

n-values of commercial YBCO tapes before and after irradiation by fast neutrons

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Abstract

The n-value is an important superconducting parameter, which represents the homogeneity of characterized superconductor as well as thermally activated depinning. In addition n-values are important for the evaluation of pinning mechanisms and pinning forces. n-values are crucial input parameters for the numerical simulations of superconducting tapes, coils and other complicated superconducting applications where E-J power law applies. In this publication, complex measurement data of n-values from different 2nd generation of high temperature superconducting (2G HTS) tapes are presented and analysed. In addition, 2G HTS tapes were step by step irradiated by fast neutron fluences up to $1 \times 10^{22} \text{ m}^{-2}$. n-values of the irradiated tapes, containing additional randomly distributed pinning centres, are presented, analysed and compared with unirradiated samples. Special attention is placed on the underlying physics resulting in power-law part of the I-V curve and on the correlation between critical currents and n-values. The measurements are performed within the temperature range of 50 K-85 K and magnetic fields up to 15 T.

I Introduction

Each I-V curve of a superconducting sample contains a power-law part close to the transition to the dissipative state. This part of the I-V curve can be described by a simple equation:

$$V/V_c=(I/I_c)^n \quad (1)$$

where V and I are the measured voltage and current, V_c is the voltage criterion, I_c is the critical current and the n -value the exponent n . Even though the n -value is an important superconducting parameter, results on 2 G HTS tapes are available only in a few recent publications [1-5]. Therefore, a comprehensive overview of experimental n -value data would be beneficial when dealing with numerical modelling of superconductors. In low-temperature superconductors, the exponential relationship (power-law part) in I - V is usually explained by the J_c nonuniformity. Warnes and Larbalestier [6] and Plummer and Evetts [7] successfully developed models based on the J_c nonuniformity, but such a mechanism is not so successful in high temperature superconductors. Magnetic flux creep is a mechanism introduced by Anderson and Kim [8,9] and describes a concept of thermally activated flux lines released from pinning centres. This phenomenon is mainly observed at higher temperatures where HTS operate. The rate of this process (R) is exponential, depending on the temperature of the superconductor according to the relation:

$$R \propto \exp\left(-\frac{U_e}{kT}\right) \quad (2)$$

where U_e represents activation energy, k – Boltzmann’s constant, T – absolute temperature. This process leads to the redistribution of flux lines resulting in a decrease of the magnetic moment of the superconductor, which is known as magnetic relaxation. Magnetic relaxation in HTS was reviewed by Y. Yeshurun et al. [10], where the direct relation between flux creep and the power law relationship in the I - V curve was pointed out. Finally, studies by J.Z Sun [11] and R. Griessen [12] provided evidence that the power-law relationship is a consequence of the flux creep. However, both effect, nonuniformity and the flux creep, do not exclude each other and can collaboratively contribute to the exponential part of the I - V curve at the same time. Nowadays, flux creep is considered as the primary reason of the power-law relationship for high temperature superconductors even though some older publications suggested otherwise (i.e. nonuniformity or inhomogeneity) [13,14]. It means that even a “perfectly uniform” HTS would have the power law part of the I - V curve. The n -value of the “perfectly uniform” HTS can be even relatively low in the case of efficient pinning centres with small activation energy. Nonuniformity is not necessarily present in large scale in modern 2G HTS tapes. Generally, the influence of the uniformity factor to the n -value is more important at lower

temperatures. With increasing temperatures and magnetic fields the uniformity factor becomes less important and importance of flux creep increases. As assumed by the uniformity based models [13,14], critical currents are limited by grain boundaries and not grains. The transition between grain boundary and grain limited currents could be a threshold, where nonuniformity is not the main reason of the power-law relationship. In any case, the n -value always strongly depends on the efficient pinning mechanism in the superconductor.

Measurements of n -values after introducing additional pinning centres require special attention. Irradiation by fast neutrons introduces randomly distributed spherical pinning centres with diameters of a few nm [15-20] into a superconductor. It is a proven method of introducing efficient pinning centres into HTS, resulting in critical currents enhancements. Significant critical current enhancement was reported in rather stronger magnetic fields and lower temperatures for several types of tapes [20-22].

Regarding the HTS applications, high n -values reduce the losses by operation close to the J_c and are necessary to operate magnets in the persistence mode. At the same time, high n -values are also associated with unstable behaviour which can cause premature quenching in superconducting machines [23]. Usually, high n -values are considered as an asset of superconductors and rapid n -value reduction at higher magnetic fields can make them unsuitable for any applications. The n -value can vary significantly in different kind of tapes as well as after irradiation. Therefore, measurements were performed on commercial 2G HTS tapes from three different manufacturers and also after irradiation by fast neutrons.

