FABRICATION AND APPLICATION OF NbTi AND Nb_3Sn SUPERCONDUCTORS

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Abstract

Superconductors allow the lossless flow of electrical current. This results in a high potential for applications in electrical energy and magnet technology. Of the many existing superconductor materials only Nb based superconductors have been developed so far into mature products[1]. Technical superconductors used in magnet technology consist nearly exclusively of the alloy NbTi and the compound Nb_3Sn. Design, fabrication technology, properties and application of these conductors are described.

Introduction

Superconductivity was discovered in 1911 in mercury shortly after it became possible to liquefy helium. Today more than thousand superconducting materials are known. Nevertheless superconductors based on Nb are virtually the only superconductor materials used in practical applications of superconductivity. In principle superconductors can be used in many electronic or electric applications. But as they must be cooled down to cryogenic temperatures, actual applications are limited mostly by economical considerations related to the complexity and cost of the cryogenic environment needed. Nb itself in elemental form can not carry significant bulk currents and is therefore limited mainly to radio frequency applications[2] and electronic applications such as SQUIDS for very sensitive current and magnetic field sensors. In order to allow high currents the Nb must be magnetically hardened by alloying and by introduction of pinning centers for the magnetic flux. This is very successfully achieved in the system NbTi. Multifilamentary wires made from NbTi/Cu composites are presently and will, in future, also be the work horse in superconductor magnet technology. As the usability of NbTi is limited to magnetic fields of about 10T, another Nb based superconductor, Nb_3Sn, is applied at higher fields. Its superior superconducting properties are related to the specific crystal structure, called A15 phase. As the intermetallic phase Nb_3Sn is very brittle complicated manufacturing technologies have to be used. Nb_3Sn type conductors are therefore nearly exclusively applied in magnetic fields above 10T up to more than 20T. Obvious potential applications of superconducting wires are in the electric power technology and in high magnetic field systems. Due to the need to cool the metallic superconductors including the Nb based ones down to the temperature of liquid helium (-269 °C or 4K) or at least close to it, power applications were not successful. This will probably change with the advent of wires made from ceramic so called High Temperature Superconductors. They allow cooling with liquid nitrogen. The application field of Nb based superconducting wires is, and will remain, the generation of high magnetic fields for different established technologies such as Magnetic Resonance Imaging (MRI) in medicine, Nuclear Magnetic Resonance Spectroscopy (NMR) in biology and chemistry, magnets for Nuclear Fusion power technology and magnets for particle accelerators in basic High Energy Physics.

Properties of Nb Based Superconductor Materials

Nb exhibits the highest critical temperature of an element and becomes superconducting at 9.2K. But it cannot carry bulk current and cannot sustain high magnetic fields. Alloying with Ti does not degrade T_c but does magnetically harden the material such that it can carry large bulk currents and can withstand high magnetic fields. Today the commercial standard alloy is NbTi$^{w}/_{0}$47 exhibiting a Tc of about 9.6K and an upper critical field, B_{c2}, of about 11T at 4.2K and 14T at 2K, respectively. In addition the phase diagram of NbTi is such that by a suitable thermo-mechanical treatment pinning centers for the magnetic flux can be introduced by α-Ti precipitations optimizing the current carrying capacity.

Nb based intermetallic compounds with A15 structure such as Nb_3Sn and Nb_3Al exhibit even better superconducting properties compared to NbTi. The only commercial compound Nb_3Sn has a $T_c \approx 18K$ and a B_{c2} up to more than 25T at 4.2K depending on the exact chemistry and microstructure. Doping with Ta and/or Ti significantly increases the high field properties of the Nb_3Sn phase. Pinning centers in Nb_3Sn are the grain boundaries requiring therefore a fine grained microstructure for high current densities.

In Figure 1 the superconducting properties are summarized for commercially available NbTi and Nb_3Sn based superconductor wires. It shows for two temperatures (4.2K and 2K) the dependence of the critical current density (i.e. the maximum possible current density) in NbTi, binary Nb_3Sn, ternary $(Nb, Ta)_3Sn$ and quaternary $(Nb, Ta, Ti)_3Sn$. As the lowest technologically interesting jc values is a few $10^4 A/cm^2$ the graph indicates the applicability limit of the different materials as a function of magnetic field.

