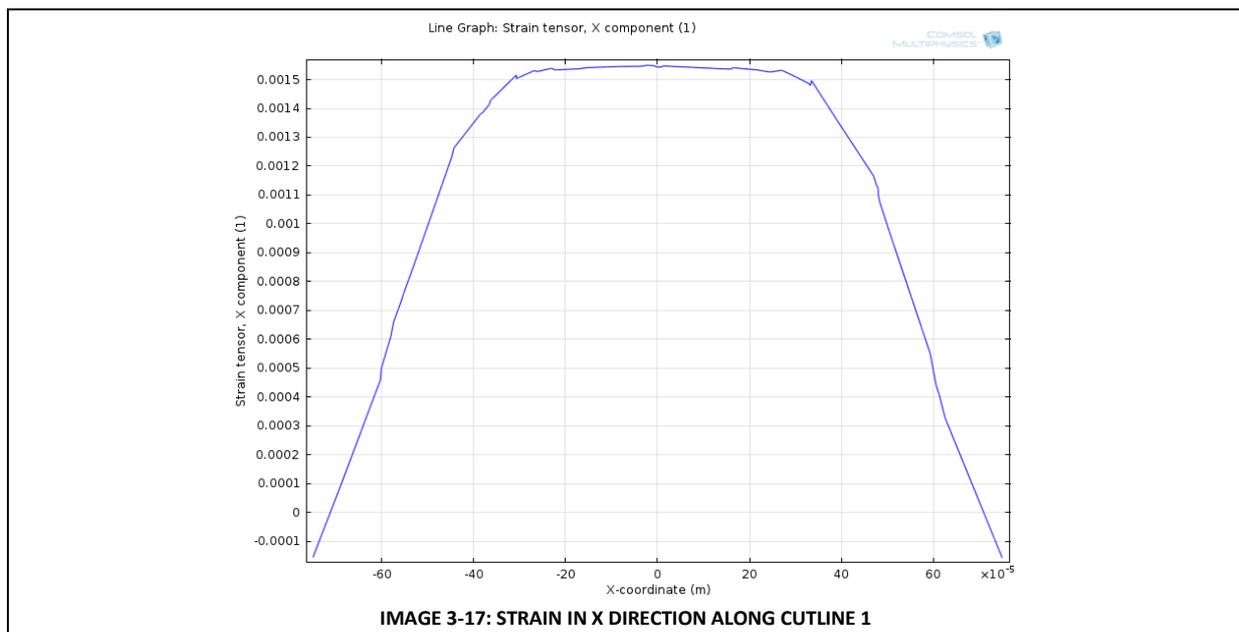


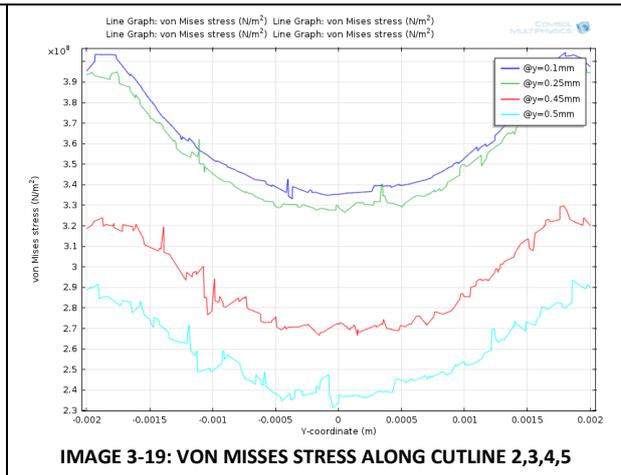
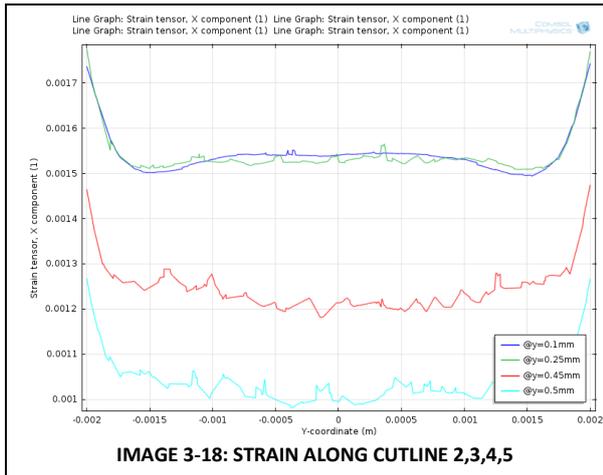
3.8.1 1 LAYER



From the 2D plot in Image 3-15 and Image 3-17 it is clear the strain in X direction is positive and uniform in most of the pushing area, as was calculated in paragraph 3.7. Just before the pushing head edge; the strain decreases. On the edge of the sample, 0.25 mm outside of the pushing area, the strain becomes slightly negative. This is very unlikely to happen so it is probably a boundary effect. The strain in Y direction has a maximum on the edges of the sample. As can be seen from the scale, it is smaller than the strain in the X direction.

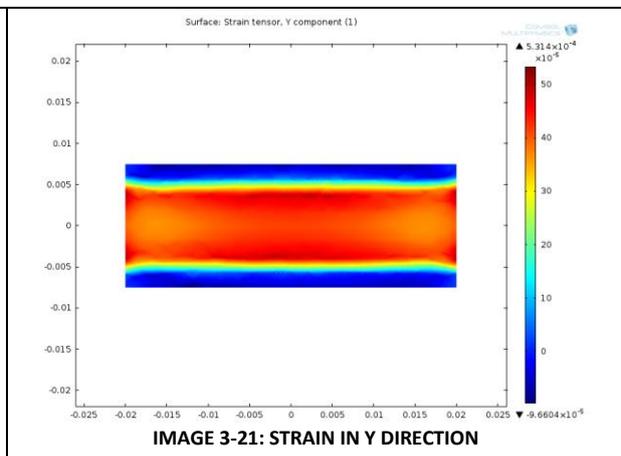
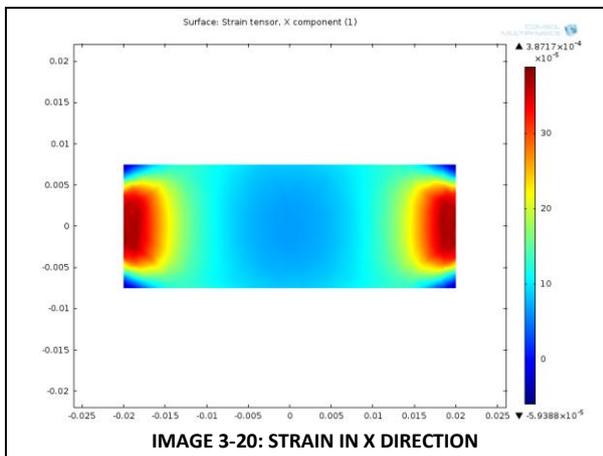
The 1D plots show more clearly that the strain increases to the outside of the pushing area. This could be a boundary effect.