II Samples

Standard tapes from three different manufacturers were used. The first is the 2G HTS SCS 4045 tape from SuperPower, which is made by Metal Organic Chemical Vapor Deposition (MOCVD) on an Ion Beam Assisted Deposition (IBAD) made MgO template [24]. The YBCO layer is 1 μm thick. Two set of samples are characterized, both sets are the SCS 4045 tapes manufactured in 2008 and 2012 respectively. The size of YBCO grains in this kind of tapes is $\sim 1 \mu\text{m}$. The second series of samples are from American Superconductor (AMSC). These tapes have RABiTS[®] (Rolling Assisted Bi-axially

Textured Substrates) substrate and Metal Organic Deposited (MOD) YBCO layer ($\sim 1\mu\text{m}$). The 4 mm wide tape is marked as 344 and the 12 mm wide tape as Amperium 8612, with a double HTS Layer [25]. The usual size of YBCO grains in RABiTS[®] tapes is 20-50 μm [26]. The third series of samples is coming from Shanghai Superconductor Technology Co.[27]. In these tapes, the MgO template is made by IBAD and Pulsed Laser Deposition (PLD) is used for $1\mu\text{m}$ thick YBCO layer. The best tested 4 mm wide sample reached the highest value of critical current. More information about all the samples are listed in Table 1. All the used 4 mm wide samples were 26 mm long and the 12 mm wide samples were 80 mm long.

III Instrumentation

All the presented instruments are adjusted for transport current characterization of short samples (apx. 3cm length) by a standard 4 point method. As data for this study are obtained from several experiments performed by different devices in different laboratories (Low temperature and superconductivity laboratory at Vienna University of Technology, Atominstitut, and EPEC superconductivity laboratory at the University of Cambridge). Most of the samples were characterized by several experimental set-ups. A brief description of experimental instrumentation is given below:

The electromagnet set-ups

Two electromagnet measurement set-ups were employed for angle-resolved transport measurements, the first one was in Vienna (Atominstitut) and the second one in Cambridge (EPEC superconductivity group). The first set-up consisted of a 1.4 T water cooled electromagnet and the second of a 800mT electromagnet where bigger area of homogenous field can be achieved. As for cryostats, a simple tubeshaped vacuum vessel (flask) and a polystyrene box were used. All the measurements were performed in liquid nitrogen. Both set-ups are designed for angle-resolved transport measurements in the maximal Lorentz force configuration. The main difference between the two measurement set-ups is the rotation mechanism. While on the first set-up the holder is statically mounted between the rotating magnet poles, while the holder in the second set-up is rotating and the magnet poles are

stable. Maximum achievable resolution on both systems is 0.5° . Both sample holders are equipped with Hall sensors and they produce identical results. The Cambridge was used preferred for wider samples reaching very high currents and the irradiated samples were characterized exclusively in Vienna due to safety certificates.

6 T measurement set-up

The 6 T measurement set-up is a helium gas flow cryostat equipped with a 6 T split coil. The main advantage of this cryostat is a wide temperature range of measurements from about 4.2 K to 150 K. The horizontal magnetic field allows to perform angle-resolved transport measurements with a rotating sample holder. The rotating sample holder is equipped with a sensitive Hall probe, a Cernox temperature sensor and a fine rotating mechanism with a stepper motor (0.1° precision). Characterized samples can have lengths up to 30 mm. Indium press contacts are typically used for current and the conductive silver glue is used for the voltage contacts.

17 T measurement set-up

The 17 T measurement set-up is a helium flow cryostat which is equipped with superconducting coils, generating magnetic field up to 17T in vertical direction. The Variable Temperature Inset (VTI) has an inner diameter of about 3 cm and the magnetic field is homogeneous in a vertical length of about 30 mm. These parameters limit the maximum samples lengths to 30 mm. Two sample holders designed for short tapes characterization are available. One places the sample with the *ab* plane and the other with the *c*-axis parallel to the magnetic field. A 300 A current source was available for the transport characterization.

Triga Mark II reactor

The TRIGA Mark II was used as an irradiation facility in this work. It is a pool type research reactor that is used for training, research and isotope production (TRIGA - Training, Research, Isotope production, General Atomic) [28]. The reactor has a maximum continuous thermal power of 250 kW, though the power can be increased up to 250 MW for about 40 ms in the pulse regime. The fuel is in

the form of an uniform mixture of 8 wt% uranium, 1 wt% hydrogen and 91 wt% zirconium, where the zirconium-hydride is being the main moderator. The maximum neutron flux density of $10^{17} \text{ m}^{-2} \text{ s}^{-1}$ at 250 kW is reached in the Central Irradiation Facility (CIF). The sample's temperature is estimated to remain below 50 °C during the irradiation procedure.

IV Results

Tapes in low magnetic fields

All the measurements in this section are performed by the electromagnetic measurement set-ups in liquid nitrogen in fields below or equal to 400 mT. As all studied commercial tapes have good grain alignment, the transition from grain boundary limited currents to grain limited currents must occur in this field range, according to [22,29] even well below 400mT. As at low fields the grain boundary limited currents may occur, both nonuniformity and flux creep must be taken into account as the reasons of the exponential part of the I-V curve. It is also important to mention that all the measurements are evaluated by a voltage criterion of 1 $\mu\text{V}/\text{cm}$ as U_c is a locked parameter by fitting with eq.(1). 4 mm tapes and wider 12 mm tapes from the three different manufacturers were characterized. In addition, two sets of 4mm wide samples from SuperPower were characterized. Critical currents as well as n-values of all types of characterized tapes are listed in table 1. Results from both, newer (2012) and older (2008) tapes are available in figure 1. The results from 4mm AMSC and SHSC tapes are shown in figures 2 and 3, respectively. All the angle-resolved measurements were performed in one of the introduced electromagnet measurement set-ups. The results of the n-values are shown together with figures of critical currents for a better illustration of critical current behaviour. Figures with n-values are generally noisier than the figures with critical currents as a consequence of fitting algorithm. n-values and critical currents were calculated from the power law fit of the exponential part of the IV curve. The beginning of the exponential part of the IV curve was determined by first three points above the noise level. The same algorithm was applied for all the measurements from all used measurement set-ups.

