

A QUASISTATIC HIGH FIELD PULSE-MAGNETOMETER-THE AUSTROMAG HIGH FIELD SYSTEM – P 13146, started at 1.8.1998: A STATUS REPORT (June 2002)

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This give a status report of the existing Austromag system where the power supply was financed by the Ministry of Science, the experimental set-up was financed by this FWF project (Proj. Nr P 13146 PHY).

The power for generating high magnetic fields comes directly from the line. The primary power of 16 MVA is provided by a transformer from 10 kV to $2 \times 840\text{V}$; this can be switched either in series, parallel or antiparallel. The ac-current is rectified by two bridges with 6 thyristors that are regulated by varying the ignition angle α . The maximum dc-power is 10 MW over 1s, or 5 MW for 2s or 1 MW for 10s. With this thyristor regulator the current versus time profile can be freely chosen with 20 points to define the profile. In particular, field plateaus are possible which are important for measurements on metallic samples with minimized eddy current. With this system a quasistatic field of up to 40T is available. The antiparallel circuit allows also a bipolar pulse which is especially valuable for a full characterisation of magnetic materials. There are now three different systems available:

- Low temperature system: this consist of the 40T magnet from Metis, a flow cryostat from Cryogenics (1.5K up to 300K) with a sample space of 12mm. In this cryostat a pick-up system was installed in order to allow magnetization measurements.
- High temperature system: This consist of a 35T magnet with a 25 mm bore. In this bore a vacuum isolated furnace was developed; inside is a pick-up system which allows magnetization measurements from room temperature up to 500°C.
- Test system: this is a room temperature system with a self made magnet which generates fields up to 25T in a bore of 25mm. The purpose of this system is to test new measuring methods.
- For the new two coil system a self made 30 T magnet with a large bore of 58 mm was constructed. Inside of this magnet the two coil system can be tested but also new techniques such as modulation method can be tested.

All together experiments between 1.5K and 800K in high magnetic fields are possible now: The system can be used for measuring the hysteresis loop (magnetization) going over a broad temperature range (from 1.5 K up to 800 K). These measurements can be done in quasi-static fields up to 40T however also with varying field sweep rates dH/dt . This is especially interesting in order to investigate magnetic viscosity effects. Additional the magneto-resistance and the magnetostriction can be measured between 1.5 K and 300 K. We are studying two main topics:

- i) Hard magnetic materials - including time dependent effects.
- ii) 3d-4f compounds with critical fields. By measuring the critical field the exchange parameter can be determined in a direct manner.

First studies on the magnetic viscosity of $\text{SmCo}_{5-x}\text{Cu}_x$ were performed. As an example we measured the temperature dependence of the coercive field of a 2/17 based permanent magnet in comparison with data as obtained in our fast pulsed field magnetometer. The values as obtained with the Austromag quasi-static system measured with a linear dH/dt of 67T/s were

below those obtained in a short pulsed field magnetometer (sinusoidal pulse; pulse duration about 10ms; about 2000T/s); this is a consequence of the magnetic viscosity of this sample. From the different values of the coercivity measured under varying field sweep rates dH/dt the magnetic viscosity parameter could be estimated. Additionally a program was started measuring the magnetostriction coefficients on technical permanent magnets. First results were obtained at room temperature on a technical anisotropic barium ferrite (supplied by Schramberg A.G.). On the aligned material the different magnetostriction coefficients λ_{ij} could be determined – as there are: λ_{pc} : H perpendicular and DMS measurement parallel to the c-axis; λ_{cc} : H and DMS measurement direction parallel to the c-axis; λ_{pp} : H and DMS measurement direction perpendicular to the c-axis; λ_{cp} : H parallel and DMS direction perpendicular to the c-axis.

Abstract

The Austromag project offers high magnetic fields for various applications in solid state physics. First the existing quasistatic high field facility reaching 40T in a long pulse (1s) was installed with respect to various facilities measuring the magnetization, the magnetoresistance and the magnetostriction – this was part of the FWF project (Proj. Nr P 13146 PHY). Beside the increase of the available field new measurement methods with improved sensitivity (vibration method, surface coils) were developed and are still under investigation. One additional target here is to improve the achievable sensitivity.