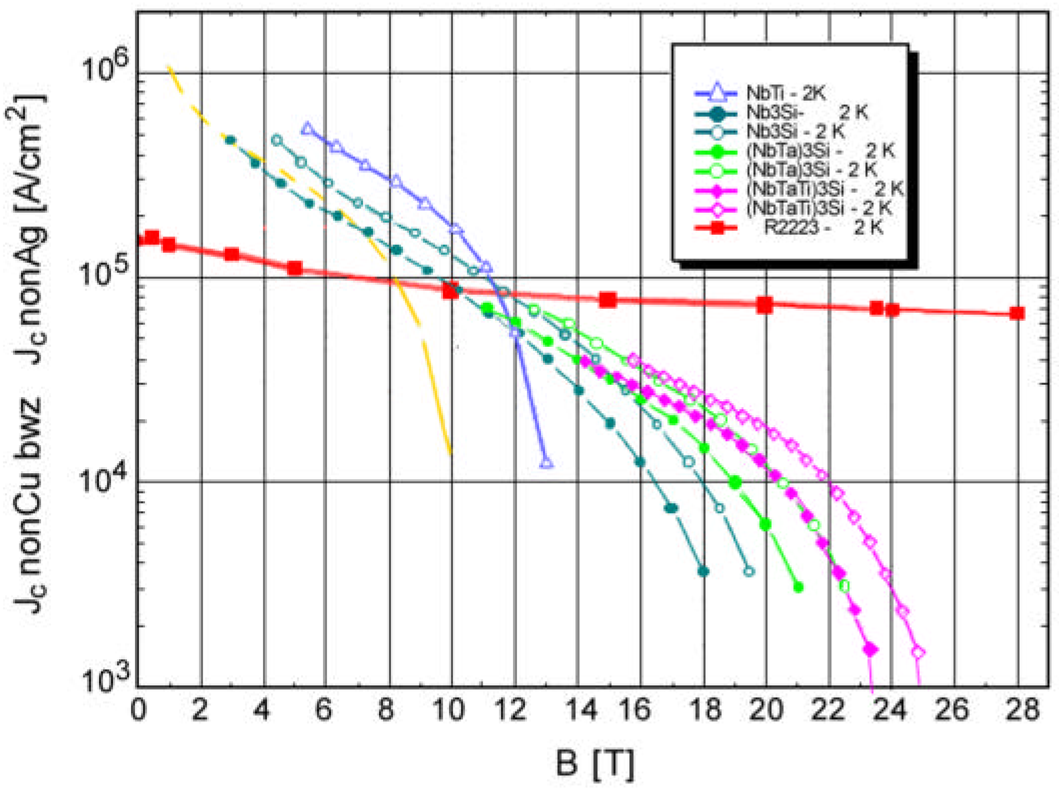

Figure 1: Critical current density as a function of magnetic field for NbTi and Nb_3Sn superconductors. The different curves for 4.2K and 2K respectively indicate the enhancement when lowering the operational temperature. For Nb_3Sn an improvement can be achieved by alloying with Ta or/and with Ti. Also shown is the jc curve for the ceramic HTS material $Bi_2Sr_2Ca_2Cu_3O_x$, indicating its superiority at very high magnetic fields.

Conductor Design and Fabrication Technology

Technical superconductors are composite conductors with the superconductor subdivided into so called filaments imbedded in a normal conducting matrix. This is required in order to achieve electromagnetic and thermal stability. Each superconductor exposed to an external field tends to shield itself from this field. This leads to superconducting (i.e. persistent) shielding currents and related magnetic fields and field energy. Any flux change (spontaneous or induced) leads to dissipation and heating of the superconductor. As the heat capacity at low temperatures is very small, tiny amounts of energy lead to large temperature excursions and can end up in a complete destruction of superconductivity i.e. a quench. The strategy to avoid such instabilities is to reduce the available magnetic energy by going to filament diameters of about 100 µm or less. The usual process to produce such a composite superconductor wire is to stack together the individual components at larger dimensions and to work down the composite by hot and/or cold working while maintaining a regular geometry of the filamentary array and continuity of the filaments. At least one hot-working step is desirable in order to achieve a good metallurgical bond between the individual components of the composite to allow its homogeneous deformation. This hot-working step usually consists of an extrusion process.

In addition to bonding, extrusion allows easy assembly of the components in the form of billets so that large quantities of conductors can be processed. Typical dimensions of extrusion billets are 150 to 250 mm diameter and 600 to 800 mm active length, resulting in typical units of 100 kg to 250 kg. This corresponds to lengths of about 5 to more than 100 km of wire, depending on wire diameter.

Not all combinations of matrix and filament materials are suitable for the fabrication of technical superconductors. For example, pure Al is too soft in comparison with Nb or NbTi and a reliable working process is not possible. On the other hand Cu and Nb or NbTi are relatively well matched and co-working processes are possible. In fact Cu/NbTi composites so far represent the most successful technical superconductors. Nevertheless conductor design and fabrication processes have to be appropriately adapted to result in high-quality conductors.

For the production of Cu/NbTi composites several methods of billet assembly may be used. For low filament numbers (up to a few hundred) NbTi rods and Cu tubes with an outer hexagonal geometry are assembled in an outer billet tube. Appropriate pieces of Cu are used to fill the voids between the filamentary array and the circular billet tube (Fig. 2a). For larger numbers hexagonal-shaped monocore conductors are bundled inside the billet tube. Above several thousand filaments the hexagons become very small and their handling and regular bundling becomes difficult and expensive. An alternate approach for large filament numbers is therefore a two-stage bundling process whereby hexagonal stacking is applied in both stages (Figure 2).

