

Microtechnology Focus

A New Paradigm for Improving the Superconducting Upper Critical Magnetic Field of Nanocrystalline Niobium Carbonitride (NbC $_{0.3}N_{0.7}$) for Fusion Energy and Healthcare

When an electrical current flows through a normal conductor, such as copper, it encounters a resistance that transforms the current's electrical energy into heat energy. It is therefore necessary to apply a permanent voltage to replenish the energy lost to the resistance and so maintain a steady current flow. There are at least three shortcomings with this situation; firstly, a constant supply of energy is required; secondly, energy is wasted in the form of heat; thirdly, the heating can itself be a problem. If, however, the electrical resistance can be removed from the conductor then these problems disappear.

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Below a certain critical temperature (T_c) a superconductor abruptly enters a superconducting state that is devoid of all electrical resistance. This is the primary reason why there has been an avid search for superconducting materials with ever-higher critical temperatures. The hope is that a room temperature superconductor will one day be found. This would remove the need for expensive cooling apparatus and the associated difficulties in maintaining the cooling over long distances. In the case of power transmission lines, for example, lossless transmission of electrical power would increase efficiency, reduce energy costs and reduce the burden on fossil fuels.

The search for high temperature superconductors is therefore an active one that has been augmented by interest from the popular press - though this is by no means the whole story. Superconductivity has immediate applications in electromagnet design. All moving charges produce associated magnetic fields. If a current is made to pass through a coiled wire, called a solenoid, a uniform magnetic field can be setup in its core. If the solenoid is made from superconducting wire and is cooled below its critical temperature, the circuit will remain resistance-free, the supply voltage can be removed, there will be no energy lost from the system and the current and field will remain constant. This lack of energy loss due to the resistance-less state ensures that the only energy cost is in setting up the super-current and in maintaining the required temperature.

SUPERCONDUCTING MAGNETS

Superconducting magnets are able to generate much higher magnetic fields than conventional electromagnets. In a conventional magnet system the electrical resistance in the coils causes the windings to run hot. This heat energy must be removed and controlled if damage is to be avoided. However, more importantly, since the magnitude of the electric current is a determining factor in the amount of heat that is produced, the heat problem limits the maximum current that can be carried by the windings. This in turn restricts the maximum magnetic field strength that can be produced. Superconducting magnets, however, do not create this heating and so their windings can be subjected to larger current densities that produce larger field strengths.

Whilst this lack of heat generation removes one particular limit on the amount of current that can be carried by a superconducting coil, the magnetic field produced by the current is unfortunately an additional limiting factor. Not only does a superconductor need to be kept under its transition temperature for it to remain superconducting but it must also be kept under a certain current density called the critical current density (J_c) . This is because superconductivity is also destroyed by large magnetic fields. All superconductors have an upper critical magnetic field (B_{c2}) above which they become normal conductors. When the critical current is reached t magnetic field at the surface of the superconductor reaches the upper critical magnetic field and the superconductor is driven into the normal state. This situation provides the motivation to find superconducting materials with ever-higher critical magnetic fields that can be used to produce more powerful superconducting magnets. Of course, other important limits cannot be ignored, such as the fact that increased magnetic fields produce increased mechanical forces, which can eventually lead to a magnet's destruction. Fortunately, increased magnetic field capabilities do not necessarily have to be used to produce magnets with increased field strengths; they can be used to produce smaller magnets for a given field strength at a reduced cost.

EXISTING MATERIALS

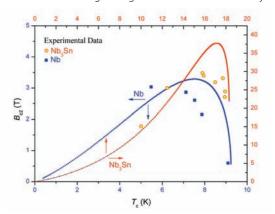
So, the search for new high temperature superconductors and high field superconductors goes on but there are also efforts being made in attempting to improve existing materials. Such is the case with the low temperature superconducting material niobium-carbonitride (NbC_{0.3}N_{0.7}). It has a maximum critical temperature in bulk form of ~ 17.8 K and an upper critical magnetic field of ~ 12 T. The most common superconducting materials used in magnet design are niobium-tin (Nb₃Sn, $T_c \sim$ 18 K and B_{c2} ~ 30 T) and niobium-titanium (NbTi, T_{c} ~ 9.5 K and $B_{c2} \sim 12$ T) [1]. The reason for this is not only due to their respectable superconducting characteristics but also because wires can be formed from them using a number of technologies including powder-in-tube technology, which enables brittle materials to be manipulated into wire form. So, what is the motivation to pursue niobium-cabonitride? Well, there is evidence to suggest that its upper critical magnetic field could be increased to ~ 42 T in powder form [2]. Furthermore, Dew-Hughes has shown that the B1 rock salt structure, of which niobium-carbonitride is composed, is resistant to radiation damage [3]. This is in contrast to the A15 compounds niobium-tin and niobium-titanium whose superconductivity is suppressed after being irradiated. This means that it might be advantageous to use niobium-carbonitride in radioactive environments such as tokomaks for nuclear fusion energy extraction. Furthermore, this material could be a viable alternative in applications such as levitating train technology, high-energy particle accelerators, energy storage devices and magnetic resonance imaging scanners (MRI) if sufficient improvements in its upper critical field can be made.