1) The Austromag system

1.1 The technical data of the “Austromag” high field installation

a) Power supply

The energy necessary for the generation of high magnetic fields necessary energy is supplied by the line of the city. Fig.1 shows a block diagram of the power regulating system which was constructed by ABB. A second cable of the total power supply of the university can be used for this facility. The primary power is 16 MVA which delivers at a 10 kV level the transformer. There the voltage is reduced to 2 x 840V which can be switched together either in series (1680V, 10 kA), parallel (840V, 20 kA) or antiparallel ($\pm 840V$, ± 10 kA). The later pulse shape is important for measuring high field hysteresis of magnetic materials. The ac-current is rectified by two bridges with 6 thyristors which can be regulated by varying the ignition angle α . The maximum dc-power is 10 MW over 1s. With this thyristor regulator the current versus time profile can be free chosen - with 20 free points within the profile. In the system a PI regulator is realised. For each field versus time profile the parameters (altogether 40) have to be optimized. This cannot be done experimentally because the high number of high field experiments will destroy the magnet before a sensefull experiment is possible. Therefore a simulation algorithm of the total system was developed in order to calculate the parameters including the heating up of the magnet (non linear regulating load). Here especially field plateau's are a problem - which are important for eddy current free measurements on metallic samples. Fig. 2 shows the used diagram for simulating the parallel circuit:

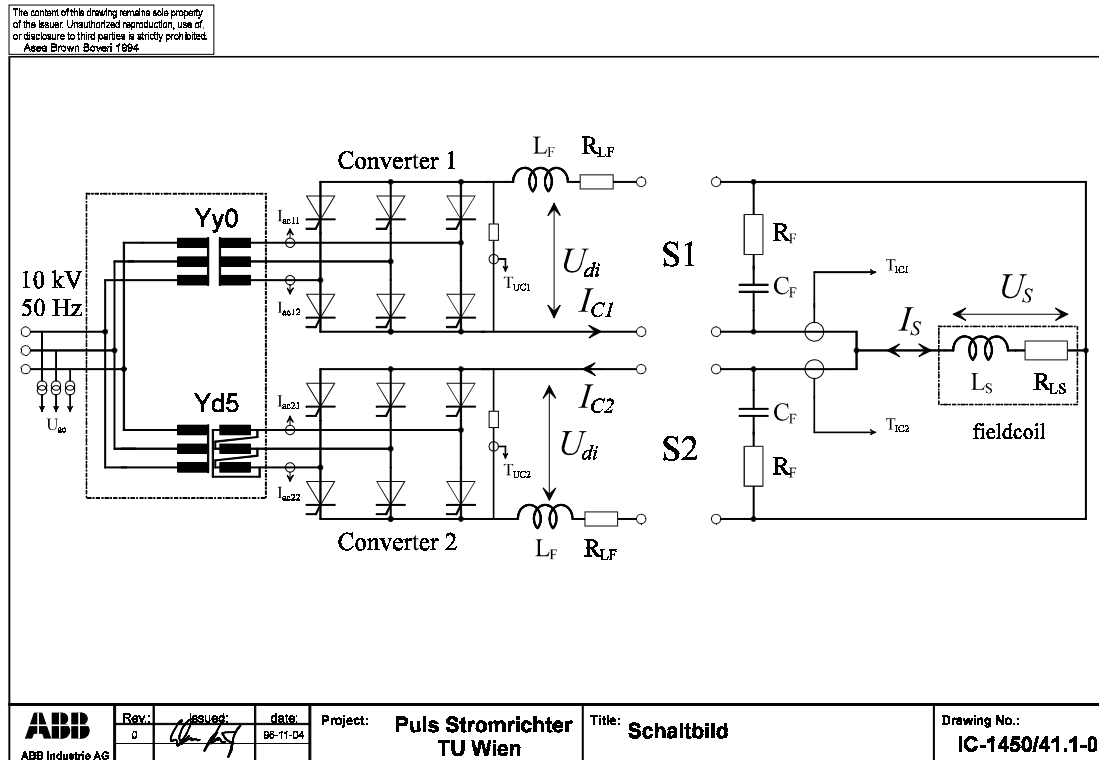


Fig.1: Block diagram of the 10 MW 1s thyristor regulated power supply from ABB.

Special emphasis is laid on the ripple of the current which limits finally the achievable experimental resolution. Also the ripple was calculated using our simulation and compared with experimental results.

In the case of the serial respectively parallel circuit the thyristors work as a 12-puls rectifier which has according to theoretical simulations an over all ripple on the dc-current of 10^{-4} , whereas in the case of the anti parallel circuit (6-pulse rectifier) the ripple increases to a value of about 10^{-3} . This ripple is improved by a factor five by using a passive filter (consisting of L_F , R_{LF} and C_F , R_F). The current coming from the two thyristor bridges is measured using a current transducer with an accuracy of 0.5%. Theoretical calculations show that a further improvement of the ripple is possible by increasing the current with a constant ignition angle $\alpha = 15^\circ$. Fig.2 (upper part) shows the apparent harmonic voltages for this case and in the lower part the distortion of the current (ITF current without filter, ITF_f distortion including the filter) is shown. It can be seen that in this case the distortions can be reduced to about $5 \cdot 10^{-5}$.