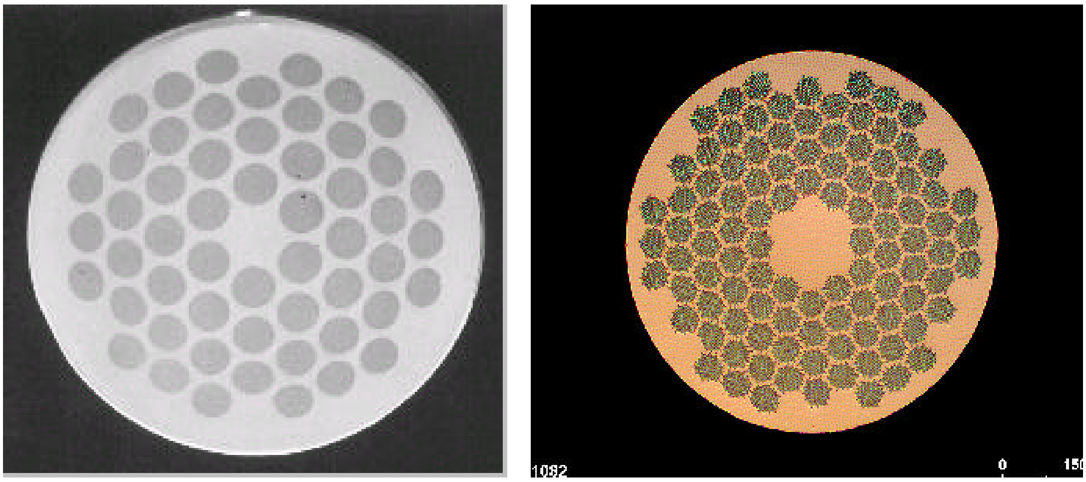

Figure 2: Examples of NbTi/Cu multifilamentary conductors. Left: Standard conductor with 54 filaments. Right: Fine filament conductor with 8670 filaments produced by a two stage bundling process.

At final dimension the wire must exhibit high critical current density. The most active pinning centres in NbTi are elongated normal-conducting α-Ti precipitates. Accordingly the wire fabrication process includes intermittent heat treatments between the drawing steps at typical temperatures of 380-400 °C with a total heat treatment time of typically 100 h. In order to achieve the critical current densities given in Fig. 1 large area reductions by cold working are required, resulting in the optimum microstructure needed for high critical current densities. As a consequence, for NbTi the large billet sizes mentioned above are required not only to achieve long lengths but also to result in high J_c.

Hot extrusion and also the heat treatments for J_c optimization can also have an adverse effect on J_c values through the formation of hard Cu-Ti intermetallic at the filament surface Figure 3. Especially at filament diameters smaller than 10 µm the intermetallic particles formed lead to sausaging (variation of the area along their lengths) and related degradation of J_c. To reduce and almost avoid this effect, fine-filament NbTi conductors are produced by applying a Nb diffusion barrier around each filament and thus using mono-core NbTi/Nb/Cu elements for the stacking process. The Nb thickness is designed to be thick enough to prevent intermetallic formation down to the diameter of the last optimization heat treatment of the multifilamentary wire. Figure 3 shows the improvement of filament uniformity achieved with this technology.

Obviously Nb_3Sn conductors cannot be produced in the same way as NbTi conductors because of the brittleness of the A15 phase. Nevertheless several fabrication methods have been developed for the production of multifilamentary Nb_3Sn wires. They all rely on the excellent ductility of Nb and apply Sn sources in the other components of the composite wire for later diffusion/reaction heat treatment to form Nb_3Sn.

In the "internal-Sn" process Nb filaments are imbedded in a pure Cu matrix. Sn sources are distributed in the Cu matrix as localized reservoirs either in the form of pure Sn or Sn alloy containing small amounts of Cu or Mg in order to harden the tin and to optimize reaction heat treatment characteristics. The Nb_3Sn phase is formed at final wire diameter by a sequence of heat treatment steps. This process in principle allows high critical current densities if the Sn content in the composites is kept high. Disadvantages of the process are related to the softness and the low melting point of the Sn (alloy). Hot extrusion of composites containing Sn is not possible due to melting of the Sn. As a consequence, metallurgical bonding of the elements in the composite is not optimum. This, in addition to the softness of the Sn (alloy) in comparison with the other elements of the composites, increases the tendency to mechanical instabilities during wire drawing and subsequent wire breakages. Research is still going on to resolve these issues.

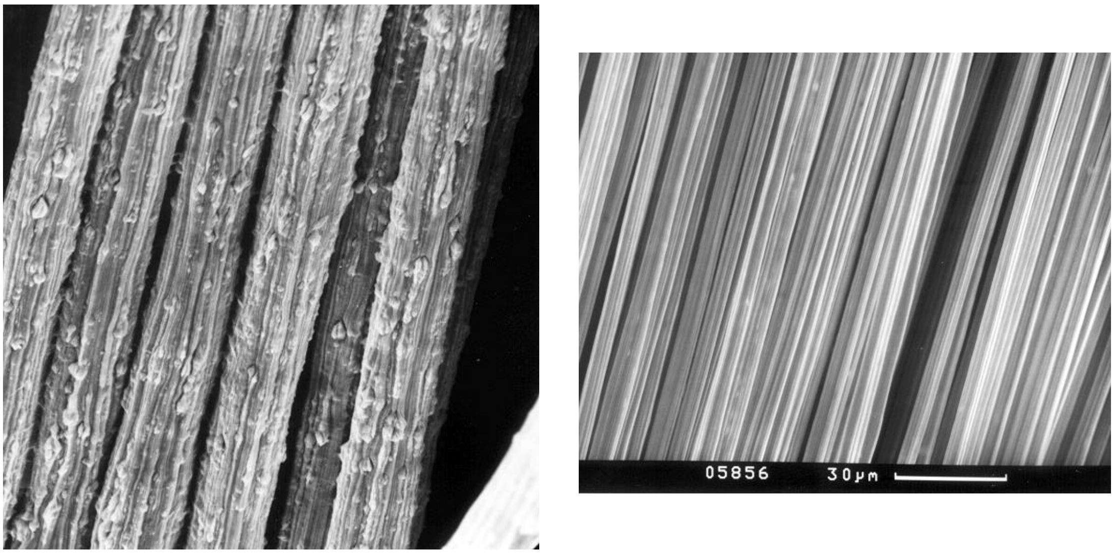

Figure 3: Quality of NbTi filaments. Left: Filaments without Nb barrier with hard intermetallic particles. Right: Improved filament quality by applying Nb barriers around each filament.