Table 1: Critical currents and n-values of the samples at 77 K and self-field.

Sample	Critical current 77 K self-field	n-value
SuperPower(2008) 4 mm	98 A	28.8
SuperPower(2012) 4mm	114 A	30.5
SuperPower(2012) 12mm	389 A	30.1
AMSC 4mm	92.5 A	36
AMSC 12mm	534 A	52.2
SHSC 4mm	167.5 A	42.14

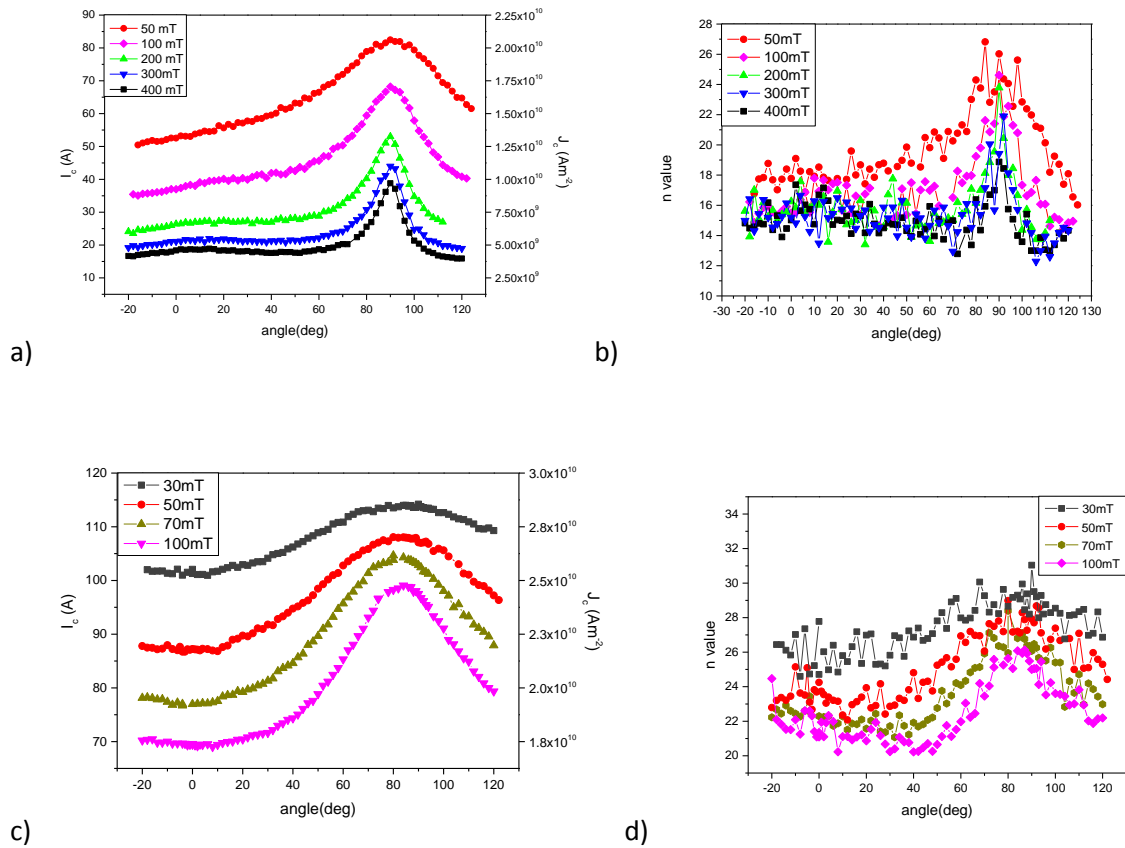


Figure 1: a) b) Critical current and n-values of the SuperPower tape (2008) at 77 K, c) d) critical current and n-values of the SuperPower tape (2012) at 77 K.

By comparing of the result at 100 mT between SuperPower(2008) and SuperPower(2012), the increase in critical current is between 45 % (at $\sim 90^\circ$) and 86% (at 0°). However n-values at 90° stayed practically unchanged (difference of $\sim 5\%$) and the difference at 0° is only about $\sim 33\%$.