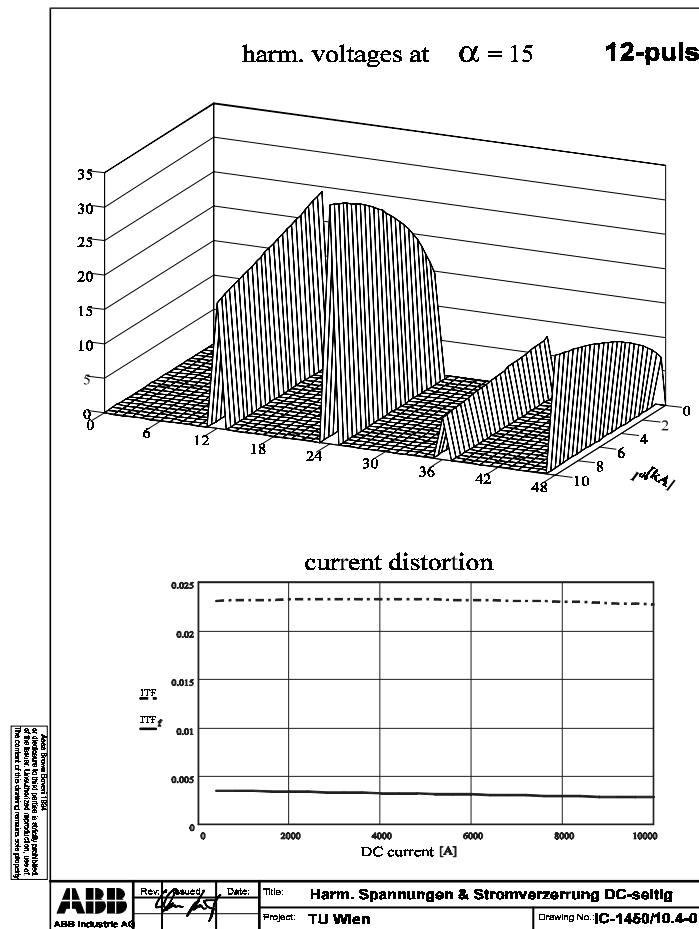


Fig.2: Harmonic voltages for ramping with a constant ignition angle (upper part) and distortion of the current - ITF current without filter, ITF_f distortion including the filter (lower part).

The field versus time profile is selected using a graphical surface program written under “In Touch”. This PC is connected to a PM A324 processor which drives directly the thyristors. The chosen $I(t)$ profile is checked with respect to the produced heat within the thyristor bridge. If some input requires lie above upper limits of the regulating electronic, the pulse can not be started. The systems contain a PI regulator - where for each point the KP- as well as the Tn-values can be chosen. This gives totally 40 free parameters which have to be optimised with respect to a smooth field versus time profile. Additionally are all switches and changeable connections watched - only if the circuit is in a proper condition the system allows to generate the current pulse.

With this system a maximum field of 40T in a bore of 20 - 30mm with a plateau of about 100ms shall be achieved. For testing the installation a liquid nitrogen cooled high field magnet (test – magnet) with the in table I given technical specifications was constructed. During first tests with the magnet a bipolar maximum field of 25T with a plateau time of 200 ms was achieved. In this magnet a hysteresograph for measuring the hysteresis loop up to 20T in temperatures between room temperature and 500°C is proposed. A second magnet with a maximum field of 40T in a bore of 25 mm for low temperature magnetisation measurements was also constructed.

Table I: Specifications of the test magnet:

parameter	value
inner diameter	30 mm
outer diameter	108 mm
height	154.8 mm
Number of windings	598
Number of layers	28
filling factor	0.85
wire	Cu+1%Ag, rectangular, $A = 21\text{mm}^2$
$\mu_0 H_{\text{max}}$	20T at $I = 5000\text{A}$ for $t = 200\text{ms}$
Inductivity (measured)	15.6 mH
Resistivity (measured)	0.16Ω at $T = 300\text{K}$
Calibration constant	3280 A/m/A
Maximal yield strength	360 MPa
resistiv losses	1.48 MW
Final temperature	110K

The field versus time profile is selected using a graphical users interface. The chosen $I(t)$ profile is checked with respect to the heat produced within the thyristor bridge. If some required input is above the upper limits of the regulating electronic, the pulse cannot be started. The system contains a PD regulator - where for each point the P as well as the D value can be chosen. This gives in total 40 free parameters that are optimised with respect to a smooth field versus time profile. In additional the heating of the magnet for each $I(t)$ profile has to be calculated. The $I(t)$ profile determines the increase of temperature (from T_A to T_E)

$$\text{according to } \int_0^{\tau} I^2(t) R(T(t)) dt = \int_{T_A}^{T_E} c(T) \cdot m \cdot dT$$

where $R(t)$ is the resistivity of the magnet, c is the specific heat and m is the mass of the magnet. All switches and changeable connections are watched as well - the system allows to generate the current pulse only if the circuit is in a proper condition.

b) Field generation

Fig.3 shows a block diagram of the entire high field set-up. The system allows – depending on the switching of the thyristor bridges- three types of pulses:

- parallel switching: $2 \times I_{\text{max}} = 13600\text{A}$, $U_{\text{max}} = 840\text{V}$: high current field pulse with one polarity.
- Serial switching: $I_{\text{max}} = 6800\text{A}$, $2 \times U_{\text{max}} = 1680\text{V}$: higher voltage; field pulse of one polarity; now used for generating the highest fields (40 T).
- Antiparallel switching: $I_{\text{max}} = \pm 6800\text{A}$, $U_{\text{max}} = \pm 840\text{V}$. Bipolar pulse as needed for real hysteresis measurements.