3.8.2 3 LAYER

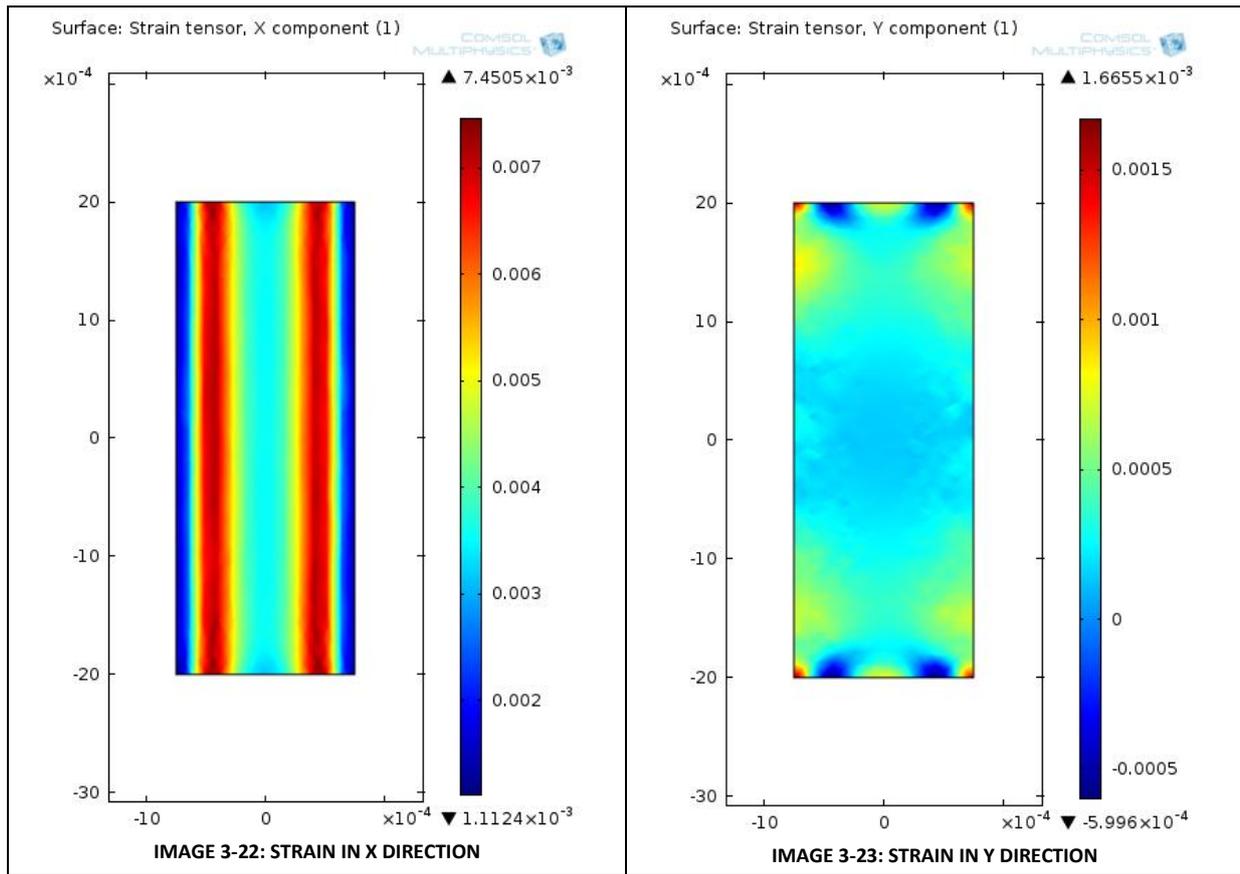
For this simulation the tape is made of 2 layers of copper each 25 μm thick and 1 layer of Hastelloy which is 50 μm thick. The X and Y direction of this model have been changed by accident. Keep this in mind when comparing the results to the other simulations.



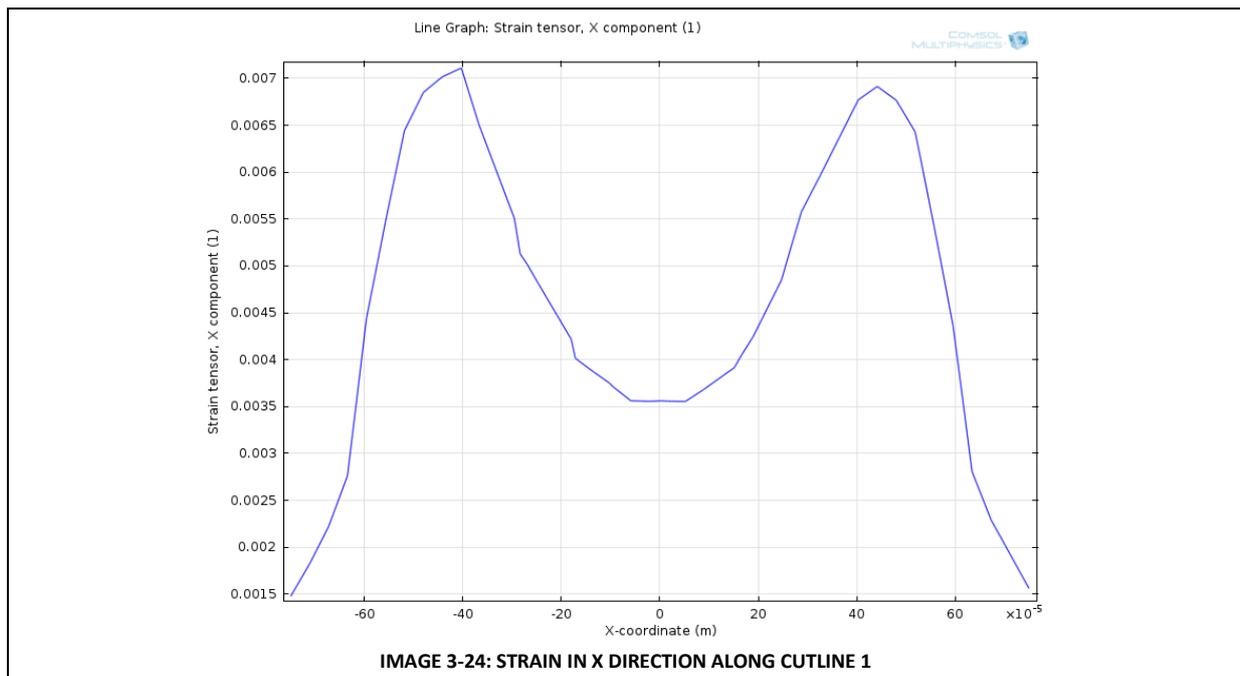
The 2D plot in Image 3-20 and Image 3-21 show the same results as for the 1 layer model.