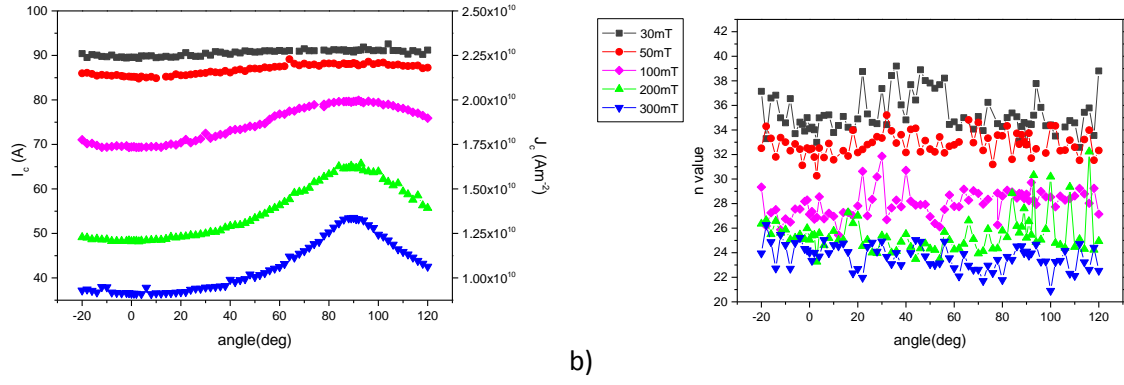


Figure 2: 4mm AMSC tape at 77K: a) critical currents b) n-values

Surprisingly, n-values of the AMSC tape are not dependent on the angle of magnetic field, even though some correlation between n-values and critical current exists also in RABiTS AMSC tape as presented in [2]. Figures 2 and 5 are even showing signs of inverse J_c - n-value correlation, which will be discussed later in this paper. In numerous experimental studies, it has been observed that n-values are usually correlated with critical currents [1,2,30], which causes that n-value varies also with external magnetic field. However, if the correlation is a consequence of relation presented by Zeldov et al. [31], then:

$$U_e = U_0 \ln\left(\frac{J_{c0}}{J}\right) \quad (3)$$

where U_0 is the $J = 0$ activation energy (pinning energy), J is current density and J_{c0} is the critical current density in absence of thermal activation. From (2):

$$V \propto \frac{dM}{dt} \propto \exp\left(-\frac{U_e}{kT}\right) \quad (4)$$

where dM/dT is the rate of magnetization change. Thus, a simple substitution:

$$V \propto \frac{J_{c0}}{J} \exp\left(-\frac{U_0}{kT}\right) \propto \exp\left(\ln\left(\frac{J_{c0}}{J}\right) \frac{U_0}{kT}\right) \propto \left(\frac{J_{c0}}{J}\right)^{\frac{U_0}{kT}} \propto J^{\frac{U_0}{kT}} \quad (5)$$

$$n \propto \frac{U_0}{kT}, V \propto J^n \propto I^n \quad (6)$$

It is shown that n-value is directly proportional to the pinning energy of flux and not to the J_c . Thus $J_c = f(U_0)$, according to (3):

$$\ln\left(\frac{J_{c0}}{J_c}\right) = \frac{U_{ec}}{U_0} \quad (7)$$

U_{ec} is activation energy at the critical current. It can be assumed that $(J_{c0} - J_c)$ is higher at higher temperatures and converging to zero at very low temperatures. If J_c is a function of (ϕ, H, T) according to (7), then U_0 is also a function of (ϕ, H, T) , however, the angular dependence of U_0 in an external magnetic field can be quite different than the one for the J_c . This can be a consequence of flux line deformation e.g. into staircase-like shapes [3,4]. Nonuniformity might play a role in the uncorrelated dependence as well.

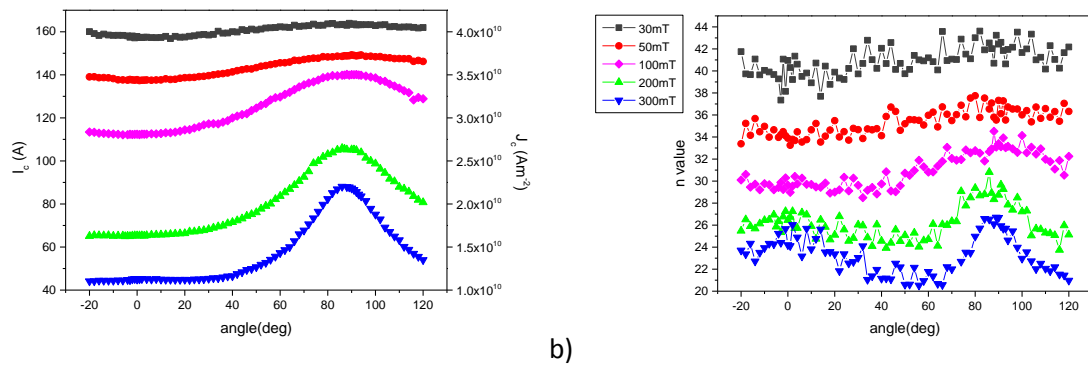


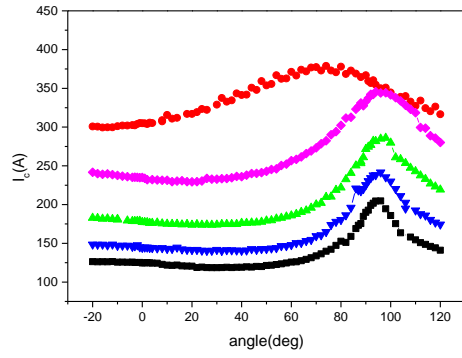
Figure 3: SHSC 4mm wide tape at 77 K a) critical currents b) n-values

The SHSC sample has shown superior properties. Critical currents were significantly higher than in the other characterized tapes. Although n-values were very high at 30 mT, in higher fields they were comparable with tapes from other manufacturers. Anisotropy of the n-values is obvious only from fields above 200 mT.