The operating temperature for performing various experiments ranges from 1.5 K up to 800 K.

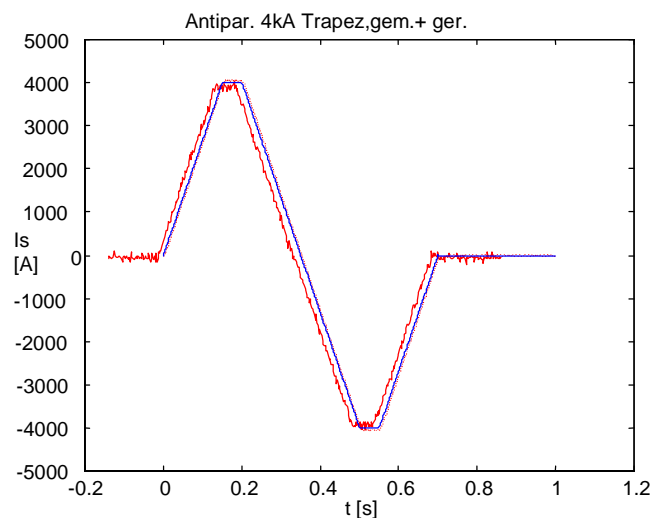
As was mentioned already for running the experiment the current versus time profile $I(t)$ can be defined with 20 points $I(t_i)$. In each point the P and I parameter for the digital PI-regulator

must also be given. The problem is that the high field magnet is a not linear regulated load because the resistivity increases with the temperature due to Joule heating of the magnet. This means that when a new profile $I(t)$ is given first a simulation has to be performed which calculates the increase of the temperature (from a starting value) of the pulse magnet. In this software is also included the role of the thyristor bridge. This allows to optimize the regulator parameter without heating up the pulse magnet with many unnecessary “shoots”.

In the following chapter the result of two different simulation procedures.as were performed with the test magnet are shown. Additionally the measured $I(t)$ profil is given the agreement between the theoretical and measured curve is very good.

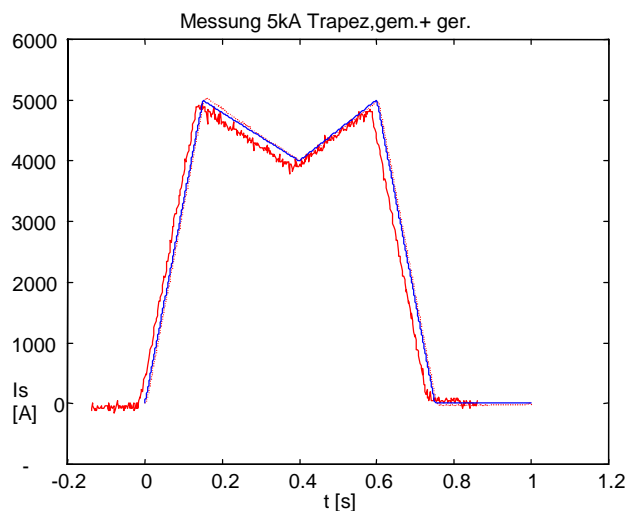
a) anti-parallel circuit with high proportional amplification factor.

$K_p = 50$, $T_n = 150\text{ms}$.



b) Parallel circuit with high amplification factor.