3.8.3 5 LAYERS

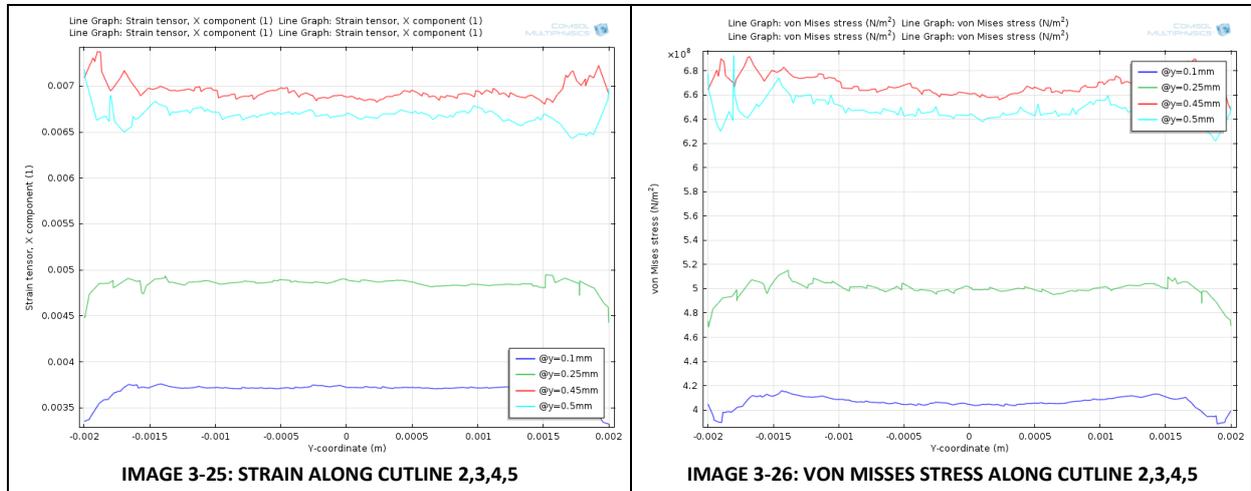
For this simulation the tape is consisting of the following layers, from the anvil up to the pushing head: copper 22 μm , silver 2 μm , YBCO 2 μm , Hastelloy 50 μm , copper 24 μm .



The 2D plot in Image 3-22 is different from the results in the 1 and 3 layer model. The strain increases going outside of the pushing area with the maximum around the edge of the pushing head. The strain in the Y direction shows no strain maximum at the edges. In this case the strain becomes slightly negative. Only in the far ends there is a large positive strain but this is in the unloaded area so this is probably a border effect.



The 1D plot gives more information. The edge of the pushing head is at ± 0.5 mm. The strain increases moving out of the pushing area. The strain reaches a maximum at ± 0.4 mm from the center of the pushing area. From there the strain decreases again.



The strain and stress look pretty constant along the sample width. At the edges there is some variation, probably due to boundary effects.

3.8.4 CONCLUSION OF SIMULATIONS

Damage to the YBCO is most likely found in the pushing area near the edge of the pushing head at 0.4 mm from the center. Damage to the YBCO near the edge of the sample is expected from the 1 and 3 layer models, however the 5 layer model disproves these results.

3.9 ETCHING

To inspect the YBCO layer the silver and copper layers which are above the YBCO should be removed. Delamination as done by Simon Otten & Rudy Lagrauw [12] involves mechanically separating the substrate from the silver and copper layers. This delamination process is very intrusive and can induce additional damage. To prevent damaging the sample we have produced an etching procedure which can be found in Etching procedure at page 31. Etching does not involve mechanical force unlike delamination which makes it a safe way to remove the top layers. However the YBCO can react with some etchants like Ferric Chloride (FeCl_3) and may be (partially) dissolved. Ferric Chloride is used to etch the copper layer; it will not affect the YBCO because it is protected by the silver layer. From N. Cheggour [2] we know a solution of $25\% \text{H}_2\text{O}_2 + 25\% \text{NH}_4\text{OH} + 50\% \text{H}_2\text{O}$ can be used to etch silver without damaging the YBCO. To prevent mechanical forces during etching, storage and damage assessment the sample is glued to a glass microscope slide using sty cast.

3.10 DAMAGE ASSESSMENT

Scanning Electron Microscopy (SEM) is used to find cracks in the super conducting layer. These cracks are $<1\mu\text{m}$ as can be seen in Image 3-27 [2]. So to find them the magnification and resolution has to be very high. The size of these cracks is near the size of visible light at 390 to 700 nm so an optical microscope is unsuitable to find them. The field of view while operating the SEM is about $20 \times 20 \mu\text{m}$ while the pushed area is up to $4000 \times 4000 \mu\text{m}$ big. To help in the damage assessment, a simulation of the pressure on the tape is done. The simulation results are used to find the locations in which damage will occur first. YBCO is highly sensitive to

strain so places where the strain is largest are most interesting. When damage is found using SEM the amount can be quantified and compared with the degradation results from the experiment.

In Image 3-27 there is a SEM picture [2] which shows damage to a YBCO tape which has been caused by transverse stress. The damage is only found near the edge of the sample.

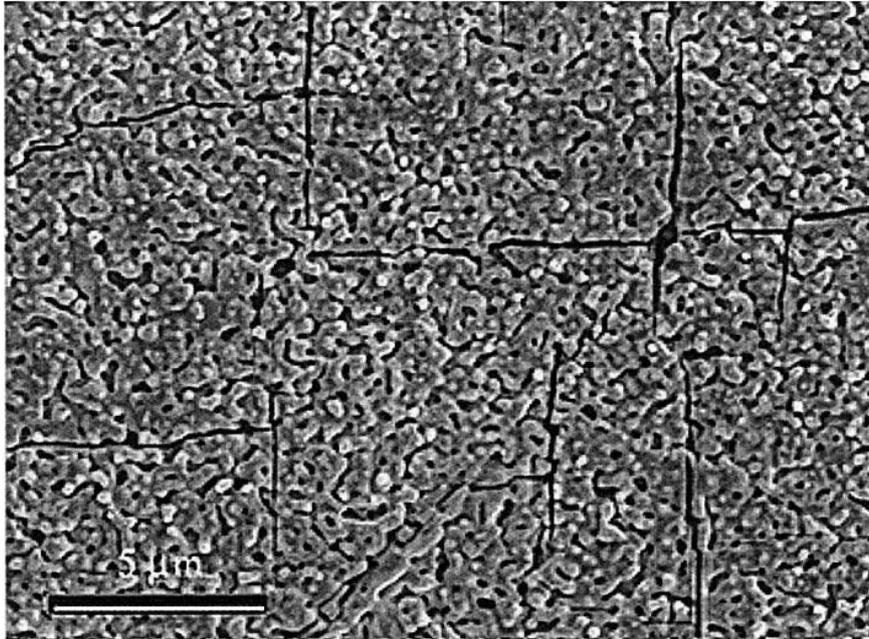


IMAGE 3-27 SCANNING ELECTRON MICROGRAPH OF YBCO FILM ON BUFFERED NI-5AT.%W ALLOY RABITS SUBSTRATE AFTER TRANSVERSE-STRESS MEASUREMENTS, SHOWING FINE MECHANICAL CRACKS LONGITUDINAL AND TRANSVERSE TO THE ELECTRICAL-CURRENT FLOW DIRECTION. THESE CRACKS WERE RARE AND LOCATED MOSTLY NEAR THE EDGES OF THE SAMPLE. THE VERTICAL AXIS OF THE IMAGE COINCIDES WITH THE DIRECTION OF THE ELECTRICAL CURRENT APPLIED TO THE SAMPLE. [2]

4 RESULTS

4.1 4 MM PUSHING HEAD

The 4mm pushing head has only been used to apply stress to the substrate side of the sample.

Sample 1 had a high and probable critical current indicating no problems with it. The measurements were done with a weekend in between, during the weekend the pressure was not released. Too much liquid nitrogen evaporated and the press got heated up. Due to this the press expended and applied a larger (unknown) force to the sample, the measurements after that show high degradation. The results are split up in pre en post weekend measurements.

Sample 5 degraded very fast. The degradation rate was lower than in the other samples. This might indicate the pushing head was blocked with ice resulting in nonparallel pushing. The non-parallel pushing ended when the ice got crushed and a not previously pushed part of the tape got pressed on resulting in instantaneous degradation. Marks on the sample from the pushing head support this theory. To prevent non-parallel loading of the pushing head it is pre-pushed to the sample by small springs.