The discussed problems can be avoided in the "bronze process", which uses Nb filaments in a CuSn solid solution matrix. As a result of the solubility limit of Sn in Cu the content of Sn in the alloy is usually limited to about 13 to 15 $^w/_0$. In order to provide enough Sn a relatively high

CuSn matrix to Nb filament area ratio of about three is required. This implies that the overall critical current densities of bronze-process conductors tend to be smaller than those of internal Sn conductors. On the other hand the hardness of the CuSn alloy allows an excellent filament quality as required for persistent-mode magnets. Also, by redistributing the CuSn in such a way that the filament separation is sufficiently high, bridging of the filaments in the reacted stage can be avoided, allowing low hysteresis losses without sacrificing critical current density and workability of the composite. Figure 4 shows examples of reacted filamentary areas with and without bridging.

Usually for both the bronze process and the internal-Sn process the rod/filament design is used. Another alternative is to use the "jelly roll" process in which alternate Nb and Cu sheets are formed into a roll and act as a filament bundle. A modification of this approach is the "modified jelly roll" in, which an extended Nb mesh instead of a solid Nb sheet is used. The jelly roll approach can be used in the bronze process as well as in the internal-Sn process. Unfortunately, it seems to be difficult to scale the process up for large quantities and especially to large wire cross-sections.

In all described processes the matrix consists of, or is transformed into CuSn bronze. In a final heat treatment step Nb_3Sn is formed at about 650-700 °C for typically 50-200 h. As a consequence the composite would contain no stabilizing Cu. Any Cu must be added as a separate element and be protected from Sn poisoning by a diffusion barrier. Candidate barrier materials are Nb and Ta. Niobium is often used as a barrier, especially in internal-Sn-type conductors. But it has to be taken into account that an Nb_3Sn layer is formed at the interfaces to the filamentary area. This tends to increase the critical current density but leads also to larger magnetization and hysteresis losses and increases the susceptibility to flux jumping. Tantalum as a barrier material avoids all the above-mentioned complications and is therefore the preferred choice for many applications. Figure 5 shows examples of internal Sn and bronze route conductors.

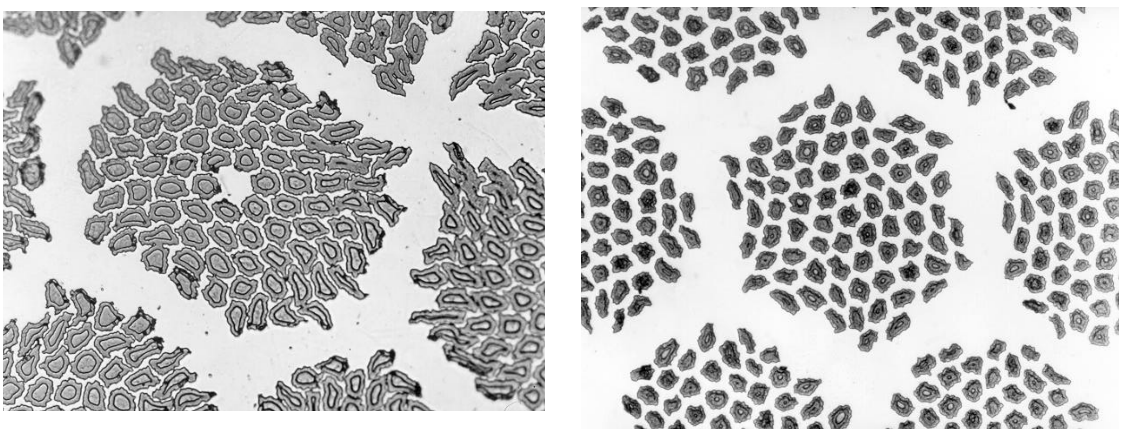

Figure 4: Cross sectional view of the filamentary area of bronze route Nb_3Sn conductors after reaction heat treatment. The reacted Nb_3Sn layer and the unreacted Nb core can clearly be distinguished (filament diameter about 4 µm). Left: Due to the growth of the filament area during reaction there is an intergrowth of the filaments within one bundle. Right: Intergrowth of filament avoided by large filament spacing.