$K_p = 50$, $T_n = 150\text{ms}$



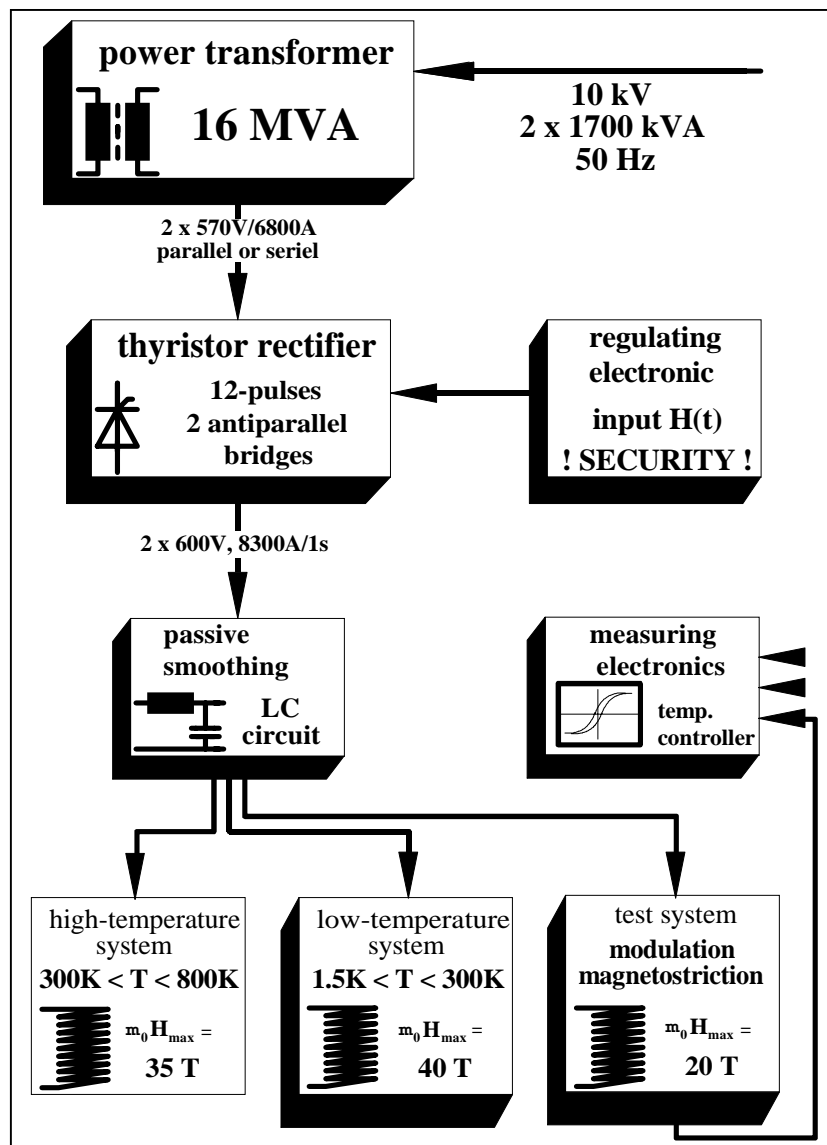


Fig.3: Block diagram of the “Austromag” high field installation.

c) Available Systems

Within the FWF project (Proj. Nr P 13146 PHY) three different experimental facilities were constructed and built up:

- **Low temperature system:** this consist of the 40T magnet from Metis, a flow cryostat from Cryogenics (1.5K up to 300K) with a sample space of 12mm. In this cryostat a pick-up system was installed in order to allow magnetization measurements. A sample holder for magnetoresistance measurements was also constructed.
- **High temperature system:** For this purpose one magnet was constructed which delivers 35T in a 25 mm bore. Because this bore was unfortunately just not sufficient for the glass isolated furnace a second self made 30 T magnet with a bore of 58 mm was constructed. In this bore a furnace which allows magnetization measurements

from room temperature up to 500°C was constructed and already tested. Also such a magnet can be used to test modulation methods using an inset coil.

➤ **Test system:** this is a room temperature system with a self made magnet which generates fields up to 25T in a bore of 25mm. The purpose of this system is to test new measuring methods. In the moment there the magnetostriction system is installed.

d) The high field magnets

All pulse magnets have to be optimized with respect to the available power, the heating of the magnet and the stresses. As an example the procedure for a self made 35T magnet is shown: In order to reduce the heating of the magnet, it is generally operated in liquid nitrogen (77 K). The field homogeneity over 30 mm is better than 1 %. In the installation as shown in Fig.3 three different magnets are used. The first magnet for the high temperature system is limited to a maximum field of 34.2 T in a bore of 30 mm. It consists of 28 layers with 23 turns for each layer - see also Fig 4. The resistivity at 77 K is 0.0206 Ω and after a high field pulse it is 0.0679 Ω ; the inductance of the magnet is 0.017 H. During a pulse with the maximum field (100 ms rise time, 100 ms plateau time, 150 ms fall time) the temperature increases from 77 K to 122 K.