Sample 6 and 7 are in agreement with the results from sample 1 (before warmup). Samples 6 and 7 show good repeatability.

The results from Lagraauw/Otten show a much higher critical stress (155MPa) than samples 1, 6 and 7. Also the degradation rate is very low. This indicated there was a problem with the press. After the checkup of the press sample 10 was tested. It shows no degradation until at least 300MPa, much higher than from previous results. This means that the anvil and pushing head surface have a large influence on the critical stress. Sample 14 from Lagraauw/Otten shows a lower critical stress, this can be explained by increasing damage to the press from experiments.

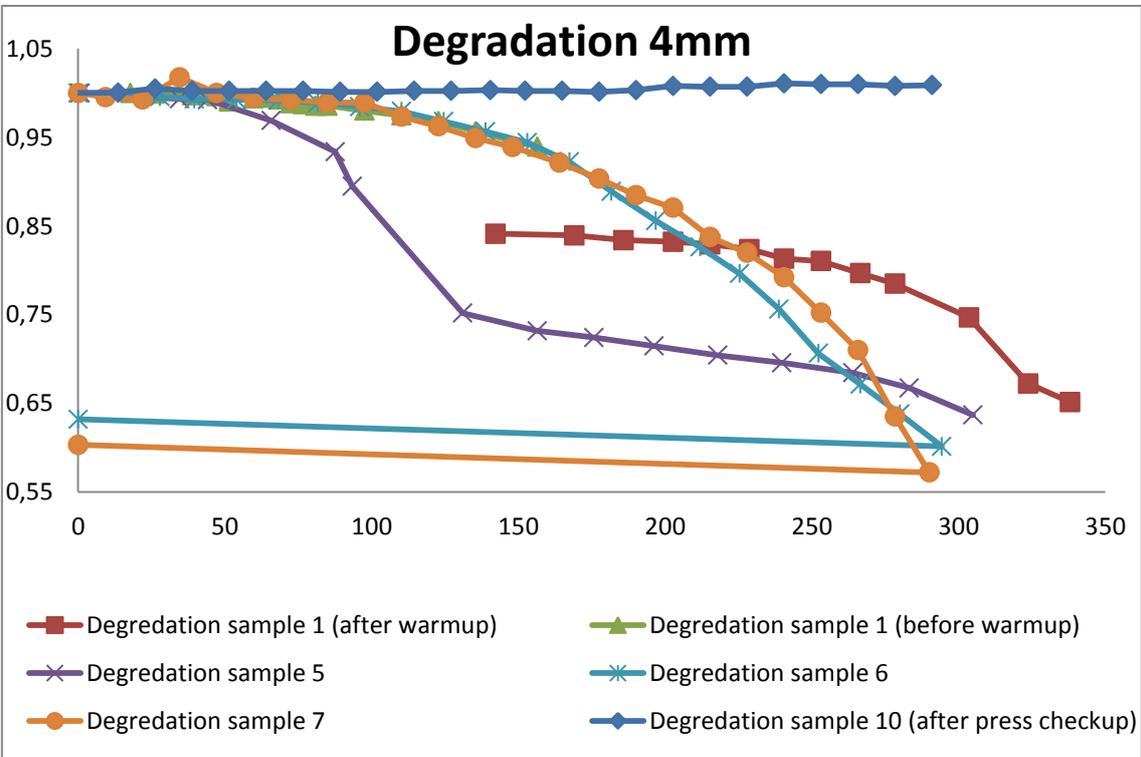


IMAGE 4-1: 4MM PUSHING HEAD BEFORE PRESS CHECKUP, DEGRADATION VS STRESS (MPA)

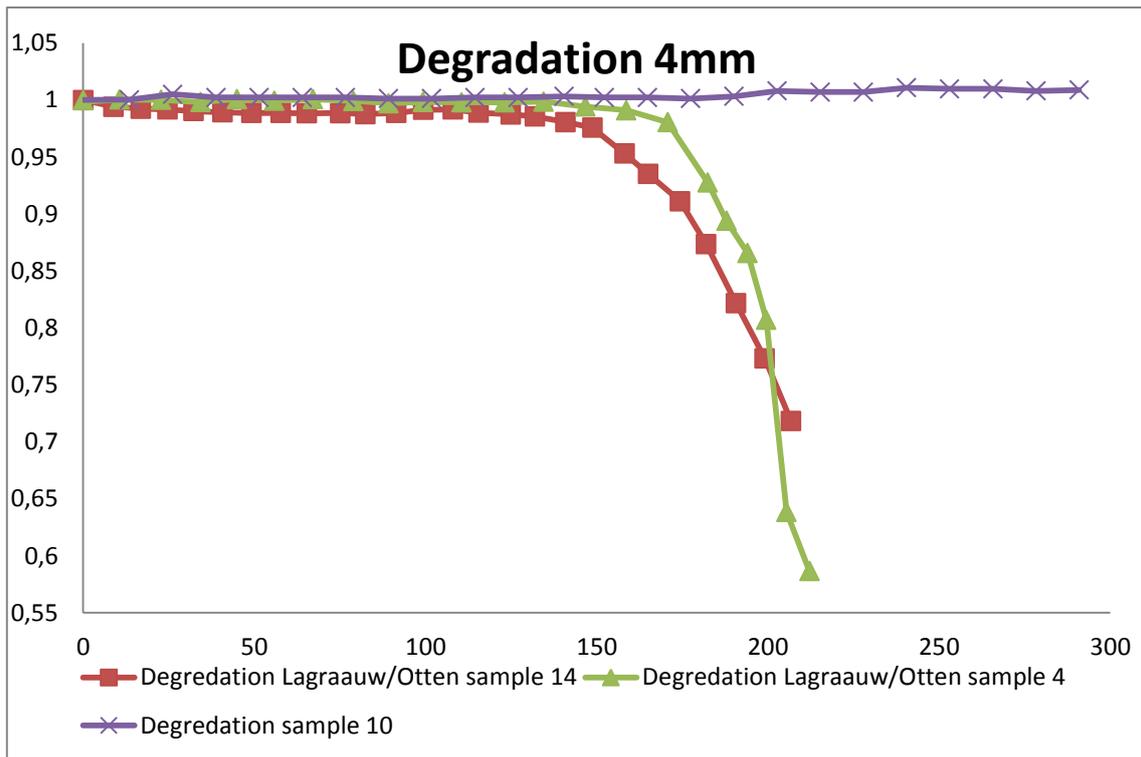


IMAGE 4-2: : 4MM PUSHING HEAD AFTER PRESS CHECKUP, DEGRADATION VS STRESS (MPA)