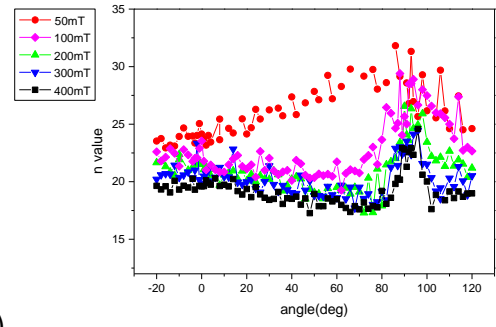
In the next step, characterization of 12mm wide tapes from SuperPower(2012) and AMSC was performed (figures 4,5). According to the manufacturers [24, 25], the 12mm tape from SuperPower should have identical structure as the 4 mm tape. Therefore, approximately three times higher critical currents and similar n-values would be expected compared to the 4mm tape. The AMSC 12 mm tape contains two HTS layers. The additional layer causes that critical currents should be about 6 times higher than in the case of the 344, 4mm wide tape. It is relatively difficult to predict the n-values of

this tape according to the results of the 344 AMSC 4 mm wide tape. In the case of the SuperPower(2012) tape, n-values are slightly higher than for the 4mm tape. This difference seems to be insignificant and most likely it is just an effect of different voltage criterion. Wider tape creates more voltage for the same electric field E and therefore critical electric field E_c , for the wider 12mm tape is smaller than E_c of 4mm tape at the same voltage criterion. For this reason, n-values were calculated for different voltage criteria [32]. The original $1\mu\text{V}/\text{cm}$ criterion, but also higher $3\mu\text{V}/\text{cm}$ and $6\mu\text{V}/\text{cm}$ voltage criteria were used. Although, n-values calculated with higher criterion are slightly lower, no significant difference can be seen between figures 4b and 4c. Even though n-values calculated by this enlarged voltage criterion are very similar to the 4 mm SuperPower(2012) tape, the used enlargement of the voltage criterion is not completely justified due to for example the edge effect of the tapes.

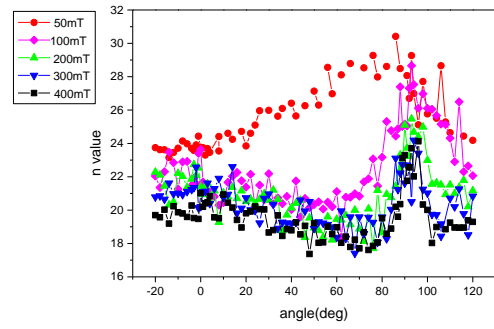
A clear *ab* peak shift can be observed in figure 4. This phenomenon was observed in other references [5,33]. It is interesting to note that the shift of the *ab* peak is obvious also in the case of n-values as well (figure 4b). Change of the criterion seems to be without too much effect by the 12 mm wide AMSC tape with two YBCO layers. This tape has shown very high n-values for both voltage criteria. The measured n-values are significantly higher especially in low fields if compared to the 344 AMSC 4 mm wide tape. (figure 5) . Most likely, other more complex mechanisms are involved in this phenomenon where identifying of these mechanisms would be a pure speculation at this stage.



a)



b)



c)

Figure 4: 12mm wide SuperPower(2012) tape a) critical currents b) n-values with criterion $1\mu\text{V}/\text{cm}$ c) n-values with criterion $3\mu\text{V}/\text{cm}$