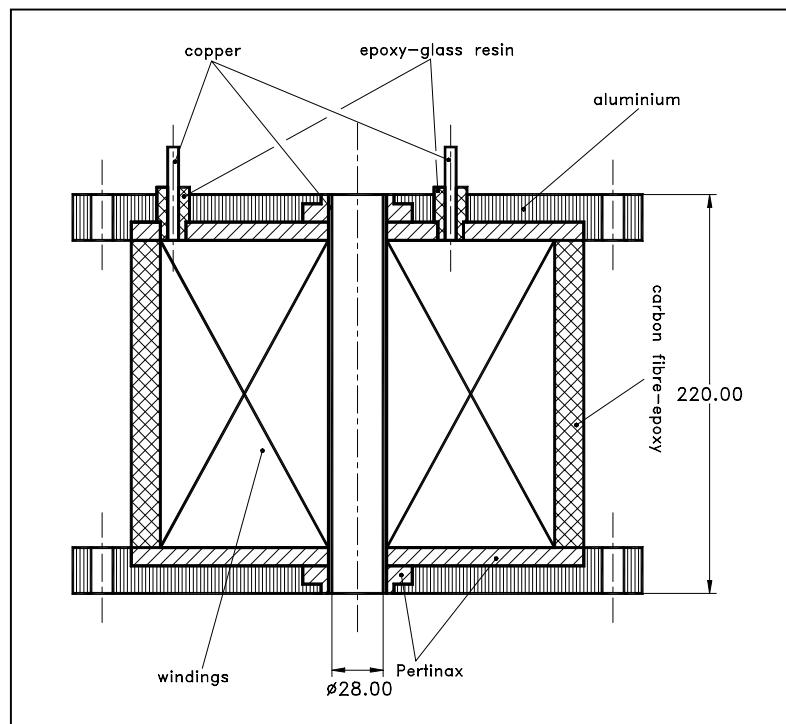


Fig.4. Drawing of the home-built 35 T magnet.

The magnet is wound using a Cu+1%Ag wire with a cross section of 3×7 mm – the yield strength of this material is 410 MPa. The magnet is reinforced with carbon fibre. The maximum tangential stress at a field of 34 T is 360 MPa on the inner layer; the stress due to plastic deformation after a 34 T field is about 300 MPa. This means the magnet is mechanically stable. **Because the bore of 30 mm was unfortunately just not sufficient for**

the glass and vacuum isolated furnace a second self made 30.3 T magnet at 8900A (at 77K) with a bore of 60 mm (58 mm available) was constructed. It is wound also of Cu-1%Ag wire with a cross section of 3x7mm and has 26 layers with 24 windings per layer. The magnet has an inductivity of 21.5 mH. This magnet accommodates the high temperature set-up. Such a magnet is also interesting for a two coil system with which in a later step fields beyond 40T shall be achieved.

The 40 T magnet was delivered by the company Metis. This magnet is reinforced with stainless steel, which has the advantage of a better heat conductivity as well as an eddy current damping for high frequency distortions. The measuring device and the sample is located in a nonmagnetic stainless steel cryostat from Cryogenics with a free sample space of 12 mm in the tail. This magnet is used for the low temperature system.

e) High temperature system

Rather unique is the combination of high fields and high temperatures. For the magnetic material characterization this is a very useful tool. A sketch of the high temperature set-up is shown in Fig.5.

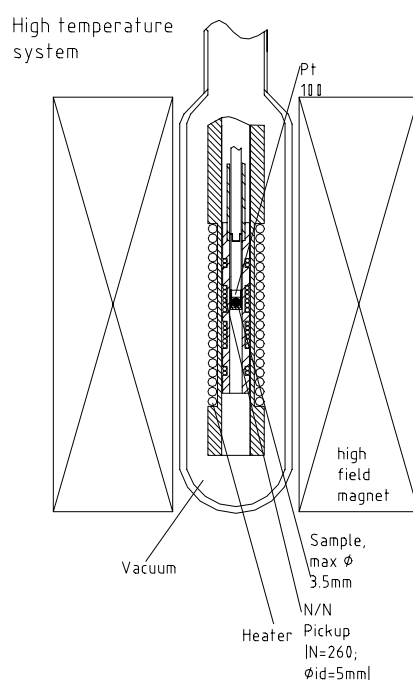


Fig.5: Sketch of the high temperature set-up.

The purpose of the system is to allow magnetization measurements between room temperature and 800 K at fields up to ± 20 T. The high temperature system consists of a vacuum isolated quartz glass tube. Inside is a coaxial furnace wound from an industrially available coaxial heater. In the furnace a N/N pick-up system is installed. This is made from Ag wire insulated with ceramic, accommodating samples with dimensions up to 4 mm. The whole system is wound on ceramic tubes. The measurements can be performed in vacuum or in a protective gas atmosphere. The magnetization is calibrated using pure Ni; the field is first calibrated using well known permanent magnets. The temperature is measured with a Pt100.

Field and magnetization calibration: To calibrate high pulsed magnetic fields is generally a not simple task. Within an industrial EC-funded project an accurate calibration procedure for M and H was developed. For the field calibration a by NMR calibrated induction coil is used. From the NMR calibration the effective winding area is determined. If this is known a field calibration using the law of induction with an absolute accuracy of 1.5% is possible. The magnetization calibration uses generally well known samples such as Fe or Ni. In pulsed magnetic fields also soft magnetic but non-conducting materials such as Fe₃O₄ can be used. Using such a procedure an absolute M-calibration within 1.5% is possible. We are additionally testing hall probes. With hall probes first pulsed fields shall be measured. Here the linearity of the hall constant may be a problem. We have for this purpose a special high field (30 T) Hall probe.