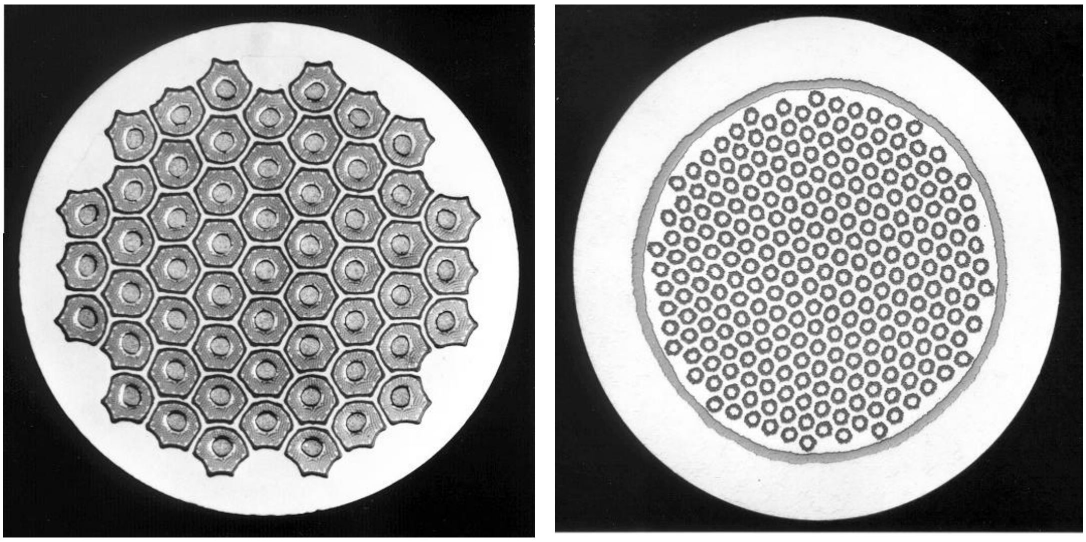

Figure 5: Examples of Nb_3Sn multifilamentary conductors. Left: Internal Sn conductor. Each bundle consists of a Sn source and a filamentary area surrounded by a diffusion barrier. The total array is imbedded in a Cu matrix. Right: Bronze route conductor, consisting of a number of filament bundles imbedded in a bronze matrix surrounded by a diffusion barrier and Cu stabilizer outside of the barrier.

An interesting approach having the potential of large jc values and avoiding the need for an extra barrier element can be found in the "Nb-tube" process. Here Nb tubes embedded in a Cu matrix are filled with Sn sources, either in the form of Sn (alloy) or of an NbSn compound powder (e.g. $NbSn_2$). During reaction heat treatment a Nb_3Sn layer is formed from the inside of the Nb tube and the outer part of the tube acts as a diffusion barrier. This approach leads to wires with very high critical current densities, but exhibits large filament diameters and is relatively difficult to scale up for large quantities and for industrial production.

Applications of Superconductors

Nb based conductors in the form of wires, cables and complicated built-up conductors including reinforcing members and cooling channels for the liquid helium have become an indispensable tool for high field magnet technology. The conductor design e.g. the complexity and the current carrying capacity strongly depend on the specific application such as magnetic field level, size and geometry.

The most prominent application is Magnetic Resonance Imaging (MRI) for medical diagnostics. Superconductivity has opened here a market of about 2.5 B$ for medical equipment. The second important application, Nuclear Magnetic Resonance (NMR) spectroscopy, is based on the same physical principle, the magnetic resonance of atomic nuclei, allowing e.g. the analysis if the chemical binding of an atom to its neighbors. As the spectroscopic resolution increases with magnetic field, NMR tends to need higher and higher fields. Therefore NMR magnets are the most prominent market for Nb_3Sn conductors. Other important applications of superconducting magnets are to be found in other analytic methods, Electron Paramagnetic Resonance (EPR), Ion Cyclotron Resonance (ICR) and Mass Spectroscopy (MS) or in other laboratory applications. Industrial process technology can largely be enhanced by superconducting magnets e.g. in the Si single crystal growth or in magnetic separation. Superconducting Magnetic Energy Storage (SMES) offers an innovative way to provide energy reserves in different time regimes and to improve the quality of electrical energy to the customers.

Another energy related application area is Nuclear Fusion. Electrical energy can not only be produced by the fission of heavy nuclei but also by fusion of hydrogen isotope to helium. The fusion reaction takes place only at very high temperatures such one has to deal with a plasma of charged particles. This plasma has to be contained by very large high magnetic fields which have to be produced by superconducting magnets.

Last but not least, superconducting magnets are needed in large scale basic research, especially in High Energy Particle Physics. In fact, this application has been since the early days, and still is, a technology driver for superconductor technology. Essentially two types of superconducting magnets are needed for this application: beam line magnets to bend and focus the accelerated particle beams and large detector magnets for detecting and analyzing the particles generated by the high energy collisions.

The most important applications of superconductors i.e. MRI, NMR, Nuclear Fusion and High Energy Physics are described in more detail in the following sections

Magnetic Resonance Imaging (MRI)

MRI is a powerful tool in medicine, biology and pharmacology. It allows the generation of thomographs of the human body and, of course, also of other objects such as of animals and of food. Other than x-ray based CT (Computer Tomography) MRI is virtually non-invasive as only magnetic fields and radio frequency fields are involved.

The size of the magnets needed depends of the size of the objects to be investigated. In terms of number of systems and volume of superconductors human whole body scanners are most prominent. In a typical system the magnetic field is produced by a set of solenoidal coils in a configuration that a very homogenous field is produced in the volume under consideration. The typical field level of a whole body scanner is between 0.5T and 1.5T. This field has to be very stable in time (<1 ppm/h). Therefore the magnet is operated in persistent mode. This means that after charging the magnet is short circuited and the power supply is disconnected. This implies that the superconductor wire has to be very homogenous along its whole length and that all joints between wires have to be superconducting.

At present functional MRI (fMRI) is more and more becoming a mature diagnostic tool. Functional imaging requires fast acquisition of images which in turn makes necessary higher fields. Typical feld levels are 3T to 4T, but there exist also systems with up to 9T. One typical example for fMRI is Functional Brain Imaging, detecting brain activity, due to the different magnetic properties of oxygen rich and oxygen deficient blood. It is interesting to note that presently there are activities to combine brain fMRI using NbTi based magnets in the multi-Tesla range with MEG (MagnetoEncephaloGraphy) measuring the small currents within the brain. The magnitude of the fields generated by these currents are of the order of fT (10^{-15} Tesla) and are measured through the use of SQUIDS (Superconducting Quantum Interference Devices) based on Nb superconductor technology. Thus the two technologies of SQUIDS and high field magnets based on Nb superconductors are meeting in Neuroimaging.