4.2 2 MM PUSHING HEAD

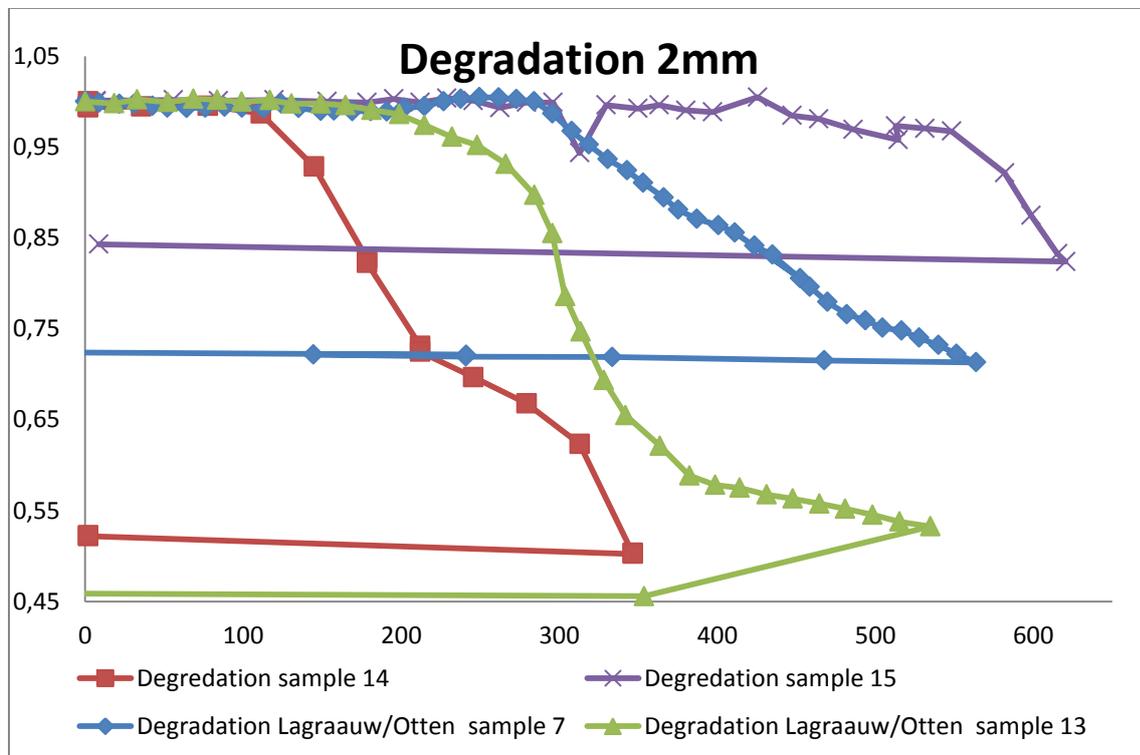


IMAGE 4-3: 2MM PUSHING HEAD, DEGRADATION VS STRESS (MPA)

The 2mm pushing head was looking a bit oxidized. To prevent damaging the pushing head it was not cleaned. The results show how sample 14 has the lowest critical stress. This can be an indication of damage to the

pushing head as the anvil has been polished. The result from Lagraauw/Otten show a decrease of critical stress between samples 7 and 13, this can also be explained by increasing damage to the press after several measurements. Sanding of the pushing head was done to make a flat surface. The sanding was done a bit (to) rough after which the surface of the pushing head became a little bit rounded, the edges of the pushing head were no longer 90 degrees. The result is a much higher critical stress. The experiment needs to be repeated with a new pushing head to verify the results. There is one point on the degradation curve of sample 15 that is a lot lower, this point should have been retested to verify the results. However it had not been noticed before the next measurement was started.

4.3 1 MM PUSHING HEAD

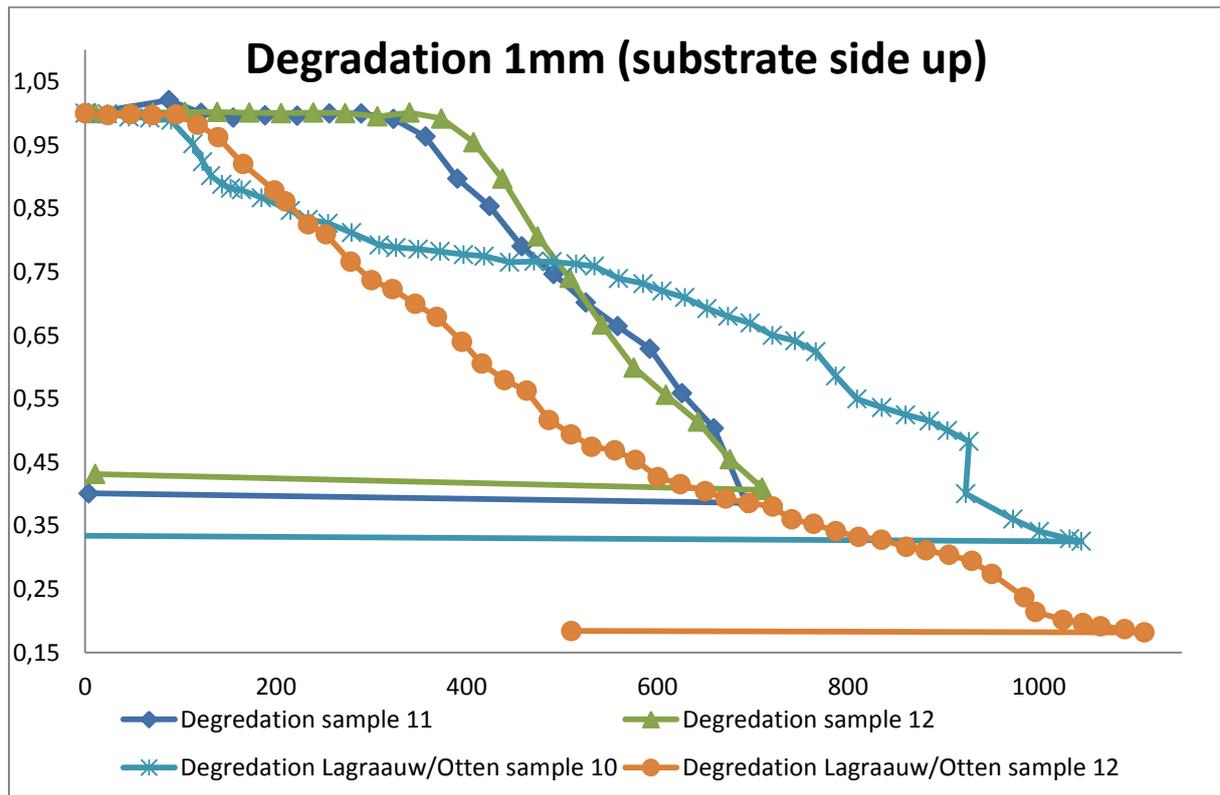


IMAGE 4-4: 1MM PUSHING HEAD, SUBSTRATE SIDE UP, DEGRADATION VS STRESS (MPA)