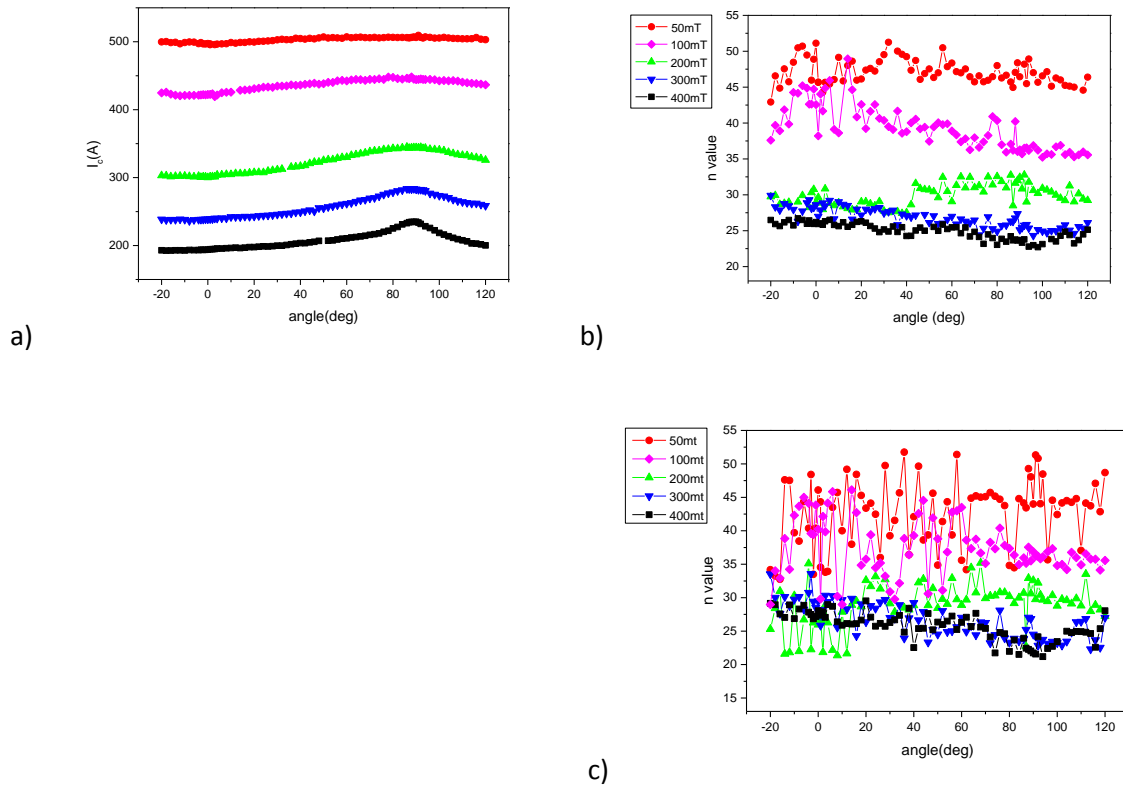


Figure 5: 12 mm wide double HTS layer Amperium tape a) critical currents b) n-values with criterion $1\mu\text{V}/\text{cm}$ c) n-values with criterion $6\mu\text{V}/\text{cm}$

Tapes in high magnetic fields and after irradiation

In this part, results in higher magnetic fields ($B \geq 1\text{ T}$) are presented. The tapes were irradiated several times by fast neutron fluences and re-measured. The presented graphs (figure 6) consist of results after each irradiation step. The type of artificial defects induced by neutron irradiation strongly depends on the kinetic energy of neutrons. Fast neutrons are neutrons with higher energies ($E \geq 0.1\text{ MeV}$) and they produce spherical defects of amorphous material with a diameter of a few nm (so called collision cascades)[15,17-19]. The point defects and clusters of point defects are created by neutrons with lower energies and they can act as effective pinning as well [34]. The Triga Mark reactor II in Vienna (CIF) was used as an irradiation facility in this work. The irradiation levels of fast neutrons applied to the samples were up to a fluences of $1 \times 10^{22}\text{ m}^{-2}$. The other irradiation levels were: $2 \times 10^{21}\text{ m}^{-2}$ and $4 \times 10^2\text{ m}^{-2}$. Since the irradiation procedure is a very time consuming process, only the 4 mm wide SuperPower(2008) tapes were characterized. The main time delays are caused by the fact that the samples are becoming radioactive emitters after the neutron irradiation procedure. In order to

perform the measurements without any health and safety hazards, it is necessary to wait until the radioactivity of the sample is decayed to the acceptable limits.

The measurements were performed in the 6 T and 17 T measurement set-ups at various temperatures from 50 K up to 85 K. Similar studies focused on only critical current enhancement/reduction of 2 G HTS tapes after irradiation are already available [20-22]. In figure 6 are shown the n-value results together with corresponding critical currents.