Figure 6 shows an outside view of a whole body scanner together with an image of human head. The typical wire geometry of superconductors used is also shown. The design is relatively simple with a few tens of filaments and a relatively large fraction of Cu-stabilizer which is needed due to the relatively large size and stored energy of the magnet. Besides the required homogeneity of the wires the design is dominated by cost considerations. At a very high Cu fraction the "wire-in-channel" design, with a pre-produced wire with low Cu-fraction is soldered in channeled Cu-profile, becomes very competitive.

Figure 6: Magnetic Resonance Imaging (MRI). Left: Typical superconductors for MRI (Monolith with 1 to 2 mm typical diameter and Wire-in-Channel WIC) with high Cu content Middle: Outside view of a whole body scanner. Right: Image of a human head.

Nuclear Magnetic Resonance Spectroscopy (NMR)

NMR spectroscopy has become a powerful analytical tool e.g. in chemistry, biology, medicine and materials research. As in MRI the magnetic resonance of the atomic nuclei is investigated. In NMR spectroscopy the spectra of resonance lines of the specific isotope under investigation are recorded and analysed to get structural information on the molecules in the sample. NMR spectrometers are ubiquitous in chemical, biotech and biomedical companies and at universities and research centers. They are used in standard applications (e.g. quality control) as well as in modern research.

As in MRI the isotope investigated is the hydrogen nucleus (proton). The resonance frequency depends linearly from the magnetic field. Usually a spectrometer is characterized by the basic proton resonance frequency rather than its magnetic field. The proportionality factor is such that 100 MHz corresponds to 2.35T. With increasing magnetic field not only the frequency increases but also the separation of lines coming from nuclei with different chemical environment increases. This means that the resolution increases with magnetic field. This fact explains the trend to go to higher and higher magnetic fields. The range of commercial NMR spectrometers presently is between 300 MHz (7T) for standard industrial applications and 900 MHz (18.8T) for high resolution spectroscopy e.g. in human genomics or proteomics. Up to 400 MHz (9.4T) the magnets use only NbTi conductors. Magnets for 500 MHz (11.8T) and higher are built with Nb_3Sn conductors in the inner high field section, and are therefore NbTi/Nb_3Sn hybrids (see Figure 1).

Figure 7 shows typical conductors of NMR spectrometer magnets and the cryostat of a spectrometer with a vertical warm bore for inserting the samples.

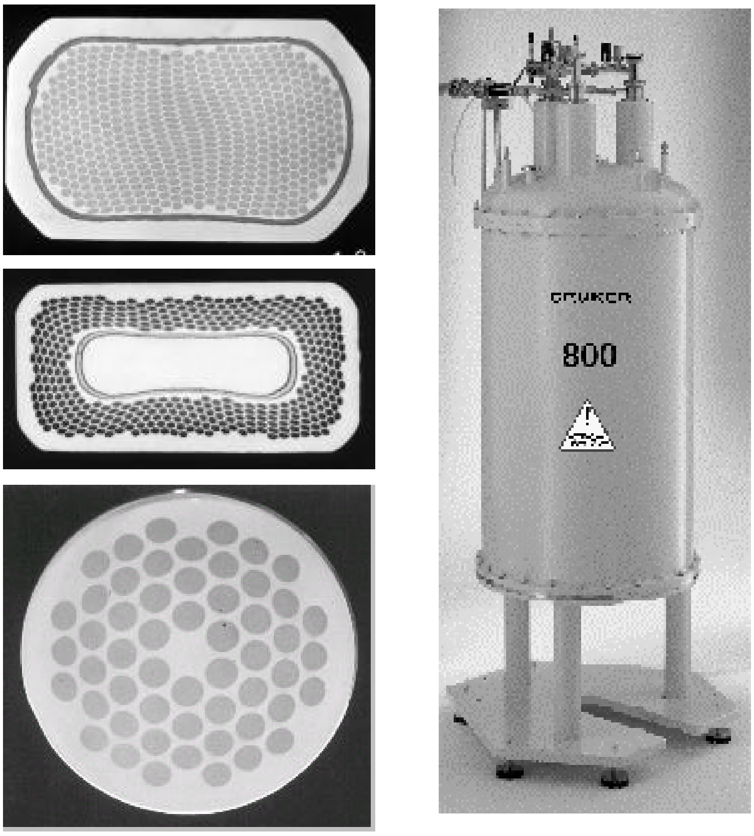

Figure 7: Nuclear Magnetic Resonance. Left: Typical superconductors for NMR magnets (From bottom to top NbTi standard conductor, Nb_3Sn with internal Cu stabilization and with external Cu stabilization). Right: NMR Magnet cryostat.

<u>Nuclear Fusion Technology</u>

Nuclear Fusion provides an option for future generation of electrical energy. The primary energy source comes from the reaction of the two hydrogen isotopes Deuterium + Tritium to form Helium + Neutron. The Neutron carries the main part of the energy which is then transformed into electrical energy. The fusion reaction takes place at very high temperatures only such that the matter is in the plasma state consisting of charged particles.