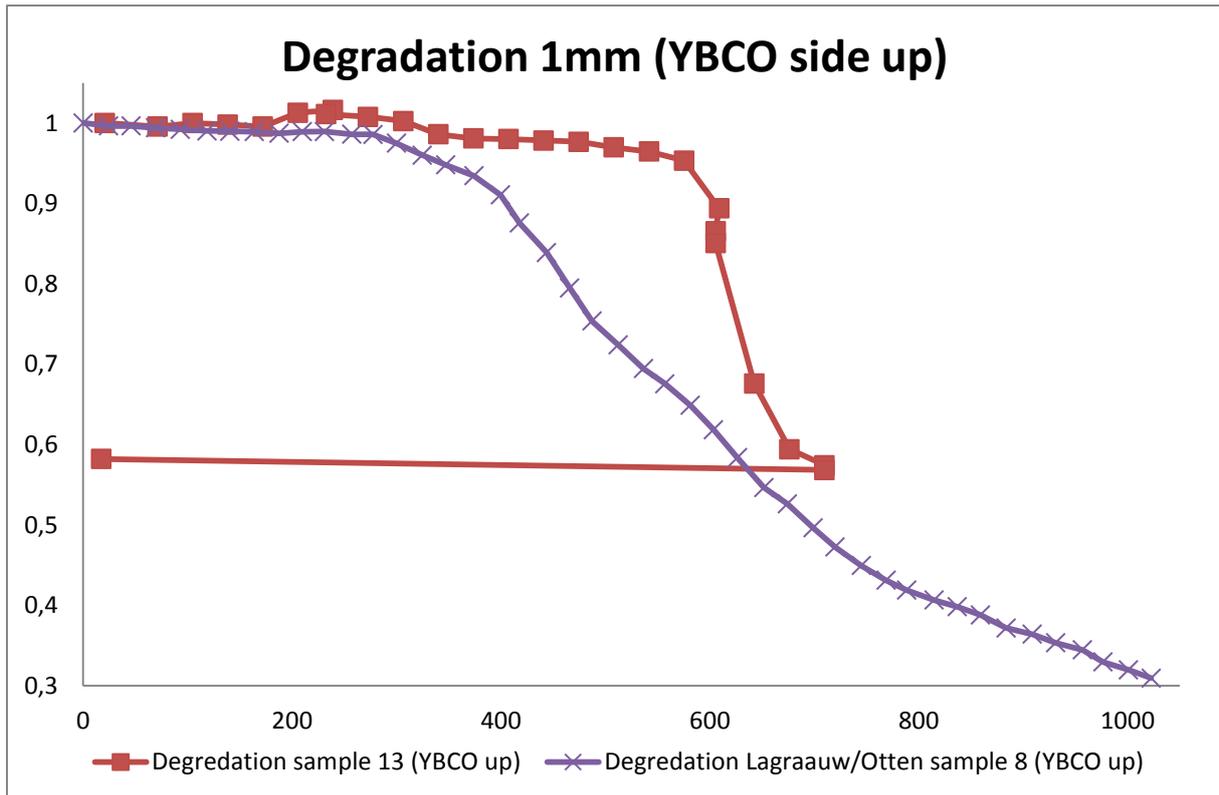


IMAGE 4-5: 1MM PUSHING HEAD, YBCO SIDE UP, DEGRADATION VS STRESS (MPA)

Sample 11 and 12. The results from this experiment are shown together with the results from Lagraauw/Otten. Measurements on sample 11 and 12 shown in dark blue and green were done under the same conditions. The results are very similar and thus the repeatability has improved. The results disagree with the results from Lagraauw/Otten, the critical pressure is a lot higher in this experiment.

Sample 13. For the YBCO side up the critical pressure is higher than for substrate side up.

Again, the lower critical stresses from Lagraauw/Otten can be explained by damage to the press.

On the sample there are some marks caused by the applied load. These marks suggest the force applied to the sample is non uniform. This is properly caused by the sample thickness which is not homogenous. The sample is thickest in the center and thinner on the edges. This means the effective pushing area is smaller than expected and so a higher stress is exerted on a fraction of the tape while the rest of the tape will be subjected to a lower or even no stress.

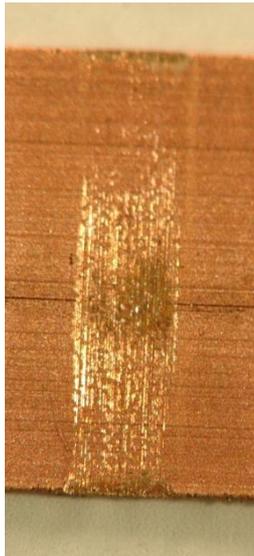


IMAGE 4-6: DAMAGE ON SAMPLE SURFACE, PUSHING HEAD SIDE



IMAGE 4-7: DAMAGE ON SAMPLE SURFACE, ANVIL SIDE

4.4 DAMAGE ASSESSMENT

There damage to the YBCO can be divided in damage on the sample edges and damage on the edge of the pushing area.

4.4.1 EDGE OF SAMPLE

In the images below the sides of the sample are shown in the pushing area. Damage is observed on one side while none is on the other side. This indicates the load applied by the press is not homogenous. Cracks on the side of the sample are mostly oriented parallel to the length direction of the tape with some smaller cracks perpendicular. There are multiple cracks which are parallel to each other. The damage observed on the edge is comparable to the results seen in Image 3-27.

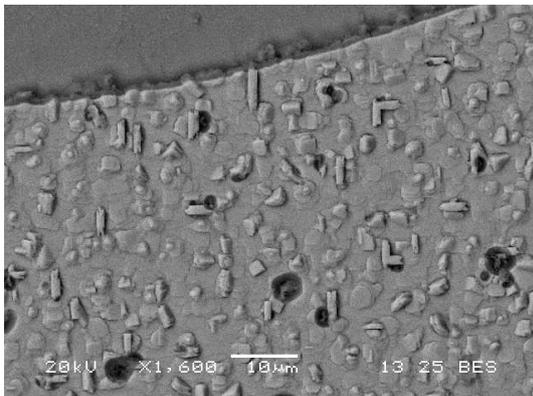


IMAGE 4-8: UNDAMAGED SIDE OF SAMPLE

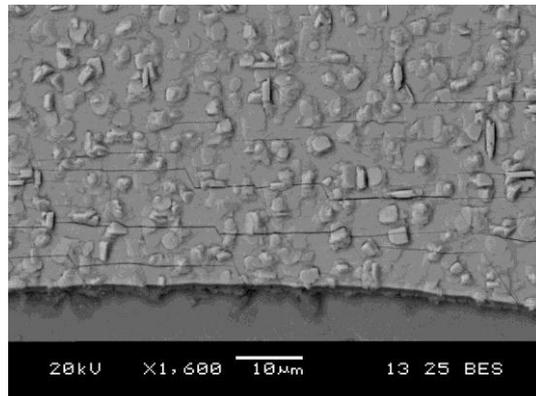


IMAGE 4-9: DAMAGED SIDE OF SAMPLE, CRACKS ARE BOTH PERPENDICULAR AS PARALLEL TO TAPE LENGTH

4.4.2 EDGE OF PUSHING AREA

Along the sides of the pushing area in the center of the sample, cracks are observed perpendicular to the length direction of the tape or parallel to the pushing area border. There are multiple cracks which are parallel to each other. There are no cracks observed parallel to the sample length direction. The observed damage is similar to the damage from tensile strain as discussed in 2.6 on page 10. This confirms the results from the simulation.