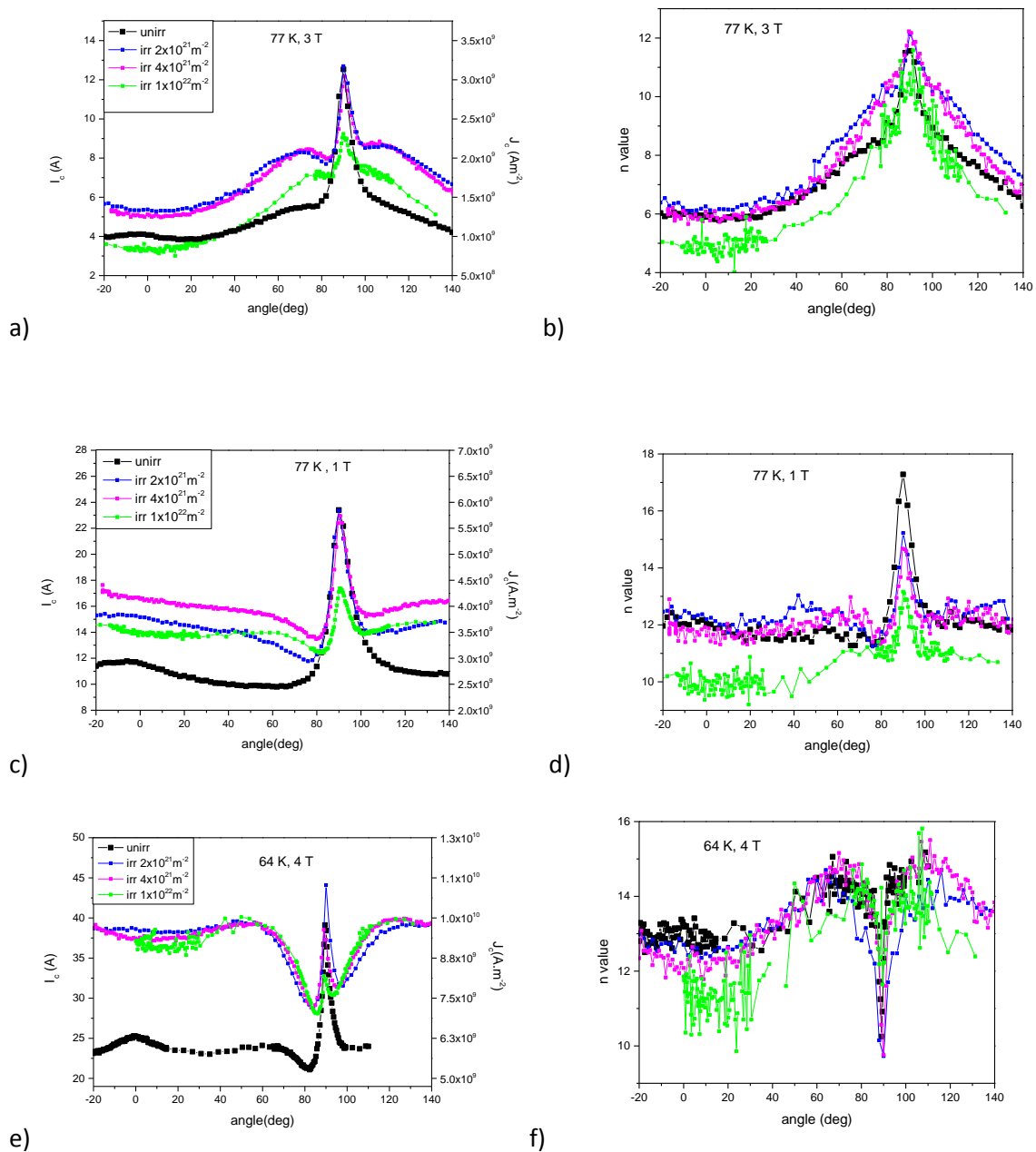


Figure 6: SuperPower(2008) tape characterized after fast neutron irradiation in the 6 T measurement set-up: a) critical currents 77 K, 3 T b) n-values 77 K, 1T c) critical currents 77 K, 1T d)n-values 77 K, 1T e) critical currents 64 K, 4 T f) n- values 64 K, 4 T.

A significant reduction of n -values over the whole scale of measured angles, fields and temperatures is observed at the highest fast neutron fluence ($1 \times 10^{22} \text{m}^{-2}$). At the lower irradiation levels, there are some regions with increase and also with reduction of the n -values. At 64 K, the position of the J_c peak corresponds to the deep drop of the n -value curves which is in literature known as inverse correlation to the J_c (figure 6f) [3-5]. Despite of this drop, the anisotropy of n -values at 64 K is rather low. The inverse correlation of the n -values by the J_c peak has been observed also at 50 K. The inverse n -value and J_c correlation is commonly related to the staircase flux lines in the superconductor [3,4]. Staircase flux lines are usually created in periodic pinning structures of high densities. Intrinsic pinning could be considered as this pinning structure at certain temperatures. Intrinsic pinning is characteristic by the small activation energy of the pinned flux; however, the high density of these pinning centres makes them very efficient at low temperatures when thermal depinning becomes less important. The change of the n -value curve between 77 K and 64 K indicates some transition in the role of intrinsic pinning in this range. This transition could cause change of parameters in relation (7), or even transition to a different relation. It was noticed in [31] that the relation between J_c and n -value can be even linear at certain low current conditions. Nevertheless, it is important to note that all the presented measurements in this section are performed at high fields where critical currents are controlled by grains only and not the grain boundaries as shown in [22,29]. This excludes models explaining n -values assuming grain boundary controlled currents and nonuniformity from [13,14]. Even the explanation by the staircase flux lines theory is limited to the low fields, as the flux lines they remain mostly straight due to their high numbers in fully penetrated superconductor in higher fields.

After the fast neutron irradiation, randomly distributed efficient pinning centres are introduced into superconducting grains. They are very efficient at wide range orientations of external magnetic fields except orientations where very high density of efficient pinning is present e.g. close to the ab peak. The fast neutron introduced pinning centres can have higher activation energies than original pinning and can enhance n -values. This enhancement is observed in figure 6, especially at 77 K (figures 6b,d). A drop of n -values is observed at the fluence of $1 \times 10^{22} \text{m}^{-2}$. The drop of n -values looks like a

consequence of very high density pinning centres. All the situations with very high density of efficient pinning centres (high neutron fluences, intrinsic pinning) have always shown smaller n -values. There is probably a threshold value of efficient pinning centres concentration after which the n -value starts to be reduced with increasing concentration. This threshold value is obviously strongly dependant on field and temperature. High irradiation fluences cause damage of the crystal structure which can be its reason of loss or partial loss of ability of flux pinning. The consequences are reduced J_c values close to the ab peak in all the presented measurements (figures 6a,c,e) ,especially at the highest fluence. The other consequence is probably partial n -value recovery at the ab peak at 64 K, 4T, at the highest neutron fluence, where the flux pinning abilities of the intrinsic pinning centres could be significantly reduced.

None of the mentioned effects is a consequence of nonuniformity in the HTS, which is experimentally proven in the following section of the paragraph. It is well known that neutron irradiation introduces disorder into the YBCO crystals. The commonly observed consequence of this disorder, which is mostly a consequence of already mentioned point defects in the oxygen sublattice, is critical temperature reduction of irradiated samples [15,33]. However if fast neutron irradiation creates nonuniformity in HTS, particular YBCO grains would have different T_c causing wider transition to the normal state. Therefore, fine T_c measurements after each irradiation step were performed. The result (figure 7) shows no influence of fast neutron irradiation on the broadening the transition. It means that the point defects caused by neutron irradiation are homogenously distributed and no additional nonuniformity is created. The homogenous distribution is possible due to their mobility and also relatively high neutron fluences.