2) Data acquisition

All signals coming from the experiments are linked to a measuring card DAS 1800 ST of National Instruments. This card has 8 differential analog input channels, an ADC with 12 bits resolution and a sampling rate of 312.5 kS/s. It has a programmable preamplifier with an input range of ± 20 mV up to ± 5 V. A Labview data handling program was developed. Additionally exists a Digital storage oscilloscope from Tektronix (Model: TDS 420A) with 4 different channels where also a Labview program was written which can be used to read and handle the data.

The cables coming from the experiment, all amplifiers etc. have to be shielded carefully in order to reduce noise coming from the thyristors. The connections generally use differential input in order to benefit from the common mode rejection. The whole measuring electronics is shielded by a grounded copper box.

The pick-up system delivers two signals which are proportional to dM/dt and dH/dt . Afterward the signal is integrated either using an analogue integrator or by software. This procedure delivers finally $M(H)$ at a certain temperature T . Beside the already mentioned problems of not well compensated pick-up system and noise on the power supply the sensitivity is limited by the resolution (of the integrator or of the ADC) and the drift of the integrator. The two signals which delivers voltages $u_M(t)$ and $u_H(t)$ are connected either to the digital storage oscilloscope or to the measuring card which is connected to a PC.

3) Noise Problems - thyristor noise

As was said in detail in Chapt.1 the current regulation is performed with thyristors. This causes distortions that consist of high frequency harmonics. Using the 6-thyristor bridge the ripple has a periodicity of 6×50 Hz whereas for the 12-pulse bridge the periodicity is 12×50 Hz. Because the usual dH/dt of this system is rather low these high frequency distortions are a large problem in all kinds of measurement. In order to reduce this noise a rather simple solution was found. The field ripple is reduced by the use of a “short circuit ring” of a good conducting material (e.g. Cu) placed in the bore of the magnet. The idea is to use the eddy currents caused by the high frequency ripple as a damping source. This short circuit ring is described by the following differential equation:

$$R i_K + L \frac{di_K}{dt} = \frac{d\Phi_{ext}}{dt} \cdot N$$

R and L are the resistance and inductance of the ring respectively, N number of turns ($=1$), and I_k is the induced short circuit current.

The flux Φ is the external flux Φ_{ext} of the pulse magnet reduced by the flux due to the induced short circuit current. The damping due to the existence of the ring is therefore in a first approximation:

$$\left| \frac{\Phi}{\Phi_{ext}} \right| = \frac{R}{\omega L} = \frac{4\rho}{\omega \cdot \mu_0 D_m \cdot b}$$

with $D_m = (D_a + D_i)/2$ (mean diameter of the ring, b is the thickness and ρ the specific resistivity of the material. This formula is valid if the skin depth is larger than b . This delivers a reduction of the field ripple up to a factor 10 which is very useful. However the thyristor noise is still a limiting factor which disturbs sensitive measurements essentially.

4) Methods to increase the sensitivity

Up to now for measuring the magnetization principally only so-called “DC-methods” were used. One just integrates everything which comes. This delivery by principle high noise and limits the achievable sensitivity. Additionally the “thyristor-noise” problems have to be overcome. Therefore are here methods proposed to enhance the signal to noise ratio using a lock-in technique but also ways to improve the coupling between the sensor (the pick-up coil) and the sample.

To increase the sensitivity two different methods for measuring the magnetisation were tested:

4.1 Vibrating sample technique:

The vibrating sample method is well established in static magnetometers. It has the advantages that also here the lock-in technique can be used which gives an improved signal to noise ratio. The pick up coils deliver an ac-signal, which is proportional to the magnetisation. The advantage of this method compared with a modulation technique is that a piezo-electric actuator can be used in the cryostat directly. Therefore the use should be much more easy. Additionally the effects of induction voltages resulting from a not ideal compensation can be suppressed because the frequency of the fundamental high field pulse is much lower than that of the induced voltage. This device is still under test.

4.2 Modulation technique

In this case the slowly with time varying field $H(t)$ is modulated with an ac-field of the shape $H_0 \cdot \sin \omega t$, where the frequency ω is high compared with the fundamental wave (e.g. pulse duration of high field pulse 500ms; modulation frequency 1kHz). Also in this case a lock-in method can be used in order to measure the induction voltage. This allows also a dramatic improvement of the signal to noise ratio. The weak point of this method is that it is also limited by the skin depth..

4.3 Surface coils

Development and construction of surface measuring coils was already started by us and tested in our pulsed field system. Small wire wound pick-up coil system consist of two wire wound

coils commercially available by Magnet-Physik AG Steingroever (Germany). The small wire wound pick-up coil has the following features: diameter 2.5 mm, height 0.4 mm, resistance around 25 Ω , the effective area $N \cdot A = 4.15 \text{ cm}^2$, and its inductance value is 43 μH approximately. Due to its small output resistance it is possible to compensate the pick-up signal using a commercial differential amplifier, getting very good results. The advantage of this coil with respect to thin film coils is its low output resistance, which reduces the noise as well as cause no phase shift problems. Additionally have these coils a much larger effective winding area. Therefore measurements with such coils are easy to perform, The induced signal is of the order of 100mV with a low noise level.