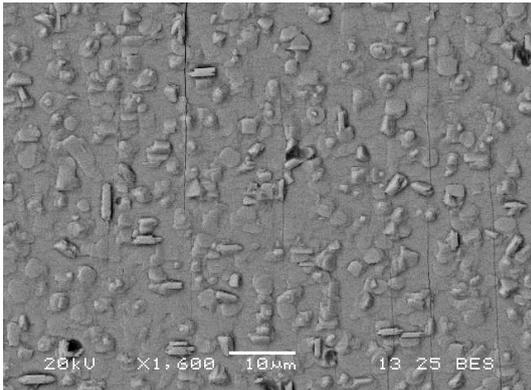


IMAGE 4-10: CRACKS ALONG THE SIDE OF THE PUSHING HEAD

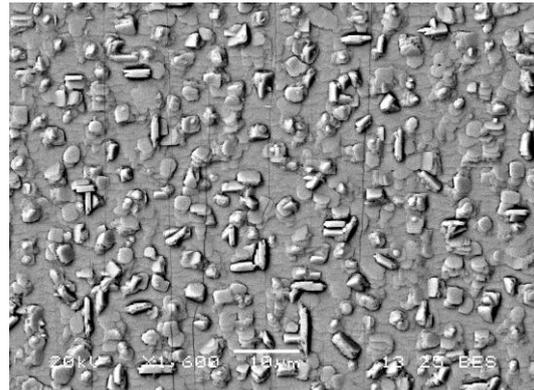


IMAGE 4-11: SOME MORE CRACKS ALONG THE SIDE OF THE PUSHING HEAD

5 DISCUSSION

The pushing head has to apply a homogeneous load. If the surface of the pushing head is not parallel to the anvil it could cut into the sample on one side or create a pressure concentration. Observation of the press with sheet copper and paste did not reveal any measureable problems. However "scratch" marks on the samples indicate the press is not applying a homogenous load. To prevent non parallel pushing, springs have been fitted that hold the pushing head on the sample with a small force which allows the operator to align the pushing head parallel to the sample. The effective pushing area can be smaller due to non-flat pushing head, anvil or sample. The marks on the samples and the cracks in the YBCO indicate the sample has been loaded unevenly; this means the effective stress was higher because the pushing area was smaller. As the stress increases the copper deforms, as a results the pushing area could increase. Bumps can cause local force in-/de-crease.

To prevent damage during soldering, the current leads were realigned to reduce the gaps between the current leads and the anvil. There will always be some small gaps and height differences which could cause damage.

5.1 MEASURING ERRORS

The distance between the voltage tabs has been measured using calipers. The distance between the tabs is about 10 mm, the measuring error 0.05 mm. Any errors in measuring the distance between the tabs results in a lower or higher field. The result of this is an over or under rated critical current, this would explain the variation in critical current between samples. However the normalized degradation is not affected by the measuring error.

There was damage to the cable connecting the setup to the meters. The cable was then replaced by a new one connected with a 7 pin connector to the old cable. The added connector acts as a thermal couple which means the voltage to the meter gets affected by thermal variations to the connector. Temperature changes during measurements could lead to too high or low fields measured. To prevent this; the connector is taped to a thermal mass and insulated from the environment to prevent sudden temperature changes.

6 CONCLUSION

The critical stress at which critical current degradation occurs is higher for loads applied to the Hastelloy side of the tape than for loads applied to the YBCO side.

The critical stress observed in all experiments is higher than in previous results from Lagraauw/Otten. Results from checking the press indicates the results are lower because of damage to the anvil and/or pushing head.

The load of the press on the sample is non-homogenous. This is caused by the non-uniform thickness of the tape. The press or samples need to be modified to apply a more homogeneous load.

Damage to the YBCO occurs along the edge of the pushing head. The cracks are parallel to the side of the pushing head. The cracks start at the side that has been stressed more as can be observed by the marks on the tape after loading. The cracks are transverse to the tape length direction; this is similar to the cracks formed by tensile strain. This indicates the mechanism that causes damage in these areas is tensile strain. The results from the simulations predict high tensile strain in these location confirming the results.

Damage to the YBCO occurs on the side of the tape, the side where damage occurs is where the stress was applied as can be seen by the marks on the tape after loading. The other side does not appear damaged. The cracks are both transverse as longitudinal to the tape length direction. The simulations indicate there is tensile strain in both these directions which could cause a superposition of cracks perpendicular to each other. The results from the simulations do not predict very high strain in these areas. From this it is like that tensile strain is causing the damage.

7 APPENDIX

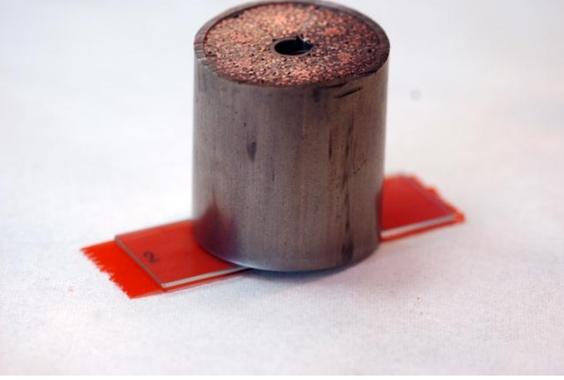
7.1 ETCHING PROCEDURE

1. Prepare your samples for etching.

The tape samples should be prepared first before etching. To remove the top layers of Cu and Ag which are cover YBaCuO layer chemical etching is used. To do it the sample first needed to be prepared. First the voltage tabs and solder from it should be removed by using desolder lint.

1. **Glue sample to a glass.**

<p>The glass needed to be cover with a sticky tape.</p>	
<p>The sample should be glued to another piece of sticky tape. The sample should be glued to the tape by YBaCuO side down, hastelloy side up.</p>	
<p>A hole should be cut through the sticky tape on the glass. The hole size should be approximately the size of the sample to etch.</p>	

<p>Some amount of stycast should be applied to cover the hole. The excessive amount of stycast is removed by another glass edge.</p>	
<p>The sticky tape can be removed now.</p>	
<p>The sample on the sticky tape should be aligned with stycast on the glass and glued.</p>	
<p>To straightened sample one should put the other piece of the glass on the top of the sample and apply some load. Let the stycast dry.</p>	
<p>Remove sticky tape. The samples's surface should be cleaned with alcohol. Be sure no stycast left on the sample surface, if there is some remove it by scraping with blade. If big amount of stycast is on the sample's surface it can be removed by make it soft first in acetone. It takes about an hour to soften stycast.</p>	

Sample is ready for etching.

2. Etching

The copper layer is removed by Iron chloride solution (FeCl_3). Approximately 7 grams of 97% Iron chloride powder must be dissolved in 100 ml. water. Use hot water, it greatly reduce time of copper etching. For fresh solution it takes about 3 – 6 min. to etch copper. The time depends from solution concentration and temperature.

It's useful to shake the solution during etching, because the near-surface layer quickly saturates with copper and the process become painfully slow.



The samples must be extensively flushed with water after copper etching.

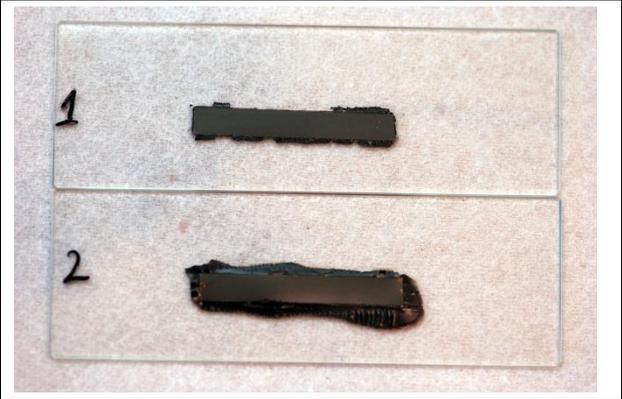


The silver layer is etched by solution of hydrogen peroxide 35% with water and ammonia. The ratio is 1:1:1. It's better to add a cold water to hydrogen peroxide and then add ammonia, because mixing hydrogen peroxide with ammonia leads to self-heating process.

Silver etching procedure in fresh solution takes about 15 – 30 sec. There is no need to shake a solution.



After silver etching samples needed to be extensively flushed with water to remove residual etching chemicals.