As a consequence the plasma cannot be contained in a vessel but it can be confined by a magnetic field. In the past decades nuclear fusion research was pushed forward by large scale fusion experiments applying larger and larger magnet systems. Several existing experiments are employing superconducting magnets as will the next generation of experiments and also a future economic fusion reactor.

To achieve good confinement the geometry of a fusion device is toroidal such that the magnetic field lines are closed. Two geometries for generating the appropriate field geometry are investigated: the TOKAMAK and the STELLARATOR. The TOKAMAK is a pulsed machine and has very high magnetic fields of typically 12T to 13T, requiring the application of Nb_3Sn. The STELLARATOR is a steady state machine and needs lower fields about 6T to such that NbTi can be used 8T but has a more complicated geometry of the magnet system.

Presently two advanced superconducting devices are under planning and under construction, respectively the Tokamak ITER (International Tokamak Engineering Reactor) and the Stellarator W7X.

Due to the size of these devices the conductor currents must be in the range of 10kA up to several tens of kA. The conductors are therefore multistage cables consisting of many individual strands. The cables are enclosed in an outer sheath acting simultaneously as a conduit for the liquid helium and as a strengthening member against the large Lorentz forces. Figure 8 shows the NbTi strand and the Cable-in-Conduit Conductor (CiCC) for W7X. The outer conduit consists of a co-extruded hardenable Al alloy. Figure 9 shows the strand and the CiCC for ITER. The strand is a low ac loss type Nb_3Sn wire and the CiCC has a central cooling channel for removal of the residual ac losses. The conduit is made from a special high strength steel.

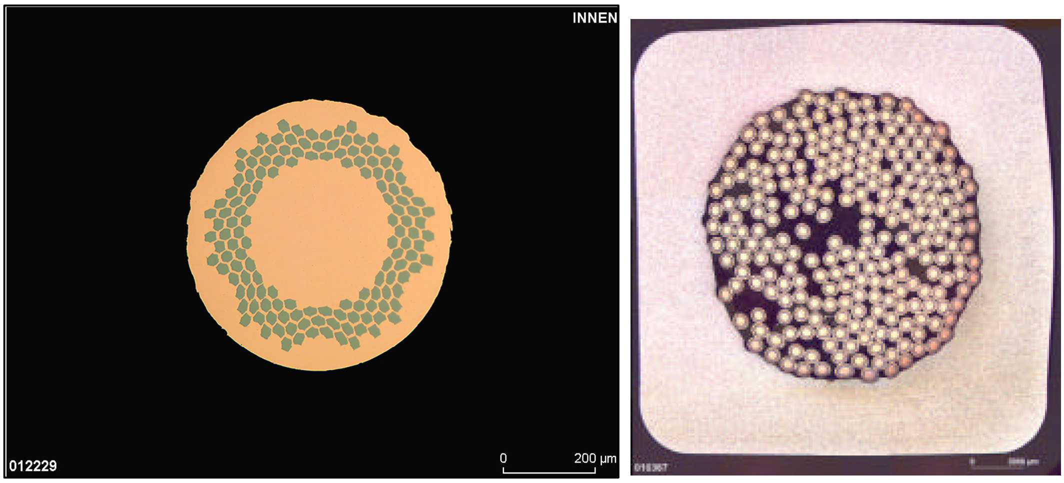

Figure 8: Conductor of W7X. Left: Basic NbTi strand (0.57 mm diameter)Right: CiCC with Al-alloy conduit (16 x 16 mm²).

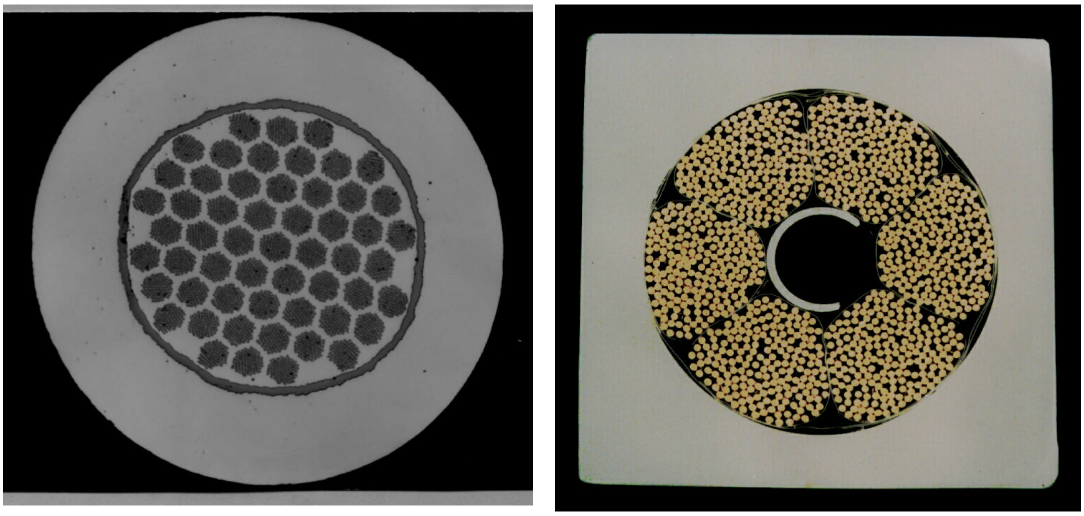

Figure 9: Conductor of ITER. Left: Basic Nb_3Sn strand (0.8 mm diameter). Right: CiCC with central cooling channel and special steel conduit (50 x 50 mm²).