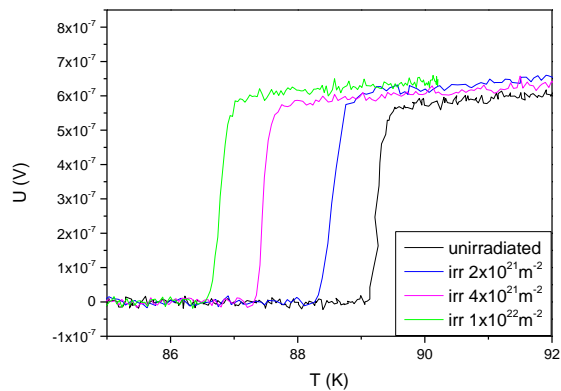


Figure 7: T_c transition transport measurements after each irradiation step performed in 17 T measurement set-up.

The SuperPower(2008) tape was characterized also in high fields up to 15 T (17 T measurement set-up) in two main magnetic field directions. This measurement should confirm results from previous angle-resolved measurements with additional information of n-values in very high fields. Results of these measurements together with measurements after irradiation by fast neutron fluences of $4 \times 10^{21} \text{ m}^{-2}$ are shown in figure 8. It is worth to note that at lower temperatures such as 64 K and 50 K, at magnetic fields parallel to the ab planes ($H \parallel ab$ - represents 90° in figure 6), n-values are not reduced with increasing magnetic field (after the initial drop at low fields).

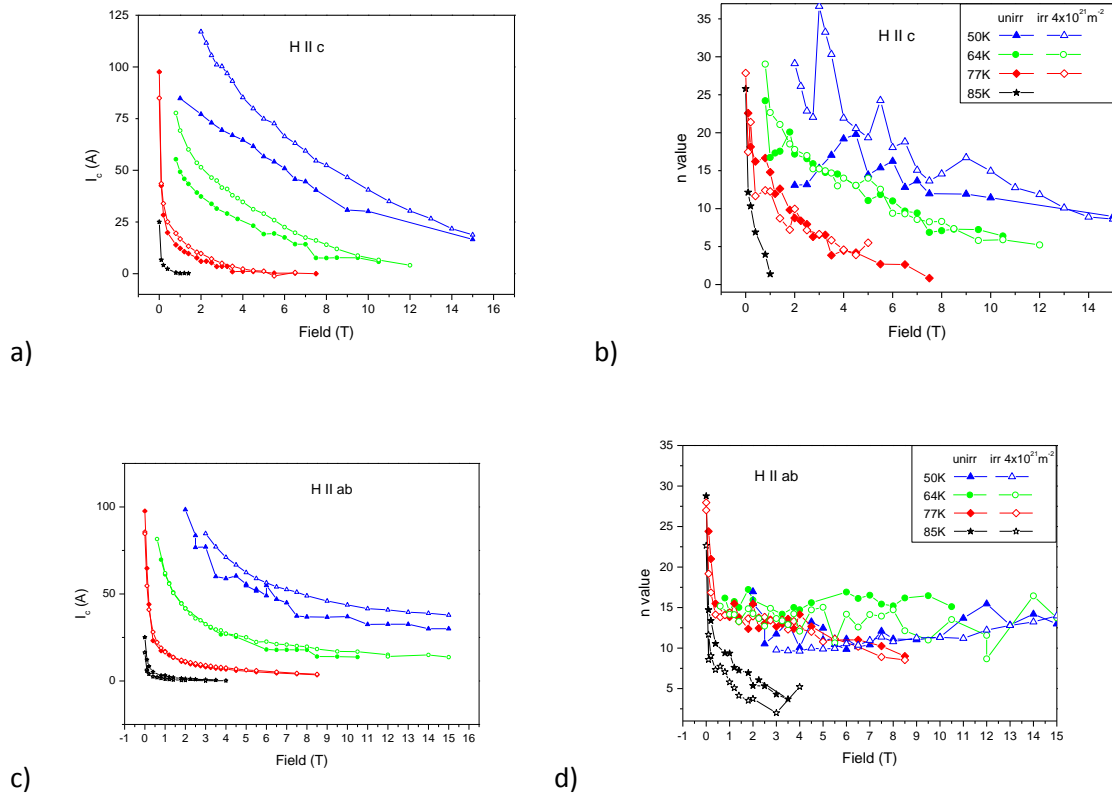


Figure 8: critical currents and n-values at different external magnetic fields, temperatures and irradiation levels: a),b) HIIab, c),d) HIIc.

V Summary and conclusions

n-values of different 4mm and 12 mm wide commercial 2 G HTS tapes at various magnetic fields and temperatures were presented. The performance of the HTS tapes characterized in higher fields is still relatively low. In addition reduction of n-values would enforce them to be operated even at lower currents, which makes them not suitable for most of the possible applications where high fields are required.

Higher n-values and relatively low anisotropy of n-values (compared to J_c) was observed in recent 2G HTS tapes. n-values were studied also after fast neutron irradiation, which introduced randomly distributed spherical defects into the superconductor. Two main factors, nonuniformity and flux creep, are considered as a source the exponential relationship of the I-V curve. Nonuniformity was excluded as the primary factor at higher temperatures and higher fields, where the currents are limited by grains

and not by the grain boundaries. In addition, it was shown that all the n -value changes after fast neutron irradiation cannot be consequence of nonuniformity as irradiation defects were distributed homogenously. Another studied phenomenon was correlation between n -values and J_c . It was shown that n -value is proportional to pinning energy which usually results into correlation with J_c . However, inverse correlation between n -values and J_c was found under circumstances, where high densities of efficient pinning centres are present.

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