7.2 SOLDERING PROCEDURE

TOOLS

- Soldering station with temperature control and a fine tip
- Calipers
- 40Pb-60Sn Rosin core solder
- Copper flux
- Iso-Propanol
- Cotton tips
- Transparent tape
- Teflon or glass surface
- Plastic coated copper wire, $0.5 \leq \text{diameter} \leq 1 \text{ mm}$
- Kapton

IMPORTANT NOTICE

- For soldering the voltage tabs and pre-tinning set the temperature of the soldering station at less than 300 C
- Temperatures over 250 C can damage the YBCO, when applying temperatures of over 250 C try to do it less than 30 seconds to prevent damage.

TIPS

- Grease and other dirt can make soldering and making markings hard, use gloves to prevent staining the sample.
- The sample can move a lot when you try to measure and solder. Use transparent tape to fix it to a flat surface.
- When soldering the tabs the sample may stick to the surface it's placed on. Using Teflon or glass as a surface will prevent this.

PREPARING THE SAMPLE

1. Clean the sample with iso-propanol to remove grease and other dirt.
2. The YBCO is (probably) on the outside of the reel. Mark this side of the tape on the end with the letter Y to prevent mistakes about which is the right side.
3. Cut a sample of 35 mm length using sharp scissors. Use a permanent marker to indicate where to cut.
4. On the side of the sample where you want to apply the force, mark the center of the length. You can do this by measuring the exact sample length and make a mark at half this distance from one end.
5. Make a mark 5 mm from both sides of the center. In these locations the voltage tabs will be soldered so they are 10 mm from each other. After marking measure the distance between the marks. If the distance is not 10 +/- 0.5mm remove the markings with iso-propanol and try again.
6. Make a mark 10 mm from both sides of the center. In these locations the quench detector tabs will be soldered. After marking measure the distance between the marks. If the distance is not 20 +/- 1 mm remove the markings with iso-propanol and try again, be careful to not remove the voltage tab markings when you do.
7. If the sample has gotten dirty; clean the surface of the tape where the voltage and quench detector tabs will be soldered using Iso-Propanol on a cotton tip without removing the markings.
8. Apply a very small amount of copper flux to the cleaned surface of the sample. Keep the diameter of the spot under 0.3 mm.
9. Pre-tin the sample in the fluxed areas using as little tin as possible. The spot should be kept as small as possible (under 0.3 mm diameter).
10. Remove any flux residue with a tissue.
11. Prepare voltage tab wires, cut 4 pieces of plastic coated wire of 0.1 mm diameter and at least 5 cm in length (for easy handling).
12. Pre-tin the 4 wires on one side.
 - a. Make a large drop of solder on the soldering iron and use a high (+350°C) temperature.
 - b. Put the last 5 mm of the wire in the hot solder for at least 5 seconds to burn the plastic coating and pre-tin the wire. The wire end should be silver colored now.
13. Solder the wires to the pre-tinned spots without increasing the area of the joint.
 - a. The wires should touch the sample surface
 - b. The wires ends should be straight at the solder joint.
 - c. The wires should be oriented along the width of the sample.
 - d. The joint should be as flat as possible(to prevent the pushing head sides to apply a force on the solder joints, measure to be sure).
14. Measure the distance between the wires very accurately, you will need these for calculating the field in the conductor.
15. Pre-tin the ends of the YBCO side of the sample.
 - a. If necessary, lean the surface of the sample using iso-propanol and a cotton tip.
 - b. Apply copper flux to the last 5 mm of the sample on both sides.
 - c. Pre-tin the ends of the sample.

SOLDERING THE SAMPLE TO THE CURRENT LEADS

16. Clean the current leads.
 - a. Heat the current lead with a 200W soldering iron until the solder on the lead is flowing.
 - b. Use a wet brush to remove dirt and old solder. The sample is held put inside 2 blocks that are on top of the current leads, remove solder until the square shape of these blocks becomes visible again.

17. Align the sample in the setup and use tape on one end to prevent moving of the sample during soldering. Put a piece of kapton in between the anvil and the sample to prevent big thermal gradients or sharp turns of the sample over the anvil.
18. Heat the current lead until the solder is flowing. The sample should not touch the current lead before the solder is flowing to prevent damage by heating
19. Push the sample straight down on the current lead with the wood stick and remove the soldering iron.
20. Use a wet brush to cool down the current lead faster.
21. When the solder is not flowing anymore you can remove the pressure on the sample applied with the wood stick.
22. Repeat steps 2 to 6 for the other current lead.
23. Remove the kapton that separates the sample from the anvil.
24. Cut the voltage and quench detector leads to size (be careful to not make them too short).
25. Pre tin the leads like in step 11.
26. Connect the voltage and quench detector wires to the press. There is a soldering tab on the press. The outside cables are for the quench detector, the inside wires are for the voltage meter.
27. Make sure everything is connected properly before cooling down, the voltage of the quench and voltage meters should give a very small voltage. If one of the meters shows a high voltage or overflow there is probably a bad connection.

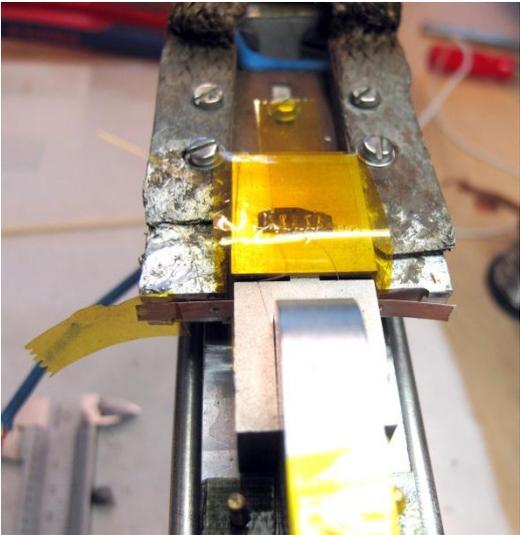
PICTURES



Pre-tinned side of sample that will be connected to current leads as described in step 15.



Voltage tabs connected to sample as described in step 14.



Sample placed in setup in preparation of soldering as discussed in step 17.



Sample soldered to current leads and voltage tabs connected

7.3 LIST OF UNCOMMON WORDS

Epitaxy refers to the deposition of an overlayer on a crystalline substrate, where the overlayer is in registry with the substrate. (Some researchers also think there must be one or more preferred orientations of the overlayer with respect to the substrate for this to be termed epitaxial growth). The overlayer is called an epitaxial film or epitaxial layer. The term *epitaxy* comes from the [Greek](#) roots *epi*, meaning "above", and *taxis*, meaning "in ordered manner". It can be translated "to arrange upon". For most technological applications, it is desired that the deposited material form a crystalline overlayer that has one well-defined orientation with respect to the substrate crystal structure (single-domain epitaxy). Epitaxial films may be grown from [gaseous](#) or [liquid](#) precursors. Because the substrate acts as a [seed crystal](#), the deposited film may lock into one or more crystallographic orientations with respect to the substrate crystal. If the overlayer either forms a random orientation with respect to the substrate or does not form an ordered overlayer, this is termed non-epitaxial growth. If an epitaxial film is deposited on a substrate of the same composition, the process is called homoepitaxy; otherwise it is called heteroepitaxy.

Quench is the event in which a superconductor enters the resistive state. This spot will be subjected to rapid joule heating which raises the temperature of the conductor and its surroundings will rise.

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