NANOCRYSTALLINE SUPERCONDUCTORS

Increasing the normal state resistivity ρ_n of a superconductor has been shown to significantly improve its upper critical magnetic field [4]. The relationship between the upper critical field and the normal state resistivity is given by,

$$B_{c2}(0) = \alpha \left[A \left(\frac{\gamma T_c}{S} \right) + B \left(\gamma T_c \rho_n \right) \right]$$

where α is a pre-factor that includes the Ginzburg-Landau parameter and a strong-coupling correction, A and B are constants, γ is the electronic specific heat coefficient and S is the Fermi surface area. Whilst this equation suggests that changes in resistivity directly affect the critical field, as indeed they do, changes in resistivity also affect γ and T_c ; both of which are suppressed by increases in ρ_n due to the broadening of the density of states at the Fermi energy, which complicates the behaviour of B_{c2} . However, theoretical work carried out in Durham University's Superconductivity Group has provided a means by which the variation in B_{c2} can be predicted using microscopic theory [5]. This can be seen in *Figure 1* for niobium and niobium-tin and can be used to predict the maximum critical field obtainable through changes in the normal state resistivity.



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Figure 1. Theoretical upper critical field as a function of critical temperature for increasing resistivity of niobium and niobiumtin. The experimental data for Nb and Nb₃Sn are included [2].

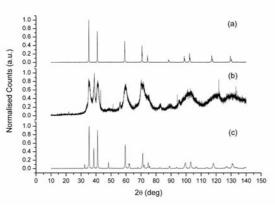


Figure 2. XRD data of niobium-carbonitride (a) sintered material (b) milled material (c) HIPed material. The broadened peaks in (b) signify a reduction in grain size from microcrystalline to the nanocrystalline scale length. The widths of the peaks in (c) have significantly reduced, indicating grain growth caused by hot isostatic pressing.

OPTIMISING THE NORMAL STATE RESISTIVITY

Increasing disorder in the structure of a material increases the normal state resistivity. This can be achieved by doping the material, which introduces impurities that increase electron scattering. However, it can also be achieved by decreasing long-range structural order. This latter method, pioneered in Durham University, employs a fairly well developed technology used for decreasing grain sizes of powdered materials, namely, mechanical milling.

MECHANICAL MILLING

Mechanical milling is a vast subject that entails finetuning a number of process variables to achieve decreased powder sizes, the formation of alloys or induced chemical reactions. The process requires a powder mix and a number of milling balls to be sealed within a milling pot. The choice of ball and pot material can be critical and in the case of niobium-carbonitride, niobium or tungsten-carbide milling media is used to reduce contamination. The pot is then spun in a planetary motion at 300rpm for varying time periods during which the powder is subjected to sustained bombardments from the balls and the sidewall of the pot. If the process variables have been chosen sufficiently carefully, the longrange order of the powder grains is completely destroyed and an amorphous form of the material is produced. Decreases in grain size can be detected using x-ray

powder diffraction (XRD). The XRD data, which produces peaks at angles for which Bragg-diffraction occurs from the planes that constitute the structure of the material, can be seen to be broadened in *Figure 2b. Figure 2a* is an example of niobium-carbonitride in bulk form; sharp peaks are prominently visible. In *Figure 2b* the XRD peak widths have been broadened in comparison. This indicates that milling has caused grain-size reduction, though in this case not necessarily to a complete amorphous state since the presence of some peaks is indicative of some remaining crystalline structures. Once the milling is complete it is necessary to further process the material using a hot isostatic press to bring back some short-range, nanocrystalline, order. In this way, the resistivity of the material can be fine-tuned.

HOT ISOSTATIC PRESSING

A hot isostatic press (HIP) subjects the milled material to a pressure of 2000 bar and temperatures that vary from 400°C to 1200°C. Different samples are processed in this way at different temperatures and then measured to determine which temperature produces the optimised material. During HIP'ing the large pressure densifies the sample and the elevated temperature promotes grain growth, transforming the sample from its milled powder form into a solidified bulk with a nanocrystalline structure. This increase in grain growth and the return of short-range order is visible in the XRD data shown in *Figure 2c*. On comparison of *Figure 2b* and 2c it can be seen that the XRD peak widths have decreased after HIP'ing, which is indicative of grain growth and the return to a crystalline structure.