5) Experimental results

In the following as an example some experimental results are shown.

a) Magnetic viscosity

For pulsed field measurements a time dependent magnetization can effect the reliability of the obtained. data. Up to now the viscosity parameter S_v was only determined in slowly time varying fields (typically T/s). It is completely unclear if the “ S_v ” as determined in slowly time varying fields can be applied for time dependence in pulsed fields with sweep rates up to 5000T/s. Therefore we started to investigate S_v applying different sweep rates of dH/dt . Here the Austromag system where a linear dH/dt can be chosen, plays a very important role because it allows well defined intermediate sweep rates of dH/dt (typically 100T/s).

As an example $\text{SmCo}_{5-x}\text{Cu}_x$ is an interesting candidate because it exhibits a strong magnetic viscosity effect. As an example we show in Fig. 6 the temperature dependence of the coercive field of a 2/17 based permanent magnet in comparison with data as obtained in our fast pulsed field magnetometer. The values as obtained with the Austromag quasi-static system measured with a linear dH/dt of 67 T/s are below those obtained in a short pulsed field magnetometer (sinusoidal pulse; pulse duration about 10 ms; about 2000 T/s); this is a consequence of the magnetic viscosity of this sample.

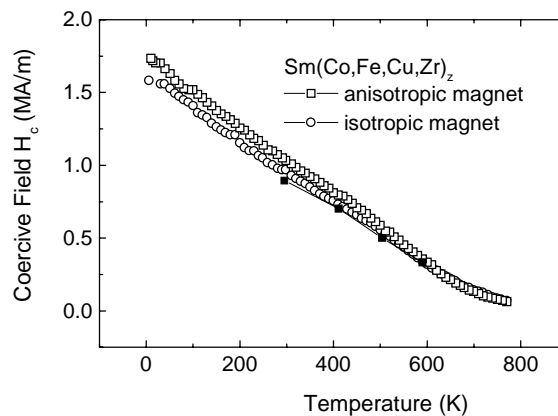


Fig. 6: Temperature dependence of the coercivity of a 2/17 based permanent magnet, measured in a short pulse system in comparison with the results obtained in the quasi-static magnetometer (■ points measured in the quasistatic system).

b) Magnetostriction measurements.

The magnetostriction is an important intrinsic parameter of magnetic materials. Therefore we started a program to measure this property on available technical permanent magnets. Because many of these samples have a high magnetocrystalline anisotropy, high external fields are necessary. The magnetostriction in high fields can be measured using standard strain gauges. Here strain gauges of the type XY 91 3/120 with an ac-bridge from Hottinger Baldwin (model KWS 3085A) with a carrier frequency of 50 kHz were used. An ac-bridge is favourable in order to suppress noise as well as induction voltages.

First measurements of the magnetostriction coefficients obtained at room temperature on a technical anisotropic barium ferrite (supplied by Schramberg A.G.) are shown in Fig. 7. The meaning of the different magnetostriction coefficients is: λ_{pc} : H perpendicular and DMS measurement parallel to the c-axis; λ_{cc} : H and DMS measurement direction parallel to the c-axis; λ_{pp} : H and DMS measurement direction perpendicular to the c-axis; λ_{cp} : H parallel and DMS direction perpendicular to the c-axis.

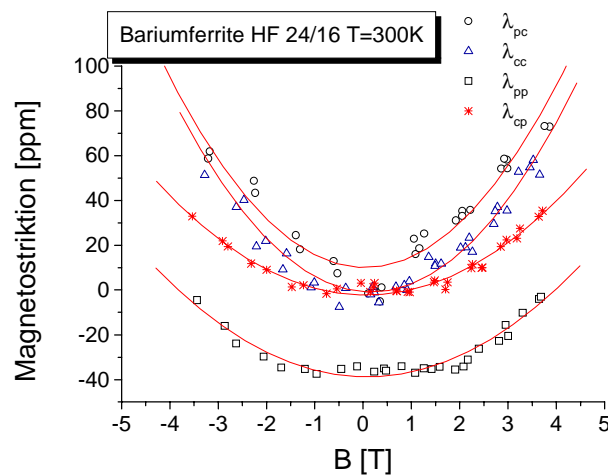


Fig.7: Magnetostriction measurements at room temperature on an anisotropic barium ferrite magnet made by Schramberg (HF24/16).

6) Summary and outlook

The main work was up to now concentrated to construct high field measuring systems. There exist now three measuring places as was described. The measuring systems work but there is still effort necessary in order to improve the signal to noise ratio. Principally exists now the possibility to measure the magnetization, the magnetostriction and the magnetoresistance in high magnetic fields. These possibilities are used to investigate various magnetic compounds in order to determine magnetic phase diagrams, exchange constants etc.

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