<u>High Energy Particle Physics</u>

Accelerator technology and High Energy Physics and Nb based superconductors have practiced a tight symbiosis for nearly 40 years. Radio frequency cavities are presently used to provide efficient acceleration of the particle beams[2], whereas NbTi conductors are used in the beam line magnets for bending and focussing and in the detector magnets in the region where the particle collisions take place and the particles produced by these collisions have to be detected and analysed. These applications acted as a technology driver since the early days of Technical Superconductors. As an example, the technology of fine filament NbTi conductors with Nb diffusion barriers was developed in connection with accelerator magnets.

Several large superconducting accelerators were built in the past and are operated successfully. At present an even larger accelerator/collider, the Large Hadron Collider, LHC, is under construction in Geneva/Switzerland. In this machine two proton beams are accelerated in opposite direction in a circle of 20 km circumference. After acceleration the two beams are brought to collision. This collision region is equipped with particle detector systems including large superconducting magnets.

The system of beam line magnets consists of 1230 dipole magnets (15 m long) and 400 quadrupoles (5 m long) plus several thousands of smaller superconducting magnets. The cross section of the superconducting cable for the dipoles is shown in Figure 10. The flat cable is slightly key-stoned in order to better match the coil geometry. It consists of 26 strands of about 1 mm diameter. Each strand consists of 8670 filaments with 7 µm diameter. The cable carries a current of more than 15 kA at 9T and 1.9K. The total quantity of material needed is 470 tons of NbTi billets and 26 tons of Nb sheets.

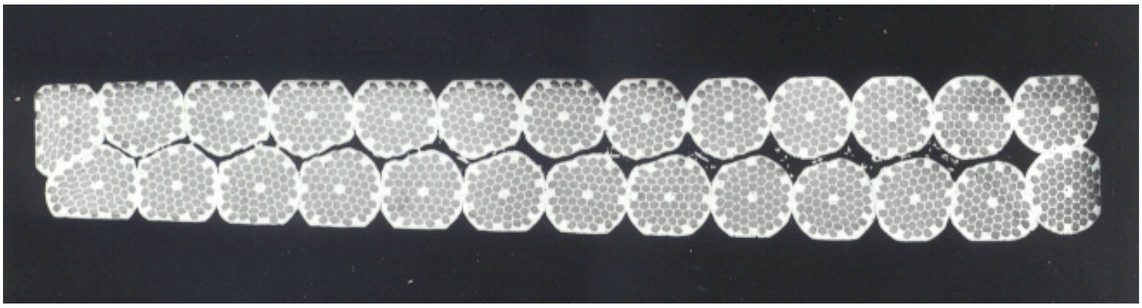

Figure 10: Superconducting cable for the LHC dipoles consisting of 26 strands with 1.06 mm diameter and 8670 filaments with 7µm diameter (Fig. 2, right).

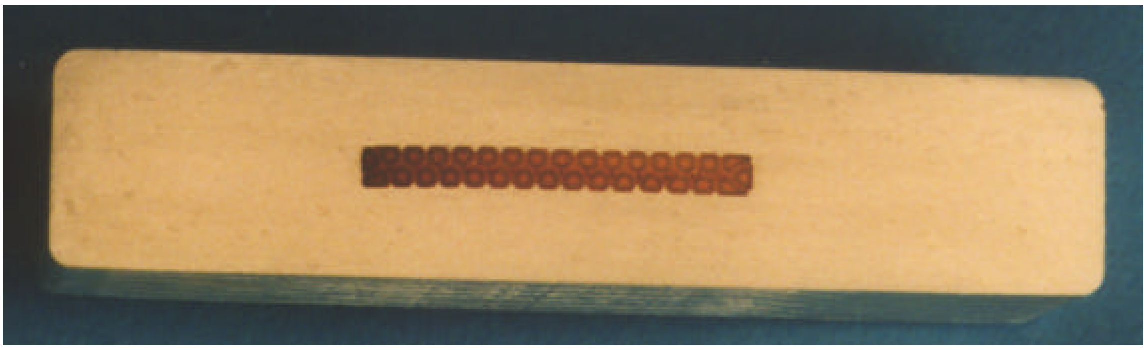

Figure 11: Superconducting cable for the ATLAS detector consisting of 38 strands with 1.3 mm diameter, co-extruded with ultra-pure Al, outer dimension 57 x 12 mm², critical current 60 kA at 5T and 4.2K.

In two locations of the ring will be collision regions with detectors (ATLAS, CMS) equipped with superconducting magnets. The conductor used for the toroidal magnet system of Atlas is shown in Figure 11. It consists of a flat cable with 38 NbTi/Cu strands. The cable is co-extruded with ultra-pure Al for electrical stabilization and quench protection. Al is chosen because its very low residual resistivity at operational temperature and field and because it is nearly transparent for the particles to be detected and crossing the magnet coil.

Conclusion

The Nb based superconductors NbTi and Nb_3Sn have become an indispensable tool to produce the high magnetic fields needed for different industrial and scientific applications. A high technological standard has been reached in the production of the pre-materials needed (NbTi as billets and rods, pure Nb or alloyed Nb as rods and sheets). The quantities needed per year are of the order of several 100 tons for NbTi and 10 tons for Nb, respectively. Concerning wires, cables and built-up conductors mature and partly sophisticated manufacturing technologies have been developed, such that the needs of the different applications can be satisfied.

References

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