MATERIAL PRODUCTION

Processing nanocrystalline niobium-carbonitride in the above ways is of course only part of the story. It is first necessary to fabricate the parent microcrystalline material itself. Furthermore, it is of paramount importance to be able to produce the very best material and insure that its high quality can be reproduced consistently and in adequate quantities. The superconductivity group in Durham University's physics department have fabricated niobium-carbonitride with a transition temperature of ~ 17.6 K by mixing niobium-nitride and niobium-carbide together in the required proportions, pressing the mix into pellets and then sintering them at 1650°C for 114 hours. They are now in the process of improving the procedure to achieve better homogeneity and the ability to fabricate increased quantities.

CONCLUSION

Niobium-carbonitride's transition temperature and resistance to radiation damage make it a viable contender for consideration in superconducting magnet designs as long as its upper critical magnetic field can be substantially improved. It is believed that the processes discussed here will lead to that improvement.

The material, in milled powder form, would then be loaded into tubes and drawn into long coiled wires, which would be HIP'ed to complete the material's optimisation.

The main prospective applications for such a conductor are in fusion reactors, where superconducting magnets are the enabling technology, and MRI scanners, where increased field strength leads to increased resolution.

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World's Fastest Desktop Scanning Electron Microscope

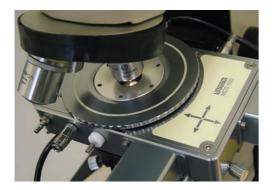
Phenom-World BV announced the launch of a new collection of sample holders and inserts for the Phenom[™] desktop scanning electron microscope (SEM). The new holders increase the range of possible samples while maintaining the Phenom's market-leading time to image. Quick and easy sample loading ensures faster time to data. Most industrial and research applications require imaging of non- or poorly-conducting samples. Imaging these samples with the charge reduction sample holder eliminates additional sample preparation and so reduces the critical time to data. Imaging samples such as paper, polymers, organic materials, ceramics, glass, and coatings is fast and trouble-free with this sample holder.

Imaging long, axial-shaped samples is a challenge in any SEM, but required in many industrial quality and failure applications. With the micro-tool sample holder, it is possible to make high-resolution images from samples such as drills, end-mills, routers, boring bars, engraving tools, needles, fibers, (fuel) injectors and pencils. Unique on the market, this holder enables top-down imaging of samples up to 100mm long. Samples are loaded into the holder without the need for tools or other preparation. The new micro-electronics insert enables non-destructive imaging of micro-electronics, solar cells and other wafer-based samples. The unique clamping mechanism makes glue or other adhesives obsolete, allowing the sample to be mounted quickly and then returned to the production process, or to be used in other quality and failure analysis machinery.

The X-view insert enables cross-sectional imaging of coatings, multi-layer semiconductors and fracture surfaces. Preparing the sample is fast and easy compared to costly and timeconsuming resin mounting. The X-view insert eliminates the need for screws and tools to clamp the sample. The introduction of the new holders is the second stage in series of marketfocused solutions following the successful launch of the FibermetricTM System in 2009.



Temperature Controlled Microscopy Systems used for Geological Applications



Heating and freezing stages are being used in thousands of laboratories worldwide. Applications may be found in just about all scientific disciplines from materials to foods, from chemistry to physics and biology. One area that continues to grow is the use in geology to study properties of the Earth. One example may be found at Kingston University where Professor of Applied Geology, Andrew Rankin, and his research group use heating/freezing stages in the temperature range from -196°C to +1500°C to investigate fluid inclusion in rocks.

Fluid inclusions are small droplets of fluid that have been trapped within crystals either during primary growth from solution or at some later stage, usually as a result of recrystallisation along healed microfractures. They are ubiquitous in both naturally occurring minerals and in laboratory-grown crystals. To the chemist or materials scientist, these gross defects cause endless obstacles in their quest to grow near perfect crystals. However, to the geologist, they provide a unique fossil record of the various fluids responsible for the formation and evolution of rocks and minerals throughout the history of the Earth. **Linkam systems** have enabled the routine study of geological fluids as well as many other samples. Accurate temperature control of a lab-based experiment is vital. Setting up the experiments is straightforward too. The optical microscope may be used to visually record sample changes as a function of temperature in conjunction with the positioning capability of the temperature